ICMS

Instituto de Ciencia de Materiales de Sevilla

Memoria de Actividades
Annual Report

Consejo
Superior de
Investigaciones
Científicas

Universidad
de Sevilla

Junta de
Andalucía

2016

Consejo Superior de Investigaciones Científicas
Universidad de Sevilla
Junta de Andalucía

INSTITUTO DE CIENCIA DE MATERIALES DE SEVILLA

Memoria de Actividades
Annual Report
2016

Centro de Investigaciones Científicas “Isla de la Cartuja” (cicCartuja)
c/ Americo Vespucio, nº 49
41092 Isla de la Cartuja SEVILLA
Tfno.: +34 954 48 95 27 Fax: +34 954 46 01 65
http://www.icms.us-csic.es
buzon@icmse.csic.es

Edita
Instituto de Ciencia de Materiales de Sevilla (ICMS)
Centro Mixto entre el Consejo Superior de Investigaciones
Científicas y la Universidad de Sevilla
Centro de Investigaciones Científicas Isla de la Cartuja (cicCartuja)
c/ Americo Vespucio, 49
41092 Isla de la Cartuja, Sevilla
http://www.icms.us-csic.es

Diseño y Maquetación
José Carlos Rivero Cabello

Comisión Memoria-ICMS
Gerardo Colón Ibáñez - Francisco José Gotor Martínez - Hernán R. Míguez García

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El ICMS en 2016

ICMS in 2016

Presentación Presentation

A través de esta Memoria 2016, el Instituto de Ciencia de Materiales de Sevilla (ICMS) pretende comunicar lo mejor de las actividades científicas realizadas a lo largo del periodo.

El ICMS es un Centro Mixto del Consejo Superior de Investigaciones Científicas (CSIC) y la Universidad de Sevilla (US), cofinanciado asimismo por la Junta de Andalucía. Está incluido en el Área de Ciencia y Tecnología de Materiales, una de las 8 en las que el CSIC estructura sus actividades científicas. Desde su creación en 1986, está integrado por personal científico de la Universidad de Sevilla y el CSIC, cuenta en la actualidad con 129 personas, 44 de las cuales son científicos permanentes.

De acuerdo con la estructura establecida en el vigente Plan Estratégico 2014-2017, nuestro centro se organiza en torno a 5 líneas de investigación, las que enraizando en disciplinas de la Química y Física del Estado Sólido y de la Ingeniería de Materiales, pretenden abarcar los aspectos más modernos y novedosos de la Ciencia de Materiales actual. Nuestro empeño fundamental para los años venideros sigue siendo el desarrollo de trabajos de investigación que definan “el estado del arte” de la disciplina, y que a su vez sirva de apoyo al desarrollo de un nuevo sistema económico en el que la base científica y tecnológica pase a ser un pilar fundamental de la misma. Por tanto, uno de nuestros más importantes objetivos es realizar una actividad de I+D fuertemente vinculada al entorno socio-económico, local, autonómico e internacional.

A pesar de las actuales limitaciones en la financiación, incorporación de nuevo personal y de disponibilidad de espacio que venimos padeciendo, en este último caso tanto para la ubicación de personal científico y técnico como para la instalación de equipamiento científico, la promoción de la actividad científica, la instalación del equipamiento científico más innovador y la incorporación de jóvenes científicos seguirá siendo nuestro gran objetivo diario.

Through this Activity Report 2016, the Institute of Materials Science of Seville (ICMS) is trying to communicate the best of its scientific activities for the period.

The ICMS is a joint Center of the Spanish Research Council (CSIC) and the University of Seville (US), also funded by the Junta de Andalucia. It is included in the “Field of Science and Technology of Materials”, one of the eight within the scientific framework of the CSIC. It was founded in 1986, and integrated by scientific staff of the University of Seville and the CSIC, including today more than 139 people, 44 of which are permanent scientific staff.

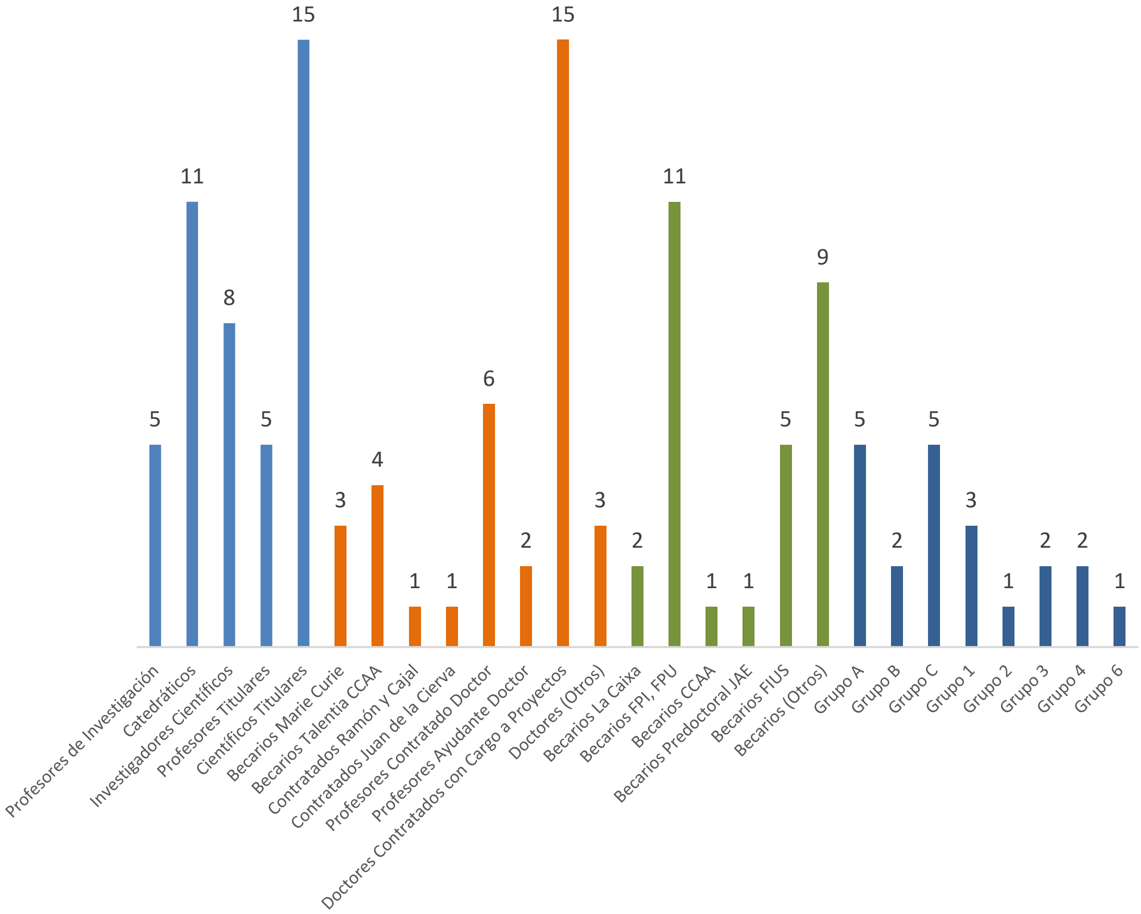
The current Strategic Plan 2014-2017 organizes our Center in 5 lines of research, which related with disciplines of solid state chemistry and physic, as well as materials engineering subjects, are intended to cover the most modern and innovative aspects of the current material science. Our basic commitment for years to come is to support, as far as possible, the development of a new economic system, in which the scientific and technological basis should become a fundamental pillar of the same. Therefore, development of a R+D activity according to the State of the Art, is the top priority, if not the only, of our research centre. In spite of the financial, personnel and space limitations we are suffering today, both for the location of scientific and technical staff and for the installation of scientific equipment, the promotion of the scientific activity, the installation of the most innovative scientific equipment and the incorporation of young scientist staff will remain our great daily goal.

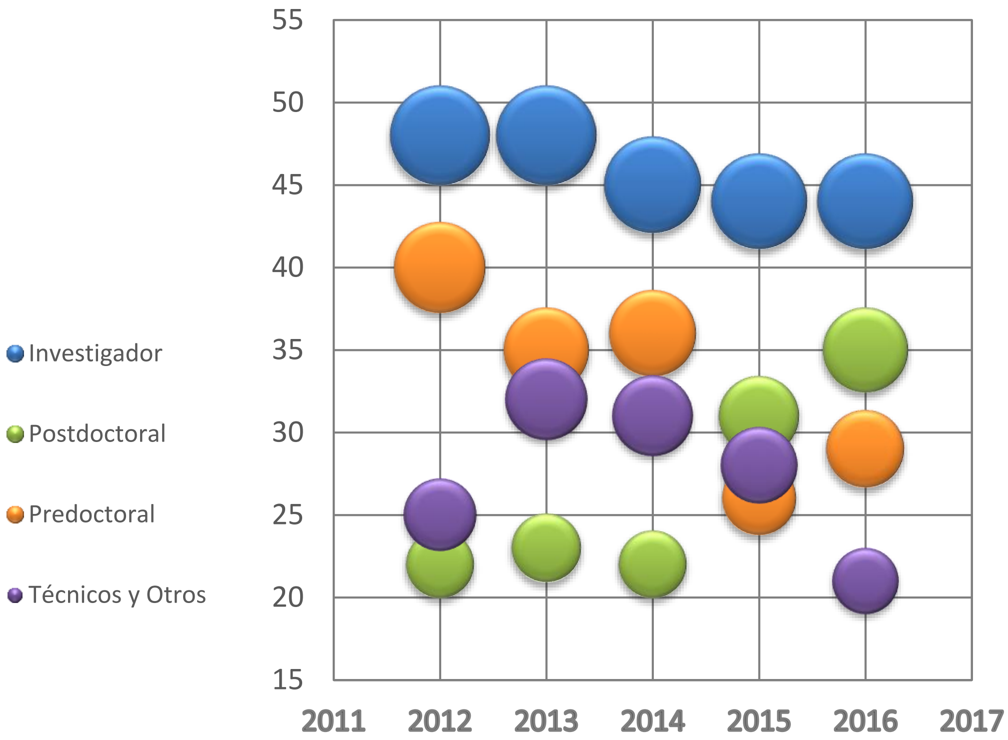
Dr. **Alfonso Caballero Martínez**
Director del Instituto de Ciencia de Materiales de Sevilla

Datos Estadísticos del ICMS
Statistical Data of ICMS

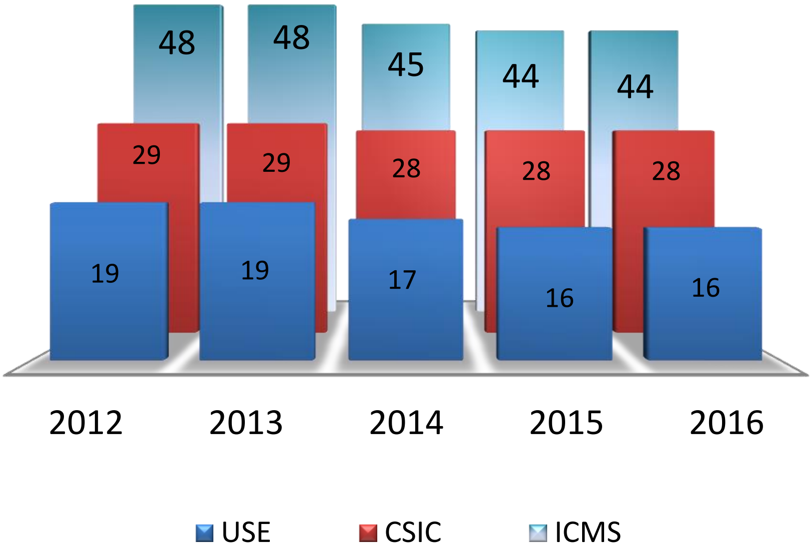
RECURSOS HUMANOS / HUMAN RESOURCES

Distribución del personal por categorías - 2016
Distribution by professional category - 2016



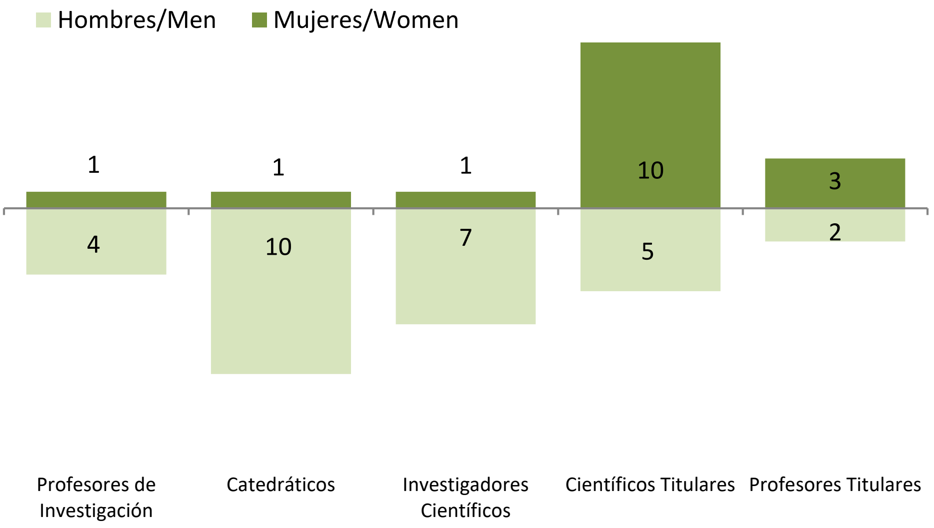


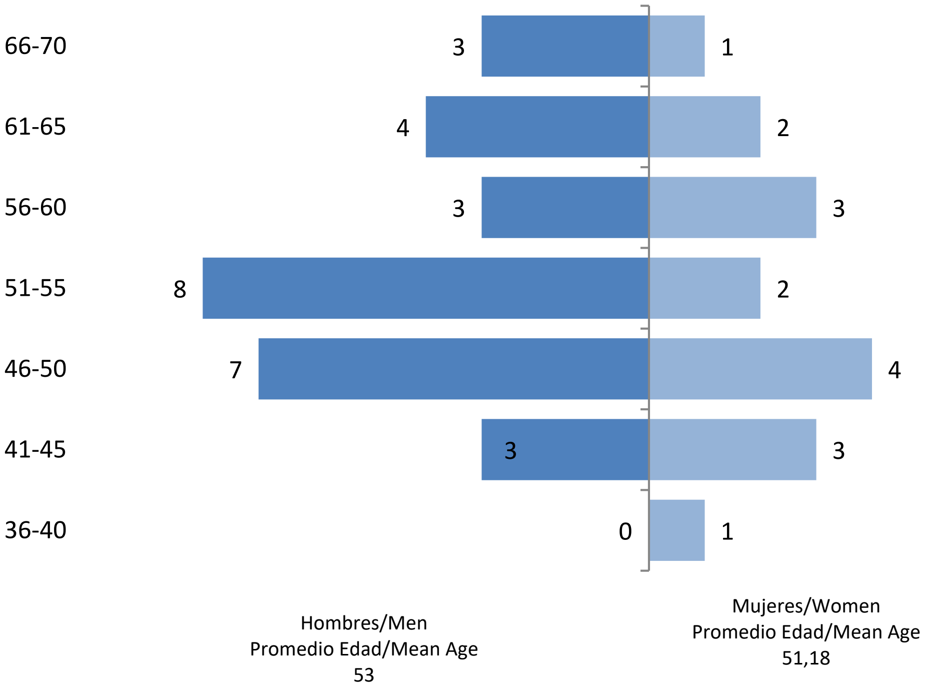
Evolución 2012-2016 del personal
Evolution of Staff



Evolución 2012-2016 del personal investigador
Evolution of Research Staff

Distribución del personal Investigador (sexo y edad)
Distribution by Scientific staff (gender and age)





FINANCIACIÓN / FUNDING

Evolución 2012-2016 de la Financiación conseguida por año
Evolution of the funding of the ICMS

PRODUCCIÓN CIENTÍFICA / SCIENTIFIC PRODUCTION

Revistas SCI en las que se ha publicado algún artículo, por orden de Factor de Impacto
Number of Scientific Papers published in a Specific Journal Arranged by Their Impact Factor (SCI)

REVISTA JOURNAL	ARTÍCULOS PAPERS	FACTOR DE IMPACTO IMPACT FACTOR (*)
PROGRESS IN MATERIALS SCIENCE	1	31,140
ENERGY & ENVIRONMENTAL SCIENCE	1	29,518
ADVANCED MATERIALS	1	19,791
LIGHT-SCIENCE & APPLICATIONS	1	14,098
ACS NANO	1	13,942
NANO LETTERS	1	12,712
ACS CATALYSIS	1	10,614
APPLIED CATALYSIS B-ENVIRONMENTAL	7	9,446
JOURNAL OF PHYSICAL CHEMISTRY LETTERS	1	9,353
JOURNAL OF MATERIALS CHEMISTRY A	3	8,867
TRAC-TRENDS IN ANALYTICAL CHEMISTRY	1	8,442
ACS APPLIED MATERIALS & INTERFACES	5	7,504
NANOSCALE	1	7,367
ADVANCED IN COLLOID AND INTERFACE SCIENCE	1	7,223
APPLIED ENERGY	1	7,182
ADVANCED OPTICAL MATERIALS	2	6,875
JOURNAL OF CATALYSIS	1	6,844
JOURNAL OF POWER SOURCES	2	6,395
CHEMICAL COMMUNICATIONS	1	6,319
CHEMICAL ENGINEERING JOURNAL	5	6,216
SENSORS AND ACTUATORS B-CHEMICAL	5	5,401
ACTA MATERIALIA	1	5,301
JOURNAL OF MATERIALS CHEMISTRY C	2	5,256
CORROSION SCIENCE	2	5,245
SCIENCE OF THE TOTAL ENVIRONMENT	1	4,900
ELECTROCHIMICA ACTA	2	4,798
CATALYSIS TODAY	4	4,636
JOURNAL OF PHYSICAL CHEMISTRY C	3	4,536
ENERGY	1	4,520
MATERIALS & DESIGN	3	4,364

REVISTA JOURNAL	ARTÍCULOS PAPERS	FACTOR DE IMPACTO IMPACT FACTOR (*)
APPLIED CATALYSIS A-GENERAL	2	4,339
INTERNATIONAL JOURNAL OF NANOMEDICINE	1	4,300
JOURNAL OF CO2 UTILIZATION	1	4,292
SCIENTIFIC REPORTS	2	4,259
JOURNAL OF MOLECULAR CATALYSIS A-CHEMICAL	2	4,211
TALANTA	1	4,162
PHYSICAL CHEMISTRY CHEMICAL PHYSICS	3	4,123
CRYSTAL GROWTH & DESIGN	1	4,055
TOPICS IN CURRENT CHEMISTRY	1	4,033
DALTON TRANSACTIONS	1	4,029
PHYSICAL REVIEW B	1	3,836
LANGMUIR	1	3,833
BIOPHYSICAL JOURNAL	1	3,656
JOURNAL OF MOLECULAR LIQUIDS	1	3,648
MICROSOPOROUS AND MESOPOROUS MATERIALS	2	3,615
INTERNATIONAL JOURNAL OF HYDROGEN ENERGY	3	3,582
JOURNAL OF DENTISTRY	1	3,456
NANOTECHNOLOGY	1	3,440
JOURNAL OF THE EUROPEAN CERAMIC SOCIETY	1	3,411
OPTICS EXPRESS	1	3,307
JOURNAL OF THE ELECTROCHEMICAL CHEMISTRY	2	3,259
JOURNAL OF ALLOYS AND COMPOUNDS	3	3,133
RSC ADVANCES	7	3,108
APPLIED CLAY SCIENCE	3	3,101
CATALYSTS	1	3,082
CERAMICS INTERNATIONAL	4	2,986
JOURNAL OF CHEMICAL PHYSICS	1	2,965
PLASMA PROCESSES AND POLYMERS	3	2,846
ULTRAMICROSCOPY	1	2,843
JOURNAL OF THE AMERICAN CERAMIC SOCIETY	1	2,841
ENERGY TECHNOLOGY	1	2,789
JOURNAL OF LUMINESCENCE	1	2,686
JOURNAL OF RAMAN SPECTROSCOPY	1	2,679
JOURNAL OF MATERIALS SCIENCE	2	2,599
SURFACE & COATINGS TECHNOLOGY	1	2,589
JOURNAL OF PHYSICS D-APPLIED PHYSICS	3	2,588

REVISTA JOURNAL	ARTÍCULOS PAPERS	FACTOR DE IMPACTO IMPACT FACTOR (*)
MATERIALS LETTERS	1	2,572
DIAMOND AND RELATED MATERIALS	1	2,561
SPECTROCHIMICA ACTA PART A-MOLECULAR AND BIOMOLECULAR SPECTROSCOPY	1	2,561
TOPICS IN CATALYSIS	1	2,486
MATERIALS RESEARCH BULLETIN	1	2,446
INTERNATIONAL JOURNAL OF REFRACTORY METALS & HARD MATERIALS	1	2,156
JOURNAL OF APPLIED PHYSICS	1	2,068
SURFACE SCIENCE	2	2,062
COMPTES RENDUS CHIMIE	1	1,879
THIN SOLID FILMS	2	1,879
PHARMACEUTICAL DEVELOPMENT AND TECHNOLOGY	1	1,860
JOURNAL OF CULTURAL HERITAGE	1	1,838
ARCHAEOMETRY	1	1,470
NUCLEAR ENGINEERING AND DESIGN	1	1,142
NUCLEAR INSTRUMENTS & METHODS IN PHYSICS RESEARCH SECTION B-BEAM INTERACTIONS WITH MATERIALS AND ATOMS	1	1,109
PHYSICS OF THE SOLID STATE	3	0,860
BOLETÍN DE LA SOCIEDAD ESPAÑOLA DE CERÁMICA Y VIDRIO	1	0,560
FERROELECTRICS	1	0,551
REVISTA DE METALURGIA	1	0,345
ACS ENERGY LETTERS	1	0
ACS OMEGA	1	0
Total	**151**	**4,933**

(*) Factor de Impacto correspondiente al año 2016
Journal Citation Reports of 2016

COMPOSICIÓN Y ESTRUCTURA

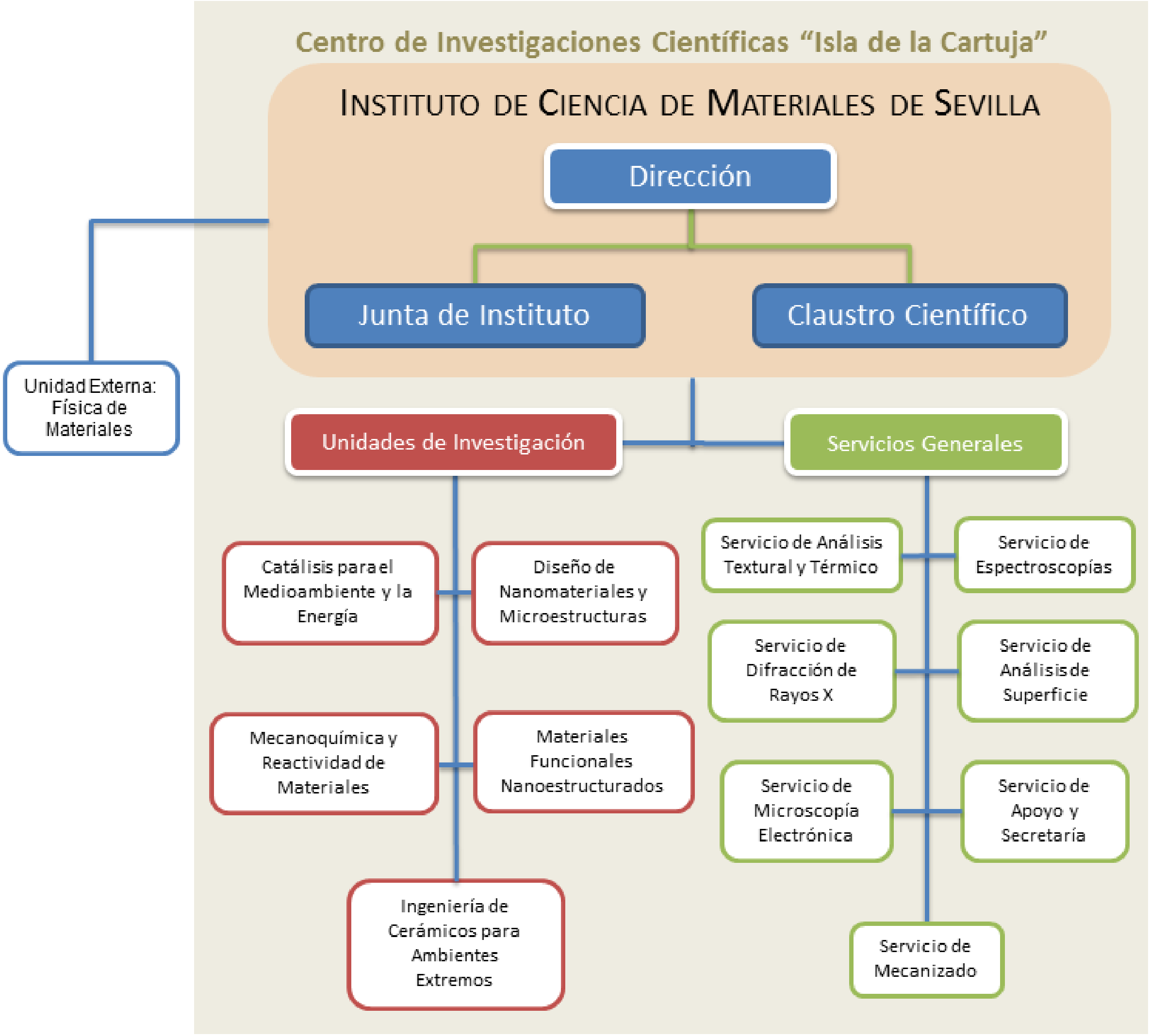
STRUCTURE AND ORGANISATION

EL INSTITUTO / THE INSTITUTE

El Instituto de Ciencia de Materiales de Sevilla (ICMS) fue creado en 1.986. En 1996 se trasladó a unos nuevos locales en la Isla de la Cartuja, integrándose, junto con otros dos Institutos Mixtos, en el Centro de Investigaciones Científicas Isla de la Cartuja (Junta de Andalucia - Consejo Superior de Investigaciones Científicas - Universidad de Sevilla). El ICMS se estructura en cinco Unidades de Investigación ubicadas en el edificio de la Isla de la Cartuja y una Unidad Externa en el campus de Reina Mercedes, Facultad de Física. En la actualidad, el Instituto está formado por grupos de investigación del CSIC y de la Universidad de Sevilla. Estos grupos tratan de aunar sus esfuerzos en diversas áreas de la física y química del estado sólido, físico-química de superficies y otras disciplinas relacionadas en la Ciencia de Materiales. Esta actividad persigue contribuir al desarrollo científico dentro de los Planes de Investigación tanto Autonómicos como Nacionales en el área de la Ciencia y Tecnología de Materiales, así como de las equivalentes de la UE.

The Institute of Materials Science of Seville (ICMS) was created in 1986. In 1996, it moved to new premises at the Isla de la Cartuja, combining with two other Mixed Institutes to make up the Isla de la Cartuja Scientific Research Centre (Junta de Andalucia - Consejo Superior de Investigaciones Científicas - Universidad de Sevilla). The Institute comprises three research units housed in the building at the Isla de la Cartuja and an external unit at the Physics Faculty on the Reina Mercedes campus. Today it incorporates research groups of CSIC and the University of Seville. The aim of these groups is to unite efforts in various areas of solid-state physics and chemistry, physical chemistry of surfaces, and other related disciplines of materials science. Such activity seeks to contribute to scientific development within the Research Plans of both regional and state authorities, and their EU equivalents, in the field of materials science and technology.

ORGANIGRAMA / ORGANIZATION CHART



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Catalysis for Enviroment and energy

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- Lda. Cristina Megías Sayago (h. octubre)
- Ldo. Alberto Rodríguez Gómez (h. nov.)
- Lda. María Domínguez Gómez
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- Lda. Ángeles López Martín
- Lda. Sara Navarro Jaén
- Ldo. José Luis Santos Muñoz

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MECANOQUÍMICA Y REACTIVIDAD DE MATERIALES
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Dra. Concepción Real Pérez
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Profesor Vinculado "Ad Honorem"

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MATERIALES FUNCIONALES NANOESTRUCTURADOS NANOSTRUCTURED FUNCTIONAL MATERIALS

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Dr. Manuel Ocaña Jurado
Dr. Agustín Rodríguez González-Elipe

Catedráticos

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Diseño de Nanomateriales y Microestructuras
Tailored Nanomaterials and Microstructure

Personal / Personnel

Profesores de Investigación

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Dra. M. Carmen Jiménez de Haro
Lda. Olga Montes Amorín (cicCartuja)
Dª M. Inmaculada Rosa Cejudo

Visitas Institucionales y Prevención de Riesgos Laborales
Dr. Antonio Ruiz Conde

Unidad "Mecanoquímica y Reactividad de Materiales"
Lda. Belinda Sigüenza Carballo

Unidad "Materiales Funcionales Nanoestructurados"
Dr. Victor J. Rico Gavira

FÍSICA DE MATERIALES (UNIDAD EXTERNA DE INVESTIGACIÓN)
PHYSICS OF MATERIALS (EXTERNAL UNIT)

PERSONAL / PERSONNEL

Catedrático

Dr. Alejandro Conde Amiano
Dra. Clara F. Conde Amiano
Dr. Alberto Criado Vega
Dr. Arturo Domínguez Rodríguez
Dr. Victorino Franco García
Dr. Antonio Muñoz Bernabé

Profesor Titular

Dr. Javier S. Blázquez Gámez
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Dra. Ana Morales Rodríguez
Dr. Francisco Javier Romero Landa

Investigador Contratado

Dr. Jhon J. Ipus Bados

Becario Predoctoral

Ldo. Luis M. Moreno Ramírez

UNIDADES DE INVESTIGACIÓN

RESEARCH UNITS

CATÁLISIS PARA EL MEDIOAMBIENTE Y LA ENERGÍA

CATALYSIS FOR ENVIRONMENT AND ENERGY

PERSONAL / PERSONNEL

Catedráticos

Dr. Alfonso Caballero Martínez
Dr. José Antonio Navío Santos
Dr. José Antonio Odriozola Gordón

Investigadores Científicos

Dr. Miguel Angel Centeno Gallego
Dr. Gerardo Colón Ibáñez

Científicos Titulares

Dra. M. Carmen Hidalgo López
Dr. Juan Pedro Holgado Vázquez

Profesores Titulares

Dr. Manuel Macías Azaña
Dra. Francisca Romero Sarria

Investigadores Contratados

Dr. Luis Bobadilla Baladrón
Dr. Victor M. González-De la Cruz

Profesores Contratado Doctor

Dra. María Isabel Domínguez Leal
Dra. Anna Dimitrova Penkova
Dra. Svetlana Lyuvimirova Ivanova

Profesores Ayudante Doctor

Dra. Leidy Marcela Martínez Tejada
Dra. Rosa Pereñíguez Rodríguez

Becarios Predoctorales

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Lda. Sara Navarro Jaén
Ldo. José Luis Santos Muñoz

Proyectos de Investigación / Research Projects

Desarrollo de Materiales Foto-Funcionales para Aplicaciones Medioambientales Development of photo-functional materials for environmental applications

Código/Code:	CTQ2015-64664-C2-2-P
Periodo/Period:	01-01-2016 / 31-12-2018
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	76.320,75 €
Investigador responsable/Research head:	José Antonio Navío Santos
Componentes/Research group:	Maria del Carmen Hidalgo López, Manuel Macías Azaña

RESUMEN / ABSTRACT

La fotocatálisis heterogénea es un proceso avanzado de oxidación que ha sido objeto de una enorme cantidad de estudios relacionados con la purificación de gases y del agua. La mayoría de estos estudios se han realizado para el tratamiento de aguas y utilizando el TiO2 o materiales basados en este óxido y más recientemente, aunque en una clara minoría, se han estudiado otros óxidos inorgánicos binarios, ternarios y cuaternarios, predominando en todo caso los estudios de estos últimos materiales para el tratamiento de aguas. En cuanto al catalizador (base del proceso fotocatalítico) que es el responsable de la eficacia o fracaso del proceso, en la última década se han desarrollado numerosos y variados métodos de síntesis que han sido principalmente probados en procesos de degradación fotocatalítica en fase acuosa. Sin embargo, pocos estudios se han realizado con óxidos mixtos (binarios, binarios-acoplados, ternarios y cuaternarios) y menos en fase gasesosa.

En base a estas consideraciones y a la dilatada y reconocida experiencia que el grupo de este Subproyecto#2 tiene el campo de la síntesis y caracterización de materiales foto-funcionales (en el UV y UV/Vis), y debido al reducido número de estudios fotocatalíticos en fase gas, en su mayoría estudiando un sólo componente, se plantea en este Subproyecto#2, el desarrollo de materiales foto-funcionales que conduzcan a materiales basados no sólo en TiO2 con propiedades mejoradas sino a otros materiales basados en este óxido y a otros óxidos inorgánicos binarios, los obtenidos por acoplamientos de óxidos binarios y ternarios, que se obtengan por procedimientos de síntesis distintos (o modificados) a los ya recogidos en la bibliografía, y cuya fotoactividad sea evaluada por el grupo del Subproyecto#1, sin que se descarte ensayos previos de actividad fotocatalítica en agua por el grupo del Subproyecto#2.

Entre los materiales que se pretenden sintetizar en el Subproyecto#2 (empleando métodos no-hidrotermales, hidrotermales y sol-gel) se contemplan: óxidos binarios (TiO2, ZnO, ZnO2, Fe2O3, WO3, Bi2O3, Ta2O5 , La2O3), óxidos binarios acoplados (TiO2-WO3, TiO2-ZnO, TiO2-ZnO2; TiO2-Ta2O5, TiO2-La2O3, ZnO-Fe2O3 y ZnO2-Fe2O3), óxidos ternarios (Bi2WO6, Bi2WO6-ZnO, Bi2WO6-ZnO2, Bi2WO6-Fe2O3, Bi2Ti2O7, ZnWO4, La2Ti2O7), contemplando la foto-deposición de metales (Pt, Ag, Au) en los sistemas que muestren una actividad fotocatalítica considerable (Semiconductores/Metal).

Los mejores sistemas evaluados se remitirán al grupo del Subproyecto#1 para el estudio de la viabilidad fotocatalítica en la eliminación de NOx,COVs, CO, CO2 y SO2 presentes en emisiones gaseosas.

Heterogeneous photocatalysis is an advanced oxidation process which has been the subject of a huge amount of studies related to gas and water purification. Most of these studies have been performed for the treatment of water mainly by using the TiO2-based materials and more recently, although in a clear minority, by using other inorganic oxides binary, ternary and quaternary, predominating in all cases, the studies of the latter materials for water treatment. In terms of the photocatalyst, which is responsible of the efficiency of the photocatalytic processse, in the last decade have been developed numerous and varied methods of synthesis that have mainly been tested on processes of degradation in aqueous phase. However, few studies have been conducted with mixed oxides (binary, binary-coupled, ternary or quaternary) and less in gas phase.

Based on the above considerations and given the long and recognized experience that members of this Subproject# 2 have in the field of synthesis and characterization of photo-functional materials (UV and UV/Vis), an due also due to the small number of photocatalytic studies in the gas phase , most of them by studying a single component, the work arises in this Subproject # 2 is the development of photo-functional materials that lead to materials based, not only on TiO2 with improved properties but other materials based on this oxide and other binary inorganic oxides, those obtained by coupling of binary oxides and ternary, which are obtained by processes of different synthesis to those already reported in the literature, and whose photoactivity will be evaluated by the group of Subproject # 1, without discarding a prior testing photocatalytic activity in water by the group of Sub-group # 2.

Among the materials that are to be synthesized in the Subproject # 2 (by using non-hydrothermal, hydrothermal and sol-gel methods) are contemplated: binary oxides (TiO2, ZnO, WO3, Fe2O3, Bi2O3, Ta2O5, La2O3), coupled binary oxides (TiO2-WO3, TiO2-ZnO, TiO2-ZnO2, TiO2-Ta2O5, TiO2-La2O3, ZnO-Fe2O3 y ZnO2-Fe2O3), ternary oxides (Bi2WO6, Bi2WO6-ZnO, Bi2WO6-ZnO2, Bi2WO6-Fe2O3, Bi2Ti2O7, ZnWO4,La2Ti2O7) studying the photo-deposition of single metals (Pt, Ag, Au) on those prepared systems that exhibit significant photocatalytic activity (Semiconductor /Metal). Best evaluated systems will be forwarded to the Subprojet 1 for the feasibility study on the photocatalytic removal of NOx, VOCs, CO, CO2 and SO2 present in gaseous emissions.

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Sistemas Catalíticos Estructurados para la Producción de Biocombustible Structured Catalytic Systems for Biofuel Production

Código/Code:	ENE2015-66975-C3-2-R
Periodo/Period:	01-01-2016 / 31-12-2018
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	302.500 €
Investigador responsable/Research head:	José Antonio Odriozola Gordón
Componentes/Research group:	Anna Dimitrova Penkova, María Isabel Domínguez Leal, Francisca Romero Sarria

RESUMEN / ABSTRACT

La dependencia de nuestro actual sistema energético de las fuentes de combustibles fósiles y sus adversos efectos medioambientales están potenciando el desarrollo de fuentes de energía de origen renovable. Este es el caso de los biocombustibles de segunda generación. Los procesos de producción de combustibles a partir de biomasa lignocelulósica y residuos orgánicos son habitualmente catalíticos y se caracterizan por la necesidad de un intenso intercambio de calor asociado al elevado efecto térmico de las reacciones químicas implicadas, dificultad para minimizar simultáneamente las limitaciones difusionales y la caída de presión en los reactores convencionales de lecho fijo y, en ocasiones, por necesitar tiempos de contacto extremadamente cortos. Todo ello hace que las tecnologías catalíticas convencionales trabajen en condiciones no óptimas.

Los sistemas catalíticos estructurados, catalizadores estructurados y reactores de microcanales ofrecen excelentes oportunidades para superar estas limitaciones ya que permiten minimizar simultáneamente las limitaciones difusionales y la caída de presión, mejorar los flujos radiales de calor y materia y permitir tiempos de contacto muy cortos con elevadas eficiencias. Los monolitos de canales paralelos longitudinales, las espumas de porosidad abierta y las mallas metálicas son sustratos que pueden fabricarse a partir de numerosas aleaciones metálicas y con diferentes densidades de celda o poro. También pueden ser recubiertas de cualquier catalizador de interés, adaptándose así a los diferentes requerimientos de cada proceso. Por otro lado, los reactores de microcanales pueden proporcionar una intensificación del proceso sin igual que va acompañada de un excelente control de la temperatura, de la calidad de producto y con mejoras sustanciales en la seguridad del proceso.

El objetivo del proyecto es estudiar sistemas catalíticos estructurados para la producción de energía de origen renovable. En concreto, se estudiarán la síntesis de Fischer-Tropsch, la síntesis directa de dimetiléter y la producción del gas de síntesis que alimentará estos procesos mediante reformado de biogás y "producer gas".Además se estudiará la reacción de desplazamiento del gas de agua que resulta clave para el ajuste de la relación H2/CO en el gas de síntesis.

Se hará especial hincapié en la influencia de las características térmicas de los sistemas estructurados en su comportamiento catalítico. Para ello se estudiará el efecto de la densidad de celdas en monolitos, densidad de poros en espumas, luz de malla en mallas apiladas, tipo de aleación metálica, espesor del recubrimiento catalítico y geometría del sustrato (incluyendo en algunos casos reactores de microcanales).Se considerarán como fases activas catalizadores muy próximos al estado del arte.

El desarrollo de estos estudios se hará con el apoyo de tres tareas transversales lideradas por cada uno de los tres grupos participantes, pero en las que participarán todos ellos: la preparación de los sistemas catalíticos estructurados, la caracterización mediante técnicas avanzadas y los estudios de modelado y simulación. Mediante este proyecto se pretende generar un conocimiento que contribuya a expandir el actual campo de aplicación de los sistemas catalíticos estructurados hacia aplicaciones energéticas sostenibles que se verían beneficiadas por las ventajas que ofrecen estos sistemas en línea con el reto Energía segura, eficiente y limpia.

The dependence of our current energy system on fossil fuels and their harmful effects on the environment are strengthen the development of renewable energy sources. This is the case of the second generation biofuels. The production of fuels from lignocellulosic biomass and wastes very often involve catalytic processes that are characterized by strong heat exchange requirements due to the high thermal effect of the chemical reactions involved, as well as by the difficulty for simultaneously minimizing transport limitations and pressure drop in conventional fixed-bed reactors. Sometimes, extremely short contact times are also required. As a result, the conventional catalytic technologies operate under non-optimal conditions. The structured catalytic systems, structured catalysts and microchannel reactors offer excellent opportunities for overcoming those limitations because they efficiently allow to minimize simultaneously both the transport limitations and pressure drop while improving the radial fluxes of mass and heat and allowing very short contact times. The monoliths with parallel channels, open cell foams and stacked wire meshes can be made of a variety of metallic alloys and cells or pore densities. They can be also coated with any convenient catalyst thus becoming appropriate for the process of interest. On the other hand, the microchannel reactors are capable of providing an incomparable intensification of the process with an excellent temperature control, and improved product quality and process safety. The objective of this project is the investigation of the application of structured catalytic systems for the production of renewable fuels. The reactions investigated will be the Fischer-Tropsch synthesis, the direct dimethyl ether synthesis and the production of the syngas that will be fed to these processes through the reforming of biogas and producer gas. The water-gas shift reaction will be investigated as well due to its important role for adjusting the H2/CO ratio of the syngas. Special attention will be paid to the study of the effect of the thermal properties of the structured systems on their catalytic performance. To this end, the effects of the cells density of monoliths, pore density of foams, mesh of metallic wire meshes, type of metal alloy, thickness of the catalytic coating and substrate geometry (including in some cases microchannel reactors) will be investigated. Catalyst close to the state-of-the-art will be considered as the active phases. The development of these investigations will be supported by three transversal tasks led by each of the three participating research groups but in which all the groups will be involved: preparation of the structured catalytic systems, characterization using advanced techniques and modeling and simulation studies. This proposal aims at generating knowledge that helps to expand the current range of applications of the structured catalytic systems towards the field of sustainable energy applications that will benefit from the advantages of these systems in line with the challenge Safe, efficient and clean energy.

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Desarrollo de catalizadores biomórficos obtenidos a partir de biomasa residual para producción de hidrógeno y refino de bio-oil Development of Biomorphic Catalysts from Residual Biomass for Hydrogen Production and Bio-oil Refining

Código/Code:	ENE2013-47880-C3-2-R
Periodo/Period:	01-01-2014 / 31-12-2017
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	225.060 €
Investigador responsable/Research head:	Miguel Angel Centeno Gallego
Componentes/Research group:	María Isabel Dominguez Leal, Carlos López Cartes, Leidy Marcela Martínez Tejada, Svetlana Ivanova

RESUMEN / ABSTRACT

El objetivo principal del presente proyecto coordinado entre las universidades de Zaragoza y el Instituto de Ciencias de Materiales de Sevilla es el desarrollo de catalizadores metálicos soportados en carbones biomórficos (CB), para su posterior aplicación a procesos de producción de hidrógeno y de refino de bio-oil. La técnica de Mineralización Biomórfica es una innovadora herramienta capaz de sintetizar materiales inorgánicos funcionales utilizando como plantilla diversas estructuras formadas en procesos biológicos. Así, a partir de materiales lignocelulósicos (biomasa) se puede preparar una gran variedad de materiales cerámicos microestructurados. No obstante, la replicación de los distintos niveles jerárquicos existentes en los tejidos biológicos sigue siendo un gran reto a día de hoy. Para avanzar en esta línea, en este proyecto se va a abordar el estudio de la síntesis, caracterización y aplicación de catalizadores metálicos soportados en carbón biomórfico (Me/CB), con distribuciones de tamaño homogénea y porosidad jerarquizada.

La preparación de estos materiales se realiza mediante descomposición térmica en atmósfera reductora (o inerte) a alta temperatura, y elevadas velocidades de calentamiento, de un material lignocelulósico (e.g. celulosa, lignina, papel) impregnado con los precursores metálicos catalíticos. De esta manera, en una sola etapa, se obtiene un soporte carbonoso biomórfico con nanopartículas de metal dispersas en su superficie. Este método de síntesis presenta una extraordinaria versatilidad, puesto que además de poder utilizar diferentes materias primas de partida, se pueden obtener catalizadores de muy distintas composiciones y contenidos metálicos, así como su estructuración en dispositivos monolíticos y espumas. Como materias primas, además de celulosa, lignina o papel, se van estudiar biomasas agrícolas residuales.

Los catalizadores tipo Me/CB se pretenden aplicar en procesos de producción de hidrógeno (descomposición de hidrocarburos ligeros, de amoniaco y deshidrogenación de ácido fórmico), en la reacción de Water-Gas-Shift (WGS), y en distintas reacciones test de refino de bio-oil (conversión de acético a acetona, hidrogenación de vainillina y ciclohexeno y conversión de m-cresol a fenol).

The main goal of this coordinated project among the Universities of Zaragoza and the Institute of Material Science of Seville is the development of supported metal catalysts on biomorphic carbons (CB) for their subsequent application in the hydrogen production and in the refining of bio-oil processes. Biomimetic mineralization is a powerful tool that takes structures formed by a biological process as templates to synthesize inorganic functional materials. It offers the advantage to fabricate materials that are difficult to produce by top-down fabrication methods and that have chemical compositions which cannot be produced by self-assembly. Given that the wood is a multifunctional material that is structured on several levels of hierarchy, a large variety of ceramic microstructured materials can be prepared using lignocellulosic materials (biomass). However, the replication of the different hierarchical levels present in vegetal tissues still remains as great challenge today. In order to get a deeper acknowledgement in this subject, this proposal is going to study the synthesis, characterization and application of metallic catalysts supported on biomophic carbons (Me/CB), prepared with uniform size distributions, and hierarchical porosity.

The preparation of the biomorphic materials will be carried out by thermal decomposition in a reducing (or inert) atmosphere, at high temperature, and high heating rates, of several lignocellulosic components (eg cellulose, lignin, paper) impregnated with catalytic metallic precursors. In this way, in a single step, it is possible to obtain a biomorphic carbonaceous support with the metallic nanoparticles dispersed on its surface. This method of synthesis of catalysts has an outstanding versatility because allows the use of different lignocellulose raw materials, with a large variety of compositions and metal contents. In addition they can be easily structured in monolithic devices or foams. As raw materials, besides cellulose, lignin or paper, it is going to be studied several types of waste agricultural biomass.

The obtained Me/CB catalysts will be applied in hydrogen production processes (light hydrocarbons and ammonia decomposition, dehydrogenation of formic acid), water-gas-shift reaction, and in several reactions test of refining of bio-oil (conversion of acetic in acetone, hydrogenation of vanillin and cyclohexene, and conversion of m-cresol into phenol).

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Desarrollo de procesos catalíticos y fotocatalíticos para la valorización del gas natural: activación y transformación de metano e hidrocarburos ligeros Development of catalytic and photocatalytic processes for natural gas valorization: Activation and transformation of methane and light hydrocarbons

Código/Code:	CTQ2014-60524-R
Periodo/Period:	01-01-2015 / 31-12-2017
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	194.810 €
Investigador responsable/Research head:	Alfonso Caballero Martínez
Componentes/Research group:	Juan Pedro Holgado Vázquez, Gerardo Colon

Ibáñez, Rosa María Pereñiguez Rodríguez, Alberto Rodríguez Gómez

RESUMEN / ABSTRACT

En el presente proyecto se pretenden realizar diversos estudios y desarrollos relacionados con distintos procesos de activación y transformación de metano en moléculas de más valor añadido. Con este fin se estudiarán tanto procesos bien establecidos de conversión indirecta, a través de reacciones de reformado (RM) para la obtención de gas de síntesis, como distintos procesos de conversión directa, en concreto la oxidación directa a metanol (DOM) y la aromatización de metano (DAM).

En lo que respecta a la reacción de reformado, se plantea el desarrollo de sistemas catalíticos con resistencia mejorada a los procesos de desactivación. Para ello se prepararán y caracterizarán nuevos catalizadores bimetálicos nanoestructurados de niquel depositados en soportes como ceria, alumina y alumina/ceria, así como soportes mesoporosos de tipo SBA-15, dopados con ceria y alumina. Como segundo metal se utlizarán cobalto o hierro. Paralelamente, se realizará un estudio de la reacción de reformado por vía fotocatalítica utilizando sistemas de Cu, Pt y Ni depositados en soportes activos clásicos como titania o ceria, así como otros de más reciente desarrollo, como son Ga2O3, nitruro de carbono o grafeno. En este caso, se pretende igualmente explorar las posibilidades de la activación fotoquímica para la reacción de oxidación preferencial de CO (foto-PROX) en presencia de hidrógeno, de utilidad en los procesos de purificación de hidrógeno procedente del gas de síntesis. Se incidirá en la preparación de sistemas con una estructura de de bandas apropiada para el control de esta oxidación selectiva de CO.

En cuanto a los procesos de conversion directa, se estudiará la reacción de DOM usando O2, H2O2 o N2O como activadores de la reacción, en combinación con sistemas basados en Au/Pd, Fe, Cu y/o Ni depositados en soportes como zeolitas ZSM-5, grafeno y TiO2. En este último caso, utilizando Au/Pd como fase metálica activa en presencia de H2O2 como especie oxidante se planteará la posibilidad de combinar la síntesis in situ de agua oxigenada con la posterior oxidación directa de metano. Igualmente, se explorará el proceso de oxidación fotocatalítica de metano a metanol como una alternativa novedosa y altamente atractiva. En este caso, el uso de nuevos fotocatalizadores de oxidación como el BiVO4 así como la presencia de mediadores redox permitirán controlar la oxidación selectiva a metanol.

Algunos sistemas estrechamente relacionados con los anteriores, y en particular los basados en Mo soportados en zeolitas ZSM-5 y MCM-22, se utilizarán para el estudio de la reacción de aromatización de metano. La proporción de aluminio, el porcentaje de molibdeno y su activación en la estructura microporosa del soporte, así como la adición de promotores como Ga, Tl o Pb serán algunas de las variables a optimizar para esta reacción. De forma paralela se podrá estudiar el proceso de aromatización fotoinducido, recientemente descrito por algunos autores.

The present project intends to study and develop different methane activation and transformation processes to obtain high value added molecules.

For this scope we propose to study well established processes of indirect conversion, through reforming reactions (RM) for syngas production, as well as those direct conversion ones, particularly the direct oxidation to methanol (DOM) and aromatization of methane (DAM).

Regarding to the methane reforming reaction, we propose the development of catalytic systems with improved resistance against deactivation processes. In this case, we would prepare and characterize new nanostructured bimetallic catalysts based on nickel supported on ceria,

alumina, or alumina/ceria, as well as mesoporous SBA-15 supports, doped with ceria and alumina. As a second metal we would use cobalt or iron. At the same time, we would perform the study of the reforming reaction by a photocatalytic process using Cu, Pt and Ni doped photoactive systems such as titania or ceria, and others recently proposed as Ga2O3, carbon nitride or graphene. In this case, we propose to explore the possibility of the photochemical activation for the preferential oxidation of CO (photo-PROX) in the presence of hydrogen, a very usefulness process for hydrogen purification from syngas synthesis. We will focus our attention in the preparation of systems with the appropriate band structure for the control of the selective oxidation of CO.

Concerning to direct conversion processes, we would study the direct oxidation of methane (DOM) using O2, H2O2, or N2O as reaction activators, in combination with systems based on Au/Pd, Fe, Cu and/or Ni deposited on different supports as ZSM-5, graphene and TiO2. In this later case, using Au/Pd as the active metallic phase in the presence of H2O2, we propose the possibility to combine the synthesis of H2O2 in situ with the subsequent direct oxidation of methane.

Moreover, we would explore the photocatalytic oxidation of methane to methanol as a novel and highly attractive alternative. In this case, the use of new photocatalytic materials as BiVO4 and the presence of redox mediators would allow us to control the selective photo-oxidation to methanol.

Other catalytic systems closely related to above mentioned, and in particular those based on Mo supported on ZSM-5 and MCM-22 zeolites, would be used for the methane aromatization reaction study. The aluminiun ratio, Mo loading and its activation in the microporous structure of the suport, as well as the addition of certain promoters as Ga, Tl or Pb would be some of the parameters to be optimized for this reaction. At the same time, recently reported photoinduced aromatization process would be studied.

Aprovechamiento CO_2 para la obtención de gas de síntesis en catalizadores CO_2 Utilization for synthesis gas obtaining: Use of structured catalysts

Código/Code:	P11-TEP-8196 (Proyecto de Excelencia)
Periodo/Period:	01-02-2013 / 31-01-2017
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	137.425 €
Investigador responsable/Research head:	Miguel Angel Centeno Gallego
Componentes/Research group:	Svetlana Ivanova, Maria Isabel Domínguez Leal, José Antonio Odriozola Gordón, Tomás Ramírez Reina, Francisca Romero Sarria

RESUMEN / ABSTRACT

Hoy en día no existen dudas acerca de que la concentración de gases de efecto invernadero, en particular la de CO_2, está aumentando de manera considerable en la atmósfera terrestre. Para evitar este aumento continuado se debe aumentar la eficiencia en la producción de

energía, disminuir la intensidad del uso de las fuentes fósiles y, finalmente, potenciar la captura y secuestro del CO_2. Todo esto debe conseguirse manteniendo el crecimiento económico y la calidad de vida. En consecuencia, si tenemos en cuenta el desarrollo de las naciones menos industrializadas y el intensivo consumo energético necesario para aumentar su nivel de vida, la captura y secuestro de CO_2 parece la alternativa más favorable.

En el presente proyecto se propone la utilización del CO_2 como materia prima para el reformado de gas natural como paso previo a la obtención de combustibles líquidos sintéticos. Usando tecnologías convencionales, esta propuesta sólo es económicamente viable asociada a grandes reservas de gas natural. Sin embargo, la tecnología de microcanales permite abordar la síntesis de combustibles sintéticos de forma discontinua y con capacidad de producción flexible de modo económicamente viable. Para ello, es necesario el diseño, caracterización y ensayo de catalizadores activos, selectivos y estables en la reacción de reformado de metano con vapor y CO_2:

$$CO_2 + 3CH_4 + 2H_2O \rightarrow 4CO + 8H_2$$

La selección de estos catalizadores culminará con la estructuración de los mismos utilizando soportes metálicos con microcanales paralelos (micromonolitos) a fin de establecer las condiciones necesarias para, en un futuro, la construcción de reactores de microcanales.

Nowadays, the concentration of greenhouse gases, GHG, in the atmosphere, specifically CO_2, is continuously increasing. In order to avoid or minimize such increment, three different strategies must be applied: i) the improvement of the efficiency on the energy production systems, ii) the lower utilisation of fossil fuels and iii) the implementation of processes of CO_2 capture and sequestration. Since the economic growth and the life quality must be maintained, particularly in the less developed countries, the last item is the most favourable approximation for a sustainable development.

In the present Project, the utilisation of CO_2 as raw material for natural gas reforming is proposed as preliminary step in the production of synthetic liquid fuels.By using conventional technologies, this proposal is economically viable only exploiting natural gas reserves. However, microchanel technology allows the discontinuous production of the synthetic fuels, with a high and flexible production in an economic way. Our project is focussed in the design, characterization and testing of active, selective and stable catalysts in the steam-dry reforming of methane, SDRM:

$$CO_2 + 3CH_4 + 2H_2O \rightarrow 4CO + 8H_2$$

The final step of the project is the structuration of the selected catalysts in metallic micromonoliths with parallel channels as an intermediate step for their future implementation on microchannel reactors.

OTROS PROYECTOS / OTHER PROJECTS

Adquisición de equipo Cromatógrafo de gases con Espectrómetro de Gases (GCMS)

Periodo/Period:	01-01-2016 / 01-12-2017
Oganismo Financiador/Financial source:	Universidad de Sevilla
Código/Code:	UNSE15-CE- 2849
Investigador responsable/Research head:	Alfonso Caballero Martínez

PATENTES / PATENTS

Procedimiento para la preparación de 2-fenil-etanol

Inventores: Juan Pedro Holgado Vázquez, Angeles María López Martín, Alfonso Caballero Martínez
Tipo de Patente: Nacional
Solicitud número: 201630216
Fecha Solicitud: 25 febrero 2016
Entidad Titular: Consejo Superior de Investigaciones Científicas y Universidad de Sevilla

Complejo catalítico conformado por la mezcla de catalizador y conductor iónico

Inventores: José Antonio Odriozola, Francisca Romero Sarria, Svetlana Ivanova, Nuria García Moncada, Miriam González Castaño, Miguel Angel Centeno
Tipo de Patente: Internacional
Solicitud número: PCT/ES16/070412
Fecha Solicitud: 1 junio 2016
Entidad Titular: Consejo Superior de Investigaciones Científicas y Universidad de Sevilla

UNIDAD ASOCIADA / ASSOCIATED UNIT

Grupo de Fotocatálisis y Electroquímica Aplicada al MedioAmbiente
Laboratory of Photocatalysis and Electrochemistry Applied to the Environment

La Junta de Gobierno del CSIC, en su reunión de fecha 22 de diciembre de 2004, aprobó la propuesta de reconocimiento de la Unidad Asociada denominada “Grupo de Fotocatálisis y Electroquímica aplicada al Medio Ambiente” de la Universidad de Palmas de Gran Canaria a través del Instituto de Ciencia de Materiales de Sevilla.

El responsable de esta Unidad Asociada, por parte de la Universidad de Las Palomas de Gran Canaria, fue el Prof. Dr. Jesús Pérez Peña, hasta el 9 de julio de 2014 fecha en que el Presidente del CSIC resuelve aprobar la continuidad de dicha asociación a través del Instituto de Ciencia de Materiales de Sevilla, con el Prof. Dr. José Antonio Navío Santos, Catedrático de la Universidad de Sevilla y el Prof. Dr. Óscar Manuel González Díaz, Profesor Titular de la Universidad de Las Palmas de Gran Canaria, éste último como nuevo responsable de la Unidad Asociada.

Las líneas principales de actuación son:
"Fotocatálisis en procesos ambientales"
"Espectro-electroquímica aplicada al medio ambiente"
"Materiales para tratamientos de aguas residuales"

Entre las acciones propuestas destacar los objetivos de preparación de nuevos materiales fotocatalizadores, así como la caracterización de los mismos y su estudio en aplicaciones de fotocatálisis solar para descontaminación y tratamiento de aguas residuales.

The CSIC's Board of Governors, in its meeting from the 22nd December 2004, passed the proposal to recognize the Associated Unit titled "Laboratory of Photocatalysis and Elec-trochemistry Applied to the Environment", of the University of Las Palmas de Gran Canaria through the Material Science Institute of Seville.

The actual person in charge of the of this Associated is Prof. Dr. Óscar Manuel González Díaz, from the University of Las Palmas de Gran Canaria and Dr. José Antonio Navío Santos from the CSIC's Institute.

The main research lines of working are:
"Photocatalysis for environmental processes"
"Spectroscopical-electrochemistry applied to environment"
"Natural processes for the treatment of waste water"

Among the proposed actions we may emphasize the objectives of preparation of new photocatalysts materials, their characterisation and the study of their applications in solar photocatalysis and treatment of waste water.

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

O-2-assisted Water Gas Shift reaction over structured Au and Pt catalysts

Gonzalez-Castano, M; Reina, TR; Ivanova, S; Tejada, LMM; Centeno, MA; Odriozola, JA
Applied Catalysis B: Enviromental, **185** (2016) 337-343
Mayo, 2016 | **DOI:** 10.1016/j.apcatb.2015.12.032

Platinum and gold structured catalysts were compared as active phases in classical and O2-assisted Water Gas Shift (WGS) reaction. Both metals were supported on iron-doped ceria mixed oxide and then, structured on metallic micromonolithic devices. As expected the WGS activity of both micromonoliths is conditioned by the nature of the noble metals being Pt the most active metal in traditional conditions. However, the addition of oxygen to the classical water gas feed turns the balance in favor of the gold based catalysts, being the presence of gold responsible for an excessive improvement of the catalytic activity.

Cascade charge separation mechanism by ternary heterostructured $BiPO_4/TiO_2/g\text{-}C_3N_4$ photocatalyst

Obregon, S; Zhang, YF; Colon, G
Applied Catalysis B: Environmental, **184** (2016) 96-103
Mayo, 2016 | **DOI:** 10.1016/j.apcatb.2015.11.027

A complex ternary $BiPO_4/TiO_2$/gC(3)N(4) heterostructure has been obtained from a simple impregnation method having good photoactivities for the degradation of phenol under solar-like irradiation. From the wide structural, surface and electronic characterization, we have stated that the formation of the ternary heterojunction notably affect photoactivity of pristine TiO_2. Thus, the best result for the binary system was obtained for 70 wt%TiO_2-30 wt% $BiPO_4$ system. The incorporation of gC(3)N(4) leads to a further improvement on the photocatalytic activity when it is specifically done over TiO_2. By means of photoluminescence spectroscopy and reactive oxygen species formation test, we propose that the effective charge carrier separation is taking place through a cascade-driven electronic mechanism. Therefore, by choosing the adequate band-engineering tailoring an important improved photoactivity can be achieved.

WGS and CO-PrOx reactions using gold promoted copper-ceria catalysts: "Bulk $CuO\text{-}CeO_2$ vs. $CuO\text{-}CeO_2/Al_2O_3$ with low mixed oxide content"

Reina, TR; Ivanova, S; Laguna, OH; Centeno, MA; Odriozola, JA
Applied Catalysis B: Environmental, **197** (2016) 62-72
Noviembre, 2016 | **DOI:** 10.1016/j.apcatb.2016.03.022

A copper-ceria bulk catalyst has been compared to a series of catalysts designed according to the as called "supported approach", corresponding to the dispersion of low content mixed copper-ceria oxide on alumina matrix. The principal characteristics of both types of catalysts are contemplated and the differences in their electronic and redox properties discussed in details. As a plus, the gold metal promotion of the catalysts is also envisaged. The advantages of the systems in the CO clean up reactions, WGS and CO-PrOx are commented. While the WGS activity appears to be ruled especially by the Cu/Ce surface to volume ratio, the CO-PrOx reaction is governed by the CuO loading. Gold addition provides benefits only at the low temperature WGS regime. Very importantly, the supported systems are always superior to the bulk configuration in terms of specific activity, a key factor from the catalyst's design perspective.

The role of Au, Cu & CeO_2 and their interactions for an enhanced WGS performance

Reina, TR; Ivanova, S; Centeno, MA; Odriozola, JA
Applied Catalysis B: Environmental, **187** (2016) 98-107
Junio, 2016 | **DOI:** 10.1016/j.apcatb.2016.01.031

The WGS reaction over multicomponent $Au/Ce_{1-x}Cu_xO_2/Al_2O_3$ catalysts is studied in this work. The systems are carefully designed aiming to take advantage of every active phase included in the formulation: gold, ceria and copper. Special emphasis is given to the CeO_2-CuO synergy and its influence on the displayed catalytic performance with and without gold. To this aim a meaningful correlation between the physicochemical properties of the mixed materials and their activity/stability is proposed. In general terms the developed catalysts present high activity under realistic WGS reaction conditions, with fairly good long term stability. In addition, the systems successfully withstand start-up/shut-downs situations, indispensable requisite for real applications in the field of pure hydrogen production for fuel cell goals.

Photocatalytic removal of patent blue V dye on Au-TiO_2 and Pt-TiO_2 catalysts

Vaiano, V; Iervolino, G; Sannino, D; Murcia, JJ; Hidalgo, MC; Ciambelli, P; Navio, JA
Applied Catalysis B: Environmental, **188** (2016) 134-146
Julio, 2016 | **DOI:** 10.1016/j.apcatb.2016.02.001

In this work it was studied the efficiency of a photocatalytic process for the removal of patent blue V. This dye is very difficult to remove by conventional treatments such as adsorption or coagulation therefore the photocatalytic process is a very interesting alternative for the removal this dye mainly because it does not require expensive oxidants and it can be carried out at mild temperatures and pressures. In this work it was tested the efficiency of Au-TiO_2 and Pt-TiO_2 photocatalysts in the Patent blue V removal. The Au-TiO_2 catalysts were prepared by two different methods: chemical reduction and photochemical deposition; Pt-TiO_2 catalysts were obtained only by photochemical deposition. In the synthesis of the catalysts prepared by photochemical deposition, it was evaluated the influence of some parameters, such as deposition time and the intensity of the light source over the physicochemical properties and photocatalytic activity of the materials obtained. An analysis of the effect of the catalyst dosage and initial patent blue V concentration over the dye degradation efficiency was also attempted. In general, it was observed that the presence of Au or Pt on TiO_2 enhances the patent blue V photodegradation; it was found that noble metal particle size and distribution on TiO_2 surface are important factors influencing the dye removal. The highest dye degradation was obtained over the Au-TiO_2 catalyst prepared by photochemical deposition, using high light intensity and 15 min of deposition time during the synthesis. A discoloration and a total organic carbon (TOC) removal of 93 and 67% respectively, were obtained over this material after 180 min of UV irradiation. These values are higher than that the obtained on S-TiO_2 (discoloration and TOC removal of about 25% and 3%, respectively).

Photocatalytic hydrogen production from degradation of glucose over fluorinated and platinized TiO_2 catalysts

Iervolino, G; Vaiano, V; Murcia, JJ; Rizzo, L; Ventre, G; Pepe, G; Campiglia, P; Hidalgo, MC; Navio, JA; Sannino, D
Journal of Catalysis, **339** (2016) 47-56
Julio, 2016 | **DOI:** 10.1016/j.jcat.2016.03.032

The present work reports the renewable hydrogen production by photocatalytic degradation of glucose over commercial and home prepared TiO_2 modified by the simultaneous presence of fluorine and Pt (Pt-F-TiO2). The obtained materials were widely characterized by different techniques (XRD, SBF, UV-Vis DRS, XRF, SEM and TEM) and it was found that surface area, anatase/rutile ratio and the distribution and size of the platinum particles are important factors influencing the effectiveness of these materials in the H-2 production. The photocatalytic H-2 production from the glucose solution was 97 mu mol of H-2 after 3 h of irradiation on home prepared TiO_2 modified by F and Pt addition, while a lower value corresponding to 31 mu mol of H-2 was obtained on commercial TiO_2 modified by F and Pt, after 3 h of irradiation. The hydrogen production rate increased by decreasing the initial pH of solution reaching the highest value of about 590 mu mol h(-1) g(-1) after 3 h of irradiation time at pH = 2. Accordingly, sugar containing wastewaters from food industry has the potential for producing hydrogen by photocatalytic process while removing organics before disposal or reuse.

Forced deactivation and postmortem characterization of a metallic microchannel reactor employed for the preferential oxidation of CO (PROX)

Laguna, OH; Dominguez, MI; Centeno, MA; Odriozola, JA
Chemical Engineering Journal, **302** (2016) 650-662
Octubre, 2016 | **DOI:** 10.1016/j.cej.2016.05.104

This manuscript is one of the few works presenting evidences of the effect of prolonged use of a microreactor. Our reactor has been designed for the PROX reaction. Near to 550 h of operation under different feed-streams, including CO_2 and H_2O, in the 100-300 degrees C temperature range, and several regeneration cycles, and a final forced deactivation during similar to 360 h resulted in the permanent loss of activity of the microreactor. This could be attributed to some phenomena whose have compromised the chemical nature of the catalyst and that of the reactor including: displacement of the coating to the mouth of the channels, detachments and cracks of the catalytic layer, migration of some elements of the metallic substrate to the surface (Fe, Cr, Y), and deposition of carbonaceous species from the reaction over the catalytic layer and/or the metallic substrate. Furthermore, sulfur compounds were detected in both inlet and outlet zones of the microreactor, coming probably from a lubricant applied over the screws that sealed the assembling of the microreactor.

This is a first approach for understanding possible effects of deactivation during long-term applications of a microreactor in the PROX reaction that could be considered as a case study useful for future designs of this kind of devices. The presented information could be extrapolated to similar reactions where thermal treatments along with highly corrosive atmospheres would be applied, in order to carry out a more appropriate design of future generations" of microreactors, with a longer useful life. For that purpose not only the adequate selection of the catalysts must be done, but also the adequate choice of the fabrication material of the reactors is needed.

Intensifying glycerol steam reforming on a monolith catalyst: A reaction kinetic model

Bobadilla, LF; Blay, V; Alvarez, A; Dominguez, MI; Romero-Sarria, F; Centeno, MA; Odriozola, JA
Chemical Engineering Journal, **306** (2016) 933-941
Diciembre, 2016 | DOI: 10.1016/j.cej.2016.08.021

In this work, a structured monolithic catalyst has been tested under a wide range of conditions (partial pressure, residence time, temperature and time-on-stream), with the aim of modeling its kinetic behavior and assessing its economic and upscaling potential. We have developed a sequential model to help us interpret both main trends and salient features. Unexpected behavior was found for certain parameter values, which led us to consider kinetic parasitic effects such as mass or heat transfer limitations. By independently invoking these effects, a conciliatory view of the results observed could not be reached. A combined explanation may prove successful, although overfitting could not be ruled out at this point. More importantly, however, the observed salient features of this stable and selective monolith catalyst may hold potential for process intensification of glycerol steam reforming, thus contributing to a more sustainable industry.

Au-supported on Fe-doped ceria solids prepared in water-in-oil microemulsions: Catalysts for CO oxidation

Laguna, OH; Centeno, MA; Boutonnet, M; Odriozola, JA
Catalysis Today, **278** (2016) 140-149
Diciembre, 2016 | **DOI:** 10.1016/j.cattod.2016.05.059

Gold catalysts were synthesized by deposition-precipitation employing Fe-doped ceria systems, previously obtained by means of the water-in-oil microemulsions methodology with different iron contents (10, 25 and 50 Fe at.%). The final catalysts were tested in the CO oxidation reaction in presence of H2. After gold deposition the crystalline structure of the supports was not altered. Moreover no XRD lines associated to gold were detected, indicating its high dispersion. Solid solution was generated in all samples, although the segregation of iron oxide was detected for the material with the highest iron loading. This phenomenon was then enhanced for the corresponding gold catalyst that also presented sintering of the gold nanoparticles.
Strong interaction between gold and the oxygen vacancies of the supports was demonstrated, as well as the promotion of the reducibility of surface Ce4+ and Fe3+species at low temperatures. A remarkable promotion of the CO conversion at lower temperatures respect to that of the supports was observed for the gold catalysts. Below 120 °C, lower the amount of iron incorporated, higher the catalytic performance of the catalyst. This behaviour is closely related not only to a high gold dispersion but also to the ability for creating additional oxygen vacancies in the support, required for the CO oxidation reaction.

Growth of carbonaceous nanomaterials over stainless steel foams. Effect of activation temperature

Latorre, N; Cazana, F; Sebastian, V; Royo, C; Romeo, E; Centeno, MA; Monzon, A
Catalysis Today, **273** (2016) 41-49
Septiembre, 2016 | **DOI:** 10.1016/j.cattod.2016.02.063

Some of the problems that occur during the operation of chemical reactors based of structured catalytic substrates, as monoliths, foams, membranes, cloths, fibres and other systems, are related to the preparation of long term stable coatings. Frequently, the deposition of the catalytic layer is carried out by washcoating, requiring this step a cautious attention, especially in the case of complex geometries, like of that of foams or cloths. In the case of the deposition of layers of carbonaceous materials (CNMs), an alternative route, avoiding the washcoating, it is their direct growth by catalytic decomposition light hydrocarbons (also called CCVD), over the surface of the metallic substrate. In this case, if the metallic substrate is of stainless steel, it already contains the catalytic active phases like Fe and Ni.
In order to optimize the process of CNMs growth over structured metallic substrates, we are studying the effect of the main operational variables of the ethane decomposition reaction on stainless steel foams. In this contribution we present a study of the influence of the temperature of the activation (oxidation and reduction) stage on the type and morphology of the

carbonaceous materials formed. The results obtained allow us to determine the optimal operating conditions to maximize the amount and the selectivity of the process to obtain a given type of CNM.

Influence of the ionic liquid presence on the selective oxidation of glucose over molybdenum based catalysts

Megias-Sayago, C; Carrasco, CJ; Ivanova, S; Montilla, FJ; Galindo, A; Odriozola, JA
Catalysis Today, **278** (2016) 82-90
Diciembre, 2016 | DOI: 10.1016/j.cattod.2016.06.040

Two different approaches are proposed in this work in order to study the influence of the ionic liquid presence in the reaction of glucose oxidation by H2O2 in mild conditions. The ionic liquids are applied either as a solvent by using homogeneous Mo based catalyst, [Mo(O)(O2)2(H2O)n] complex, or by using it as an integral part of a heterogeneous catalyst, organic inorganic hybrids based on Mo Keggin structure. Both catalytic strategies resulted in acceptable glucose transformation degrees but lead to different oxidation products depending on the role of the ionic liquid. The hybrid approach restrains the number of the received products being the most selective one. A detailed study of the effect of the hybrid nature and reaction conditions is proposed in the second part of this study.

Selectivity control in oxidation of 1-tetradecanol on supported nano Au catalysts

Martinez-Gonzalez, S; Ivanova, S; Dominguez, MI; Corberan, VC
Catalysis Today, **278** (2016) 113-119
Diciembre, 2016 | **DOI:** 10.1016/j.cattod.2016.06.019

Selective oxidation of tetradecanol, a model higher fatty alcohol, on Au/CeO_2-Al_2O_3 catalyst has been investigated to assess the factors that control selectivity. The analysis of the effect of operation conditions (temperature, run time and alcohol/metal (AIM) ratio) on catalytic performance revealed a quite complex reaction network, in which acid formation starts only after a certain level of conversion is reached. This level depends linearly on the total support surface available, indicating that it must be saturated by species generated by the reaction itself to allow acid formation to start. Addition of water to reaction medium did not modify this level, indicating that such species is not adsorbed water, as previously hypothesized, but probably spilled over hydrogen species. The resulting drastic change in the selectivity trends makes the ratio A/M a critical factor to control selectivity to aldehyde and to acid. Selectivity to ester is less sensible to operation parameters. It is noteworthy that aldehyde yields up to 27% with 90% selectivity, and acid yields up to 40% with 81% selectivity can be reached by proper selection of operation parameters.

Photocatalytic Escherichia coli inactivation by means of trivalent Er^{3+}, Y^{3+} doping of $BiVO_4$ system

Adan, C; Marugan, J; Obregon, S; Colon, G
Applied Catalysis A: General, **526** (2016) 126-131
Septiembre, 2016 | **DOI:** 1010110.1016/j.apcata.2016.08.002

$BiVO_4$ samples doped with different contents of Er^{3+} and Y^{3+} were prepared by a simple surfactant free hydrothermal method. X-ray diffraction reveals that the doped materials consist of a heterogeneous structure formed by a mixture of tetragonal and monoclinic phases, being found Er^{3+} and Y^{3+} co-doping clearly stabilize the tetragonal structure of $BiVO_4$. The monoclinic $BiVO_4$ samples shows a strong absorption in the visible light region leading to band-gap values of around 2.4eV while the tetragonal $BiVO_4$ displays higher band-gap values of 2.9 eV. The photocatalytic activity of the catalysts was investigated for the oxidation of methanol and inactivation of Escherichia coli showing that all the $BiVO_4$ catalysts are photocatalytically active in the oxidation of methanol and are able to inactivate more than 99.99% of bacteria not only under UV light but also under visible light irradiation. The results revealed that the co-doping of Er^{3+} and Y^{3+} into $BiVO_4$ exhibited enhanced photocatalytic activity for methanol oxidation under simulated solar light irradiation. The inactivation of E.coli show similar results for the doped systems although in relative terms of activity the Er^{3+},Y^{3+}-$BiVO_4$ sample show a better use of the visible light, leading to a higher activity than P25-TiO_2.

Towards the hydrogen production by photocatalysis

Colon, G

Applied Catalysis A: General, **518** (2016) 48-59

Mayo, 2016 | **DOI:** 10.1016/j.apcata.2015.11.042

Nowadays, problems derived from climate change urgently demand us to focus our attention on new alternatives to fossil fuels. Within this framework, the photocatalytic production of hydrogen as a clean fuel from oxygenates arises as a necessary option that must be considered. Thus, the development of highly efficient photocatalyst is crucial in order to achieve a viable technology under the industrial point of view. For this sake, it is necessary to understand the principles of photoreforming reaction. In this brief review we will revisit the different photocatalytic materials proposed in the literature highlighting on the role of different co-catalysts.

Impact of structured catalysts in amine oxidation under mild conditions

J.L. Santos; P. Navarro; J.A. Odriozola; M.A. Centeno; O.D. Pavel; B. Jurca; V.I. Pàrvulescu

Catalysis Today, **273** (2016) 266-272

Septiembre, 2016 | **DOI:** 10.1016/j.cattod.2016.05.001

A structured graphene/graphite catalyst grown on a commercial austenitic stainless steel sheet providing a micromonolith was obtained by submitting the nude stainless steel structure to a carbon-rich atmosphere (first 300 mL/min of a reductive H-2/N-2 (1:1) flow, then to 180 mL/min of a CH4/H-2 (1:5)) at high temperature (900 degrees C) for 2 h. The preparation procedure resulted in a homogenous surface coated with a carbon-rich film as observed by EDX and SEM images. Further characterizations by Raman spectroscopy revealed characteristic Raman lines of graphene and crystalline graphite disposed in a hierarchical organization. The disposal of the obtained surface layers was also confirmed by grazing incidence X-ray diffraction. Besides this, XRD indicated the overlapping diffraction lines of graphite, cementite and M7C3 carbides. The graphene nature of the outermost layer was also confirmed by XPS. The catalytic behavior of the structured graphene/graphite catalyst was evaluated in the selective oxidation of heptylamine. At 200 degrees C it afforded a total conversion with a combined selectivity in heptanonitrile and N-heptylidene-heptylamine of 67% (10% heptanonitrile) that corresponds indeed to a very efficient system in the absence of any metal. Kinetic experiments with the scope to calculate the activation energies were also performed.

In-situ hydrogasification/regeneration of NiAl-hydrotalcite derived catalyst in the reaction of CO_2 reforming of methane: A versatile approach to catalyst recycling

Abdelsadek, Z; Sehailia, M; Halliche, D; Gonzalez-Delacruz, VM; Holgado, JP; Bachari, K; Caballero, A; Cherifi, O
Journal of CO2 Utilization, **14** (2016) 98-105
Junio, 2016 | **DOI:** 10.1016/j.jcou.2016.03.004

A novel approach describing the in-situ regeneration of NiAl hydroalcite derived catalyst between two cycle reaction systems of CO2 reforming of methane, also known as dry reforming of methane (DRM) is described herein. The catalyst was initially prepared by co-precipitation method at pH = 11 and calcined at 450 degrees C for 6 h. The obtained material was characterized using X-ray diffraction (XRD) spectroscopy, Fourier transform infrared (FTIR) spectroscopy, Brunauer-Emmett-Teller (BET), atomic absorption spectroscopy (AAS), scanning electron microscopy (SEM), transmission electron microscopy (TEM), thermogravimetry (TG/ATD) and temperature programmed reduction (TPR-H-2) techniques. Following treatment of our catalyst under DRM conditions, the catalyst was subjected to in-situ hydrogasification conditions to promote regeneration followed by a second DRM cycle. An increase of 15.7% in the conversion of CH4 and 17.3% in the conversion of CO2 was attained, while the ratio of resulting H-2/CO augmented by 14%. The ratio of H-2 consumed over the course of two hours hydrogasification, to that generated over ten hours of DRM, was 9.6%. The small particle sizes of resulting Ni degrees species as well as their high stability were both key factors contributing to the increase in the amount of H-2/CO produced prior to and after regeneration.

Nanogold mesoporous iron promoted ceria catalysts for total and preferential CO oxidation reactions

Reina, TR; Ivanova, S; Idakiev, V; Tabakova, T; Centeno, MA; Deng, QF; Yuan, ZY; Odriozola, JA
Journal of Molecular Catalysis A-Chemical, **414** (2016) 62-71
Abril, 2016 | **DOI:** 10.1016/j.molcata.2016.01.003

Herein, a series of highly efficient gold based catalysts supported on mesoporous CeO_2-Fe_2O_3 mixed oxides for CO elimination reactions have been developed. The materials have been fully characterized by means of XRD, Raman and UV-vis spectroscopies among other techniques. We identify the Ce-Fe synergism as a fundamental factor controlling the catalytic performance. Our data clearly reveal that the CO oxidation activity is maximized when the electronic and structural properties of the support are carefully controlled. In this situation, fairly good catalysts for environmental applications as for example H-2 streams purification for fuel cell goals or CO abatement at room temperature can be designed.

Comparison of supported TiO_2 catalysts in the photocatalytic degradation of NOx

Rodriguez, MJH; Melian, EP; Diaz, OG; Arana, J; Macias, M; Orive, AG; Rodriguez, JMD
Journal of Molecular Catalysis A-Chemical, **413** (2016) 56-66
Marzo, 2016 | **DOI:** 10.1016/j.molcata.2015.12.007

A comparison is made in this study of the effectiveness of various commercial catalysts in the oxidation of NO_x by heterogeneous photocatalysis. The following catalysts were considered: Aeroxide TiO_2 P25, Aeroxide TiO_2 P90, Hombikat UV-100, Kronos vlp7000, CristalACTIV PC105, CristalACTIV PC500, Kemira 650 and Anatasa Aldrich. All catalysts were deposited by a dip - coating technique onto borosilicate 3.3 glass plates. Optimization of catalyst load showed no significant enhancement of photoactivity, in general, above a deposited mass of 1.16 mg cm(-2). Differences between photocatalyst activity were more apparent at longer illumination times. Photoactivity decreased in the presence of humidity and differences in the adsorbed products were detected. Photocatalyst activity was strongly influenced by specific surface area, with the best results obtained by the catalysts with the largest surface area, namely the PC500, Hombikat and Kronos. Photocatalyst stability was demonstrated in successive reuse cycles.

TiO_2-clay based nanoarchitectures for enhanced photocatalytic hydrogen production

Perez-Carvajal, J; Aranda, P; Obregon, S; Colon, G; Ruiz-Hitzky, E
Microporous and Mesoporous Materials, **202** (2016) 120-127
Marzo, 2016 | **DOI:** 10.1016/j.micromeso.2015.10.007

New functional TiO_2-clay nanoarchitectures based on layered and fibrous silicates and incorporating Pd and Pt noble metal nanoparticles (NPs) have been synthesized by applying a sol–gel methodology that involves the use of commercial organoclays. The incorporation of the noble metal NPs can be done using two different approaches: i) direct addition to the synthesis medium of a noble metal precursor (typically acetylacetonate) during the generation of the nanoarchitecture, and ii) selective photodeposition of the noble metal NPs in a post-treatment of the TiO2-clay nanoarchitecture. The resulting materials have been characterized by means of XRD, FTIR, Raman, 29Si-NMR, FE-SEM, TEM and N_2 adsorption–desorption isotherms. The efficiency of these nanoarchitectures in the photocatalytic hydrogen production has been tested in the photoreforming of methanol. The higher rate in the hydrogen production corresponds to the nanoarchitectures containing Pt and TiO_2 NPs derived from sepiolite.

Intensification of hydrogen production by methanol steam reforming

O. Sanz, I. Velasco, I. Pérez-Miqueo, R. Poyato, J.A. Odriozola, M. Montes
International Journal of Hydrogen Energy, **41** (2016) 5250-5259
Marzo, 2016 | **DOI:** 10.1016/j.ijhydene.2016.01.084

This paper studies the methanol steam reforming intensification to enhance the hydrogen production in a multi-channel block type micro-reformer. The effects of operating parameters such as reforming temperature, space velocity and catalyst layer thickness on reforming performance are investigated. For optimized design and operating conditions, the 8 cm3 reformer unit produced 170 LH2/h containing on dry basis 75.0% H2, 23.5% CO2, 0.06% CH3OH and 1.44% CO at 648 K allowing the production of 218–255 W in a commercial PEMFC with 80% hydrogen utilization. This study shows that high methanol conversion can be achieved with high Pd/ZnO catalyst loading at 648 K with very low CO content (<1.5%) in the outlet stream.

Promoting effect of Sn on supported Ni catalyst during steam reforming of glycerol

Bobadilla, L. F.; Romero-Sarria, F.; Centeno, M. A.; Odriozola, J. A.
International Journal of Hydrogen Energy, **41** (2016) 8234-8244
Junio, 2016 | **DOI:** 10.1016/j.ijhydene.2016.04.119

The promoting effect of Sn on the catalytic performance of supported Ni catalyst in the reaction of glycerol steam reforming was studied. The physico-chemical properties of the prepared samples were investigated by X-ray fluorescence (XRF), BET surface area, in situ X-ray diffraction (XRD), laser Raman spectroscopy, X-ray photoelectron spectroscopy (XPS), scanning electron microscopy (SEM) and temperature-programmed oxidation (TPO) techniques. The characterization results of the samples after reduction treatment (in the same conditions than the activation before catalytic activity measurements) revealed the formation of NiSn alloy. The Sn-doped catalyst exhibited a high activity and it was demonstrated that the Sn addition increase the catalyst stability and durability by decreasing the coke deposition.

Nickel catalyst with outstanding activity in the DRM reaction prepared by high temperature calcination treatment

Smolakova, L; Kout, M; Capek, L; Rodriguez-Gomez, A; Gonzalez-Delacruz, VM; Hromadko, L; Caballero, A
International Journal of Hydrogen Energy, **41** (2016) 8459-8469
Junio, 2016 | **DOI:** 10.1016/j.ijhydene.2016.03.161

The catalytic performance of some Ni-Ce/Al_2O_3 catalytic systems (11 wt.% Ni and 3 wt.% Ce) were checked after being submitted to different calcination and reduction treatments. It was found that, the reduced Ni-Ce/Al_2O_3 catalysts were more active and stable in the dry reforming reaction of methane than thecorresponding not-reduced catalysts. This high activity was initially connected with the smaller size of pre-reduced Ni species, which at the same time leads on to the formation of filamentous carbon. The best overall performance was obtained for the reduced catalyst after being calcined at 1000 degrees C. This catalyst presents a very high stability, a low level of carbon formation, maintaining the nickel particle size constant during reaction. Surprisingly, although its reduction degree is only 12% at 750 degrees C, its catalytic activity is similar to the full reduced catalysts. So, the small number of reduced metallic particles of this catalyst shows a very high activity, much higher than the other catalysts.

Au/CeO_2 Catalysts: Structure and CO Oxidation Activity

Centeno, MA; Reina, TR; Ivanova, S; Laguna, OH; Odriozola, JA
Catalysis, **6** (2016) 158
Octubre, 2016 | DOI: 10.3390/catal6100158

In this comprehensive review, the main aspects of using Au/CeO_2 catalysts in oxidation reactions are considered. The influence of the preparation methods and synthetic parameters, as well as the characteristics of the ceria support (presence of doping cations, oxygen vacancies concentration, surface area, redox properties, etc.) in the dispersion and chemical state of gold are revised. The proposed review provides a detailed analysis of the literature data concerning the state of the art and the applications of gold–ceria systems in oxidation reactions.

Recycling of construction and demolition waste generated by building infrastructure for the production of glassy materials

Dominguez, A; Dominguez, MI; Ivanova, S; Centeno, MA; Odriozola, JA
Ceramics International, **42** (2016) 17217-175223
Noviembre, 2016 | **DOI:** 10.1016/j.ceramint.2016.06.157

The use of waste materials generated by construction and demolition industry to yield valuable glassy materials, i.e. enamel for glazed ceramic tiles and cellular glasses is presented in this study. Both types of materials are produced by one-step treatment at moderate temperatures after simple waste chemical composition adjust. The enamels are manufactured directly from the initial waste powder by melting, while the expanded materials result from mixing of the vitreous material obtained after waste vitrification with an adequate foaming agent and posterior thermal treatment. Through the manuscript the feasibility of one step production of second generation profit materials is discussed in order to help achieving sustainable development and environmental protection.

In-situ Raman spectroscopy study of Ru/TiO_2 catalyst in the selective methanation of CO

Martinez Tejada, LM; Munoz, A; Centeno, MA; Odriozola, JA

Journal of Raman Spectroscopy, **47** (2016) 189-197

Febrero, 2016 | **DOI:** 10.1002/jrs.4774

Raman spectroscopic technique has been used to characterize a Ru/TiO_2 catalyst and to follow in situ their structural changes during the CO selective methanation reaction (S-MET). For a better comprehension of the catalytic mechanism, the in-situ Raman study of the catalysts activation (reduction) process, the isolated CO and CO_2 methanation reactions and the effect of the composition of the reactive stream (H_2O and CO_2 presence) have been carried out. Raman spectroscopy evidences that the catalyst is composed by islands of TiO_2–RuO_2 solid solutions, constituting Ru–TiO_2 interphases in the form of $Ru_xTi_{1-x}O_2$ rutile type solid solutions. The activation procedure with H_2 at 300 °C promotes the reduction of the RuO_2–TiO_2 islands generating Ru^o–Ti^{3+} centers. The spectroscopic changes are in agreement with the strong increase in chemical reactivity as increasing the carbonaceous intermediates observed. The selective methanation of CO proceeds after their adsorption on these Ru^o–Ti^{3+} active centers and subsequent C—O dissociation throughout the formation of $CH_x/C_nH_x/C_nH_xO/CH_x$—CO species. These intermediates are transformed into CH_4 by a combination of hydrogenation reactions. The formation of carbonaceous species during the methanation of CO and CO_2 suggests that the CO presence is required to promote the CO_2 methanation. Similar carbonaceous species are detected when the selective CO methanation is carried out with water in the stream. However, the activation of the catalysts occurs at much lower temperatures, and the carbon oxidation is favored by the oxidative effect of water.

Ru-Ni Catalyst in the Combined Dry-Steam Reforming of Methane: The Importance in the Metal Order Addition

Alvarez, MA; Centeno, MA; Odriozola, JA

Topics in Catalysis, **59** (2016) 303-313

Febrero, 2016 | **DOI:** 10.1007/s11244-015-0426-5

Biogas is one of the main biomass-energy resources. Its use for syngas production with a H-2/CO ratio close to two would have huge environmental, social and economic impact in the actual energetic scenario. However, the use of dry reforming, where the two main components are transformed into syngas, does not allow the desired H-2/CO ratio. For this reason, the addition of water is proposed. The process was performed with two Ru-Ni catalysts where the metal order in the impregnation process was varied. The catalysts were prepared either by simultaneous or consecutive impregnation of the active phases and its catalytic performance in the combined dry-steam reforming of methane was tested. The catalysts were characterized by XRF, XRD, S-BET, TPR-H-2 and Raman spectroscopy. The existence of a strong Ni-Ru interaction is evidenced by Raman spectroscopy and TPR-H-2 in the sample synthesized by the simultaneous impregnation. Concerning the catalytic activity, this sample presents the highest CH_4 and CO_2 conversion values in the entire composition rate and the lowest amount of carbon deposits after reaction. After pulse, and reactivity tests it was concluded that the higher Ni-Ru interaction displayed by the catalyst synthesized by the simultaneous impregnation, enhances the carbon gasification.

The role of carbon overlayers on Pt-based catalysts for H-2-cleanup by CO-PROX

Romero-Sarria, F; Garcia-Dali, S; Palma, S; Jimenez-Barrera, EM; Oliviero, L; Bazin, P; Odriozola, JA

Surface Science, **648** (2016) 84-91

Junio, 2016 | **DOI:** 110.1016/j.susc.2015.12.017

In this work, we analyze the effect of the activation method on the catalytic activity of Pt-based catalysts supported on alumina in the PROX reaction. For this, model Pt/Al2O3 catalysts with variable amounts of acetic acid were prepared and their thermal evolution studied by FTIR spectroscopy. From the analysis of the nature of the platinum surface upon acetic acid decomposition and the gas phase evolved products, we have demonstrated the formation of partially hydrogenated carbon overlayers that tailor the activity of Pt-based catalysts in the PROX reaction.

Liquid-phase oxidation with hydrogen peroxide of benzyl alcohol and xylenes on Ca-10(PO4)(6)(OH)(2) - CaWO4

Dominguez, MI; Cojocaru, B; Tudorache, M; Odriozola, JA; Centeno, MA; Parvulescu, VI

Comptes Rendus Chimie, **19** (2016) 1156-1165

Octubre, 2016 | **DOI:** 10.1016/j.crci.2015.10.013

A W-containing apatite (W/HAp) catalyst was prepared following a hydrothermal synthesis route and served as a model catalyst. Crystallographic analysis indicated that the resulting material contained hydroxyapatite, Ca10-3xWx(PO4)(6)(OH)(2), W-hydroxyapatite, calcium tungstate, CaWO4, and tricalcium phosphate, Ca-3(PO4)(2). The catalyst was investigated in liquid phase oxidation of benzyl alcohol and xylenes using hydrogen peroxide as an oxidant. For comparison, commercial calcium phosphate, hydroxyapatite and CaWO4 were tested in the same reaction. Calcium phosphate and hydroxyapatite appeared as inactive and decomposed hydrogen peroxide non-selectively. A moderate activity but low hydrogen peroxide efficiency was observed for the CaWO4 phase. In contrast, the W/HAp catalyst showed a reasonable activity and a better hydrogen peroxide efficiency in the oxidation of benzyl alcohol and xylenes. This new W/HAp catalyst showed, after six cycles, losses of the activity below 15% compared to the fresh catalyst with no effect on the selectivity. It is noteworthy that ICP-OES analyses showed no tungsten leaching that is the main advantage of this catalyst.

LIBROS Y OTRAS PUBLICACIONES / BOOKS AND OTHER PUBLICATIONS

O.H. Laguna, M.I. Domínguez, M.A. Centeno, J.A. Odriozola
“**Catalysts on metallic surfaces: monoliths and microreactors**”, en: New Materials for Catalytic Applications, Editorial Elsevier, Estados Unidos (2016) pags. 81-120
ISBN: 978-0-444-63587-7

T.R. Reina, J.L. Santos, N. García-Moncada, S. Ivanova, J.A. Odriozola
“**Development of robust mixed-conducting membranes with high permeability and stability**”, en: Perovskites and Related Mixed Oxides Concepts and Applications, Editorial Wiley-VCH, Alemania (2016) pags. 719-737
ISBN: 978-3-527-33763-7

O.H. Laguna, L.F. Bobadilla, W.Y. Hernández, M.A. Centeno
“**Low temperature CO oxidation**”, en: Perovskites and Related Mixed Oxides Concepts and Applications, Editorial Wiley-VCH, Alemania (2016) pags. 451-473
ISBN: 978-3-527-33763-7

Colón Ibañez, G.; Senanayake, S.; Kubacka, A.
“**Hydrogen from oxygenated molecules**”
Applied Catalysis A: General, 518 (2016) page 1 (Editorial)
DOI: 10.1016/j.apcata.2016.03.003

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

PARTICIPACIÓN EN LA ORGANIZACIÓN DE CONGRESOS Y REUNIONES / PARTICIPATION IN ORGANISING AND MEETINGS

5th International Conference on Structured Catalysts and Reactors ICOSCAR-5
22 – 24 junio [San Sebastián, España]
José Antonio Odriozola [Miembro del Comité Organizador)
Miguel Angel Centeno [Miembro del Comité Organizador)
Francisca Romero Sarria [Miembro del Comité Organizador)
M. Isabel Leal Domínguez [Miembro del Comité Organizador)

II Encuentro de Jóvenes Investigadores de la SECAT
27 – 29 junio [Ciudad Real, España]
Elena Jiménez Barrera [Miembro del Comité Organizador)

Challenges and prospects for solid state chemistry CPSSC16

9 septiembre – 10 septiembre [Sevilla, España]
Alfonso Caballero Martínez [Miembro del Comité Organizador)
José Antonio Odriozola Gordón [Miembro del Comité Organizador)

COMUNICACIONES / COMMUNICATIONS

4th Latin American Congress on Photocatalysis. Photoelectrochemistry and Photobiology. 4th LACP3

25 – 30 abril [Bucaramanga, Colombia]

Phenol and methyl orange photodegradation over dye-sensitized TiO_2
J.J. Murcia, E.G. Avila, H.A. Rojas, S. Ivanova, A. Penkova, O.H. Laguna
Poster

French Conference on Catalysis, FCCAT1

23 – 27 mayo [Frejus, Francia]

Direct catalytic conversion of glucose to lactic acid
C. Megías-Sayago, S. Ivanova, M.A. Centeno, J.A. Odriozola
Poster

7th International Symposium on Catalysis on Carbon

12 – 16 junio [Estrasburgo, Francia]

Carbon-based biomorphic catalyst for Water Gas Shift reaction
J.L. Santos, F. Cazaña, P. Tarifa, A. Monzón, C. López-Cartes, M.A. Centeno
Comunicación oral

Gold on carbon for biomass conversion: the case of selective base-free glucose oxidation
C. Megías-Sayago, J.L. Santos, M. Chenouf, F. Ammmari, S. Ivanova, M.A. Centeno, J.A. Odriozola
Comunicación oral

5th International Conference on Structured Catalysts and Reactors ICOSCAR5

21 – 24 junio [San Sebastián, España]

Impact of structured catalysts in amine oxidation under mild conditions
J.J. Santos, J.A. Odriozola, M.A. Centeno, O.D. Pavel, B. Jurca, V.I. Pârvulescu
Comunicación oral

Thermal profiles during the combustion of ternary mixtures (H_2, CO and CH_4) in metallic micromonoliths coated with Pt/Al_2O_3 catalysts
O.H. Laguna, M. Rodríguez Rodríguez, M.A. Centeno, J.A. Odriozola
Poster

Use of CO_2 for syngas production with H_2/CO ratio of 2 employing structured catalysts
A. Alvarez, J.M. Martínez T., M.A. Centeno, J.A. Odriozola
Poster

Selective lactose oxidation in liquid phase on Au/Al_2O_3 metallic monolith catalysts
S.A. Regenhardt, C.I. Meyer, O. Sanz, S. Ivanova, M.A. Centeno, J.A. Odriozola, M. Montes, T.F. Garetto, A.J. Marchi
Poster

Improvement of a platinum based catalyst for WGS reaction enhancing the rate-limiting step
N. García-Moncada, L. Jurado, L.M. Martínez T, F. Romero-Sarria, J.A. Odriozola
Poster

17th Nordic Symposium on Catalysis 2016
14 – 16 junio [Lund, Suecia]

Fischer-Tropsch synthesis over Zr-promoted Co/γ -Al_2O_3 catalysts
J. Barrientos, V. Garcilaso de la Vega, B. Venezia, M. Boutonnet, S. Järås
Poster

Challenges and prospects for solid state chemistry CPSSC16
9 septiembre – 10 septiembre [Sevilla, España]

Crystal structure of new polyoxometalate/ionic liquid hybrids and their application in catalysis
C. Megías-Sayago, E. Alvarez, S. Ivanova, J.A. Odriozola
Poster

Effect of Preparation pH on Bismuth Titanate materialswith high visible light photocatalytic activity
P. Zambrano, J.A. Navío, M.C. Hidalgo
Poster

Efficient UV-photocatalytic activity of ZnO and Ag/ZnO synthesized by a facile method
M.A. C.A. Jaramillo-Páez, J.A. Navío, M.C. Hidalgo, M. Macias
Poster

High UV-photocatalytic activity of ZnO synthesized by three different procedures: A comparative assessment
C.A. Jaramillo-Páez, J.A. Navío, M.C. Hidalgo, M. Macias
Poster

Remarkable enhancement in photocatalytic activity of {001} faceted TiO_2 loaded with Pt
M.A. Lara, J.A. Navío, M.C. Hidalgo
Poster

Nnoparticles Pt-Cu: In situ analysis by DRX and NAP-XPS
R. Castillo Barrero, F. Romero-Sarria, J.A. Odriozola
Poster

Synthesis and impedance spectroscopy of doped Zirconia based ionic conductors
N. García Moncada, R. Poyato, F. Romero-Sarria, J.A. Odriozola
Poster

Rh/$MgAl_2O_4$ structured catalyst for syngas production via dry reforming of biogas
V. Garcilaso, M.A. Centeno, J.A. Odriozola
Poster

Ruthenium nanoparticles in CO_2 methanation: influence of the synthesis method in catalytic behaviour
S. Navarro-Jaén, A.E. Szego, O.H. Laguna Espitia, F. Romero-Sarria, J.A. Odriozola
Poster

CARBON2016 The World Conference on Carbon
10 – 15 julio [Pennsylvania, Estados Unidos de América]

Modification and characterization of the surface chemistry of a colombian metallurgical coke
D.A. Rico, Y.Y. Agámez, E.R. Romero, M.A. Centeno, J.A. Odriozola, J.J. Díaz
Poster

3rd International Symposium on Catalysis for Clean Energy and Sustainable Chemistry, CCESC2016
7 – 9 septiembre [Madrid, España]

Ru-Ni catalysts for CO_2 conversion using methane reforming reactions
A. Alvarez, M.A. Centeno, J.A. Odriozola
Poster

XXV Congreso Iberoamericano de Catálisis CICAT2016
18 – 23 septiembre [Montevideo, Uruguay]

Catalizadores platino-carbón para la reacción de Water Gas Shift
J.L. Santos, P. Tarifa, F. Cazaña, A. Monzón, C. López-Cartes, M.A. Centeno
Comunicación oral

Coque metalúrgico modificado como soporte de catalizadores para licuefacción directa de carbón
D.A. Rico, E. Romero, Y.Y. Agamez-Pertuz, M.A. Centeno, J.A. Odriozola, J.J. Díaz
Comunicación oral

Influencia del grado de hidroxilación de la alúmina sobre las especies originadas tras la adsorción de CO sobre un catalizador modelo de Fischer-Tropsch
E.M. Jímenez-Barrera, Francisca Romero-Sarria, J.A. Odriozola
Comunicación oral

Síntesis y caracterización de nanopartículas Pt-Cu como catalizadores para reacciones de purificación de corrientes de hidrógeno: eliminación del carbón superficial
R. Castillo, F. Romero-Sarria, C. López-Cartes, J.A. Odriozola
Comunicación oral

Catalizadores $Co/Ce_xZr_{1-x}O2/Al_2O_3$ para la reacción de Fischer-Tropsch: estudios de actividad y selectividad
V. Garcilaso de la Vega, J. Barrientos, O.H. Laguna, M. Boutonnet, M.A. Centeno, J.A. Odriozola
Poster

Fotocatalizadores basados en TiO_2 sensibilizado con colorantes y su efectividad en la eliminación de contaminantes orgánicos
J.J. Murcia, A. Martínez, E. Giovanna, H.A. Rojas, S. Ivanova, A. Penkova, O.H. Laguna
Poster

Influencia de la reacción de WGS de conductores iónicos basados en óxidos de Zr
N. García-Moncada, R. Poyato, O. Sanz, F. Romero-Sarria, J.A. Odriozola
Poster

Oxidación selectiva de lactosa en fase líquida sobre catalizadores monolíticos metálicos de Au/Al_2O_3
C.I. Meyer, S.A: Regenhardt, O. Sanz, S. Ivanova, M.A. Centeno, J.A. Odriozola, M. Montes, A.J. Marchi, T.F. Garetto
Poster

Nanopartículas de rutenio como catalizadores para metanación de CO y CO_2
S. Navarro, O.H. Laguna, M.A. Centeno, J.A. Odriozola
Poster

CONGRESOS Y REUNIONES NACIONALES / NATIONAL CONGRESSES AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

II Encuentro de Jóvenes Investigadores de la SECAT

27 – 29 junio [Ciudad Real, España]

Especies superficiales sobre un catalizador modelo para Fischer-Tropsch: influencia del agua
E.M. Jimenez-Barrera, P. Bazin. F. Romero-Sarria, M. Daturi, J.A.Odriozola
Comunicación oral

Óxidos de Zinc preparados por tres procedimientos diferentes. Actividad Fotocatalítica comparada bajo Iluminación UV
C. Jaramillo-Paez, J.A. Navío, M.C. Hidalgo, M. Macias
Poster

FORMACION / TRAINING

FORMACIÓN DE GRADUADOS / MASTER DEGREE THESIS

Título:	**Generación de nanoestructuras carbonosas sobre superficies metálicas y cerámicas**
Autor:	Pablo Navarro Vicente
Directores:	Miguel Angel Centeno Gallego, José Antonio Odriozola Gordón
Grado:	Trabajo Fin de Grado
Centro:	Universidad de Sevilla
Año Académico:	2015-2016 (12 enero 2016)

Título:	**Sistemas metálicos de aplicación en terapia de hipertermia oncológica**
Autor:	Natalia Nevado González
Directores:	Svetlana Lyubomirova Ivanova, María Isabel Domínguez Leal
Grado:	Trabajo Fin de Grado
Centro:	Universidad de Sevilla
Año Académico:	2015-2016 (15 febrero 2016)

Título:	**Conversión química de nanotubos de carbono en portadores de fármacos**
Autor:	Adolfo Diaz Centeno
Directores:	Laura López, Svetlana Ivanova

Grado: Trabajo Fin de Grado
Centro: Universidad de Sevilla
Año Académico: 2015-2016 (1 julio 2016)

Título: **Síntesis, caracterización y ensayo de catalizadores hibridos ALPO-liquido ionico en reacciones de transformacion de glucosa**
Autor: Julia Ángeles Curquejo Morales
Directores: José Antonio Odriozola, Svetlana Ivanova
Grado: Trabajo Fin de Grado
Centro: Universidad de Sevilla
Año Académico: 2015-2016 (1 julio 2016)

Título: **Caracterización y aplicaciones catalíticas de biocarbones**
Autor: Antonio Manuel Gil Mora
Directores: José Antonio Odriozola, Miguel Angel Centeno
Grado: Trabajo Fin de Máster
Centro: Universidad de Sevilla
Año Académico: 2015-2016 (1 julio 2016)

Título: **Síntesis y caracterización de materiales compuestos metal-cerámica (CMC) con aplicaciones catalíticas**
Autor: Anthony Edward Szego Nagle
Directores: Miguel Angel Centeno, Francisca Romero Sarria
Grado: Trabajo Fin de Grado
Centro: Universidad de Sevilla
Año Académico: 2015-2016 (15 julio 2016)

Título: **Adsorcion de colorantes en nanoparticulas de silice. Aplicaciones biomedicas**
Autor: Francisco José Fernández Fernández
Directores: Svetlana Lyubomirova Ivanova, María Isabel Domínguez Leal
Grado: Trabajo Fin de Grado
Centro: Universidad de Sevilla
Año Académico: 2015-2016 (19 julio 2016)

Título: **Generación de recubrimientos adsorbentes sobre superficies cerámicas**
Autor: Inmaculada Suárez Díaz
Directores: Miguel Angel Centeno, María Isabel Domínguez Leal
Grado: Trabajo Fin de Grado
Centro: Universidad de Sevilla
Año Académico: 2015-2016 (19 septiembre 2016)

Título: **Desarollo de materiales para su uso como sensores de ácidos grasos en aplicaciones biomédicas**
Autor: Macarena Palmero Llopis
Directores: Svetlana Lyubomirova Ivanova, María Isabel Domínguez Leal
Grado: Trabajo Fin de Grado
Centro: Universidad de Sevilla
Año Académico: 2015-2016 (20 septiembre 2016)

Título: **Hidrogenación de CO_2 sobre catalizadores de Ru: estudios en ‘‘operando’’**
Autor: María Domínguez Gómez
Directores: María Pérez Cadenas, José Antonio Odriozola, Francisca Romero Sarria
Grado: Trabajo Fin de Máster
Centro: Universidad de Sevilla
Año Académico: 2015-2016 (10 octubre 2016)

Premios y Reconocimientos / Prizes and Acknowledgements

Rosa Pereñíguez García. **Premio Real Maestranza de Caballería 2015**. Los estudios llevados se centran en la preparación y caracterización de materiales catalíticos nanoestructurados, aplicados a la descontaminación ambiental y a la búsqueda de fuentes alternativas de energía, por ejemplo, mediante la producción de hidrógeno empleando sistemas basados en perovskitas. Los Premios para Investigadores Jóvenes de la Real Maestranza se entregan con la colaboración de la Real Academia Sevillana de Ciencias y están dotados con 6.000 euros. Su propósito es recompensar cada año los méritos científicos de investigadores jóvenes destacados y estimular su vocación y dedicación a la ciencia.

Estancias y Visitas de Personal del ICMS en otros Centros
Personnel of the ICMS in Other Laboratories

Université Louis Pasteur
Estrasburgo, Francia **Elena María Jiménez Barrera** 5 septiembre / 5-diciembre

Instituto Politecnico de MIlán
Milán, Italia **Nuria García Moncada** 1 septiembre / 2 diciembre

Dipartamento di Chimica Industriale “Tosso Montanari” of the University of Bologna
Bologna, Italia **Cristina Megías Sayago** 5 septiembre / 21 diciembre

ESTANCIAS Y VISITAS DE INVESTIGADORES EN EL ICMS

PERSONNEL OF THE OTHER LABORATORIES IN THE ICMS

Université Setif1		
Setif, Argelia	**Meriem Chebout**	1 octubre 2015 / 30 octubre 2016
Universidad Nacional		
Bogotá, Colombia	**Nicolás Rodríguez Riaño**	23 sept. 2015 / 30 septiembre 2016
Departamento de Ingeniería Química. Facultad de Ingeniería. UDEA		
Medellin, Colombia	**Oscar Felipe Arbeláez Pérez**	16 junio / 19 julio
Faculty of Sciences		
Gabes, Tunez	**Imen Jaouali**	25 abril / 8 julio
Ecole Nationale Superiere de Chimie de Lille		
Lille, Francia	**Olivier Bruyen**	25 abril / 8 julio
	Rémi Depond	1 junio / 31 julio
Facultad de Química. Universidad de Bucarest		
Bucarest, Hungria	**Mihaela Mireaia Transdafir**	7 julio / 1 octubre
Universidad Juárez Autonómica de Tabasco		
Juárez, México	**Melina del Carmen Uribe López**	1 septiembre / 31 octubre

EQUIPAMIENTO CIENTÍFICO AVAILABLE EQUIPMENT

- Molino de bolas de movimiento planetario PM100 de RETSCH
- Espectrofotómetro Infrarrojos: Thermo-Nicolet Nexus FT-IR; Thermo-Nicolet 380 FT-IR
- Accesorio DRIFTS, celdas de alta y baja temperatura
- Sistema de vacío cuarzo/vidrio para adsorción de moléculas sonda seguido por FT-IR
- Espectrómetro de masas: Balzers Thermostar
- Sistema TPR-TPO (con posibilidad de realizar pulsos) seguido de espectrómetro de masas (Balzers) y detector de conductividad térmica. PID Eng&Tech.
- Equipos comerciales de actividad catalítica Microactivity Reference PID Eng&Tech (4)
- Microscopio metalográfico: Leica DMIRM
- Rotavapor: Heidolph Hei-VAP Value
- Equipo de ultra-alto vacío para espectroscopía XPS y Auger equipado con cañón de argón para realizar devastado iónico.
- Estufas: P-Selecta; P-Selecta digiheat

- Ph-metros: Crison pH burette 24; Crison phMeter Basic 20; Crison micropH2000.
- Cromatógrafos (2): Agilent Technologies 7890 A GC System; Agilent Technologies 6890 N Network GC System.
- Micro-cromatógragos, microGC (2): Micro Gas Cromatograph CP-4900 Varian (2)
- HPLC: Varian 356-LC, Solvent Delivery Module Varian ProStar.
- Horno de soldadura: Microtest Máquina de ensayos EM2/200/FR
- Baño de ultrasonidos: P-Selecta Ultrasons Medi-II
- Horno Energon
- Horno para tratamiento de aceros a alta temperatura equipado con medidores de flujo e inyector de agua.
- Planta Piloto de Integración de reacciones catalíticas Reference PID Eng&Tech
- Espectrómetro Uv-Vis (Varian Cary 100, con esfera integradora para
- muestras sólidas)
- Analizador de Carbón Orgánico (TOC-V CHP Shimadzu 5000A)
- Cromatógrafo (HPLC Agilent Technologies 1200)
- Espectrómetro IR (Varian 660-IR FTIR Spectrometer)
- Dip-Coater con cámara de temperatura (SS-00 AB Table Dry Oven MTI
- Corporation)
- Espectrofotómetro FTIR con celdas DRIFTS y ATR.
- Sistema de análisis TPR/TPO con detector TCD y espectrómetro de masas.
- 6 reactores catalíticos de gases con detección por cromatografía de gases y espectrometría de masas.
- 2 reactores catalíticos de líquidos que permiten el seguimiento de hasta 8 reacciones de forma simultánea con control de temperatura y flujo de gases.
- 4 reactores catalíticos de líquidos de alta presión y temperatura con agitación interna y control de flujo de gases.
- Reactores fotocatalíticos con lámparas de Xe y Hg.
- Espectrofotómetros FTIR con accesorios DRIFTS, ATR y sistema de vacío en cuarzo/vidrio para adsorción de moléculas sonda
- Espectrómetros de masas
- Sistemas TPR-TPO (con posibilidad de realizar pulsos) seguido de espectrómetro de masas y detector de conductividad térmica.
- Microscopio metalográfico
- Rotavapor
- Estufas
- Ph-metros
- Cromatógrafos de Gases
- Micro-cromatógragos, microGC
- Cromatografos HPLC
- Horno de soldadura: Microtest Máquina de ensayos EM2/200/FR
- Baño de ultrasonidos
- Hornos
- Horno para tratamiento de aceros a alta temperatura equipado con medidores de flujo e inyector de agua.
- Planta Piloto de Integración de reacciones catalíticas Reference PID Eng&Tech
- Espectrómetro Uv-Vis, con esfera integradora para muestras sólidas)
- Analizador de Carbón Orgánico

- Dip-Coater con cámara de temperatura
- Reactores catalíticos de gases con detección por cromatografía de gases y espectrometría de masas.
- Reactores catalíticos de líquidos que permiten el seguimiento de hasta 8 reacciones de forma simultánea con control de temperatura y flujo de gases.
- Reactores catalíticos de líquidos de alta presión y temperatura con agitación interna y control de flujo de gases.
- Reactores fotocatalíticos con lámparas de Xe y Hg.

INGENIERÍA DE CERÁMICOS PARA AMBIENTES EXTREMOS

ENGINEERED CERAMICS FOR EXTREME ENVIRONMENTS

PERSONAL / PERSONNEL

Catedráticos

- Dr. Miguel Angel Castro Arroyo
- Dr. Manuel Jiménez Melendo
- Dra. Pilar Malet Maenner
- Dr. Julián Martínez Fernández
- Dr. Antonio Ramírez de Arellano-López

Científicos Titulares

- Dr. José Jesús Benítez Jiménez
- Dra. María Dolores Alba Carranza

Profesores Titulares

- Dr. Alfonso Bravo León
- Dra. Caroline M. Clauss Klamp

Investigadores Contratados

- Dr. Miguel Antonio Cruz Carrillo
- Dra. Esperanza Pavón González

Profesor Contratado Doctor

- Dra. M. Mar Orta Cuevas
- Dr. Joaquin Ramírez Rico

Becarios Predoctorales

- Ldo. Rafael Cabezas Rodríguez
- Lda. Aurora Gómez Marín
- Lda. María del Pilar Orihuela Espina
- Ldo. Francisco J. Osuna Barroso
- Lda. M. Carmen Vera García

PROYECTOS DE INVESTIGACIÓN / RESEARCH PROJECTS

Nuevo concepto de caldera de biomasa basada en materiales biocerámicos y combustión porosa para operación eficiente con residuos

Bioceramic Materials for New Biomass Domestic Bolier Concept based on Porous Combustion for a Wide Biomass/Residues Feedstock

Código/Code:	MAT2016-76526-R
Periodo/Period:	30-12-2016 / 29-12-2019
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	60.500 €
Investigador responsable/Research head:	Joaquin Ramírez Rico /Ricado Chacartegui
Componentes/Research group:	Manuel Jiménez Melendo, Julián Martínez Fernández

RESUMEN / ABSTRACT

En la Unión Europea se generan anualmente más de cinco toneladas de residuos por personas, siendo aproximadamente el 60% de esta cantidad materia orgánica. La tecnología de las calderas de biomasa actuales no permiten el uso de estos residuos funcionando con altos valores de eficiencia, bajas emisiones y alta fiabilidadad de operación, siendo especialmente relevante en calderas de pequeño tamaño.

El principal objetivo de este proyecto es desarrollar un nuevo concepto de tecnología para calderas de biomasa doméstica capaz de operar con gran variedad de mezclas de biomasas y residuos agrícolas. Para ello se aprovecharán las sinergias de la integración de investigadores del: i) Grupo Maquinas y Motores Térmicos, GMTS, especialistas en combustión, calderas y máquinas térmicas ii) Grupo Materiales Biomiméticos y Multifuncionales, MBM, especialistas en la obtención de cerámicos porosos bioderivados, así como en caracterización físico-química y microestructural. El proyecto se completa con la colaboración de empresas en la evaluación de la tecnología y su aplicabilidad industrial En la Unión Europea se generan anualmente más de cinco toneladas de residuos por personas, siendo aproximadamente el 60% de esta cantidad materia orgánica. La tecnología de las calderas de biomasa actuales no permiten el uso de estos residuos funcionando con altos valores de eficiencia, bajas emisiones y alta fiabilidadad de operación, siendo especialmente relevante en calderas de pequeño tamaño.

El principal objetivo de este proyecto es desarrollar un nuevo concepto de tecnología para calderas de biomasa doméstica capaz de operar con gran variedad de mezclas de biomasas y residuos agrícolas. Para ello se aprovecharán las sinergias de la integración de investigadores del: i) Grupo Maquinas y Motores Térmicos, GMTS, especialistas en combustión, calderas y máquinas térmicas ii) Grupo Materiales Biomiméticos y Multifuncionales, MBM, especialistas en la obtención de cerámicos porosos bioderivados, así como en caracterización físico-química y

microestructural. El proyecto se completa con la colaboración de empresas en la evaluación de la tecnología y su aplicabilidad industrial

El proyecto se basa en la innovadora integración de material biocerámico microporoso en las cámaras de combustión de calderas de biomasa de modo que actúen con diferentes funciones: combustor microporoso, filtro de partículas y recuperador de calor. Estas funcionalidades pueden ser simultáneas, en función de la región del flujo en que se encuentren y el rango de temperaturas de esa región. Este material biocerámico es desarrollado a partir de precursores vegetales para obtener elementos de Carburo de Silicio (SiC). Para ello se usan materiales locales sin tratar, produciendo elementos hechos a medida con propiedades microestructurales adecuadas para trabajar con altas temperaturas. Así, productos con geometrias complejas pueden ser obtenidos con relativamente bajo coste comparados con otros materiales con características macánicas y químicas similares. La integración de componentes basados en estos materiales posibilita nuevos diseños de calderas de biomasa con un alto control de la combustión, las temperaturas y la emisión de partículas. El nuevo diseño evita la sinterización y fusión de las cenizas, actuando en la formación y evolución de contaminantes, inhibiendo los mecanismos de producción de dioxinas y activando la completa oxidación del monóxido de carbono (CO) y soots. El nuevo concepto permitirá la operación con una importante varieda de mezclas biomasa/ residuos agrícolas con bajas emisiones aun cuando el combustible pesente un alto contenido de cenizas, resolviendo el principal reto para el desarrollo del uso residuos agrícolas en calderas de biomasa (especialmente las de menor tamaño). El desarrollo de esta tecnología permitirá ampliar los recursos de la Unión Europea para calefacción de uso doméstico. En la actualidad este uso supone un 30% del consumo energético total en la misma. La propuesta incluye el estudio de los procesos básicos de combustión, flujos, fabricación a medida de las matrices de materiales biocerámicos, así como estudio y desarrollo de prototipos de componentes y del sistema final. Estos serán estudiados a nivel de ensayos de laboratorio con residuos agrícolas, forestales y de la industria olivarera.

EU generates more than five tons of waste per person every year and about 60 % is organic waste. Current biomass domestic boiler technology does not allow the use of these residues with high efficiency, ultra-low emissions and high reliability operation. The main objective of this proposal is the development of a new concept of biomass domestic boiler technology able to combine these characteristics for operation with multiple biomass/residues blends. It is based on the integration of novel bioceramic porous materials matrices in combustion chamber and gases pathflow with functions as microporous combustors, particles filters and heat accumulators. These functions are simultaneous depending on the region of the boiler. Matrices of bioceramic materials are developed from wood precursors to obtain SiC elements through a process patented by the University of Seville. It uses local raw material, and produces parts with tailor made microstructure/properties, adequate for high temperature and reactive operation. Products with complex geometries can be obtained at relatively low cost compared with other materials of similar chemical and mechanical properties. The integration of components based on these materials allows new designs of biomass boilers with high control of combustion, temperature and particle emission. It avoids ash sintering and melting, acting on the formation and evolution mechanisms of ash and dioxins and activating the complete oxidation of CO and soots. The new concept allows the operation to a wider biomass/residues feedstock with low emissions and low maintenance even with fuels with high ash content, produced from many residues, solving main challenges for their extended use and increasing the European fuel resources for domestic heating. Domestic heating in Europe consumes 30%

of the total energy. The proposal includes prototypes development, fuel supply characteristics and preparation (geometry, compactness, composition, etc.) and combustion products management. Biomass/residues blends from agriculture, forestry, olive oil industry among others will be tested both in laboratory.

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Tratamiento sostenible de residuos industriales: Materiales adsorbentes de diseño y bionanocomposites en la inmovilization de metales pesados y productos de fision Susteinable industrial waste treatment: designed adsorbent materials and bionanocomposites for inmobilizing heavy metals and fision products

Código/Code:	MAT2015-63929-R
Periodo/Period:	01-01-2016 / 31-12-2018
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	60.500 €
Investigador responsable/Research head:	Maria Dolores Alba Carranza
Componentes/Research group:	Miguel Angel Castro Arroyo, Ana Carmen Perdigón Aller, María del Mar Orta Cuevas

RESUMEN / ABSTRACT

El Proyecto de investigación que se presenta aborda la exigencia tecnológica y medioambiental de desarrollar metodologías avanzadas para la eliminación de agentes contaminantes. El interés y los esfuerzos encaminados al desarrollo de nuevas tecnologías orientadas a tratamientos más eficientes en la inmovilización y revalorización de los residuos peligrosos es creciente en los planes de I+D+i. El objetivo central del proyecto se basa en el diseño de una estrategia de funcionalización de silicatos laminares de alta carga expansibles y en la síntesis de bionanocomposites a partir de ellos para conseguir una actividad eficaz respecto de la inmovilización de residuos tóxicos y peligrosos, catiónicos y aniónicos. Este objetivo es un cambio cualitativo en el trabajo que se viene desarrollando en el diseño de sistemas modelos adsorbentes con aplicaciones medioambientales de clara repercusión en la mejora de la calidad de vida de la población y conservación del medioambiente, ya que la funcionalización de diseño de los silicatos sintéticos permitirá la adsorción de un amplio abanico de adsorbentes en estado de oxidación aniónico-catiónico. La finalidad y objetivos del proyecto se centran en la Focus Area WASTE de H2020 y esta Focus Area se desarrolla dentro del reto 2 y 5 de H2020 y dentro del Reto 5 y 3 de los Planes Estatales de Investigación.

El proyecto ha despertado el interés de diversas empresas observadoras, EPOs, (ENRESA y la Agencia de la Energía y para la Sostenibilidad del Ayuntamiento de Sevilla) impulsando la colaboración público-privada. Por tanto, la investigación desarrollada auna los principios básicos de la estrategia estatal de Ciencia y Tecnología: Poner la I+D+I al servicio de la ciudadanía, del bienestar social y de un desarrollo sostenible, hacer de la I+D+I un factor de mejora de la competitividad empresarial (transferencia de los resultados al sector privado, ver interes de los EPOs) y reconocer y promover la I+D como un elemento esencial para la generación de nuevos conocimientos de excelencia.

La viabilidad de la propuesta se garantiza porque el equipo de investigación, EI, por un lado, ha desarrollado con éxito la síntesis de silicatos laminares hidratables de alta carga, a través de un método novedoso que permite ajustar la carga deseada para el material, y, por otro, ha conseguido exitosamente su organofuncionalización (patente ES2362597B1). Además, ha desarrollado la metodología necesaria para el correcto progreso de este proyecto, en estrecha colaboración con otros Grupos de investigación internacionales de reconocido prestigio (e.g. CNRS-Universidad de Lille, Universidad de Cambridge...). Además el EI ha demostrado que potencia la agrupación de las capacidades y competencias científico-técnicas esenciales para abordar esta propuesta de marcado caracter transversal.

The focus of the project addresses the requirement of advanced environmental technology methodologies for removing pollutants. Recently, the interest and efforts to develop new technologies for more efficient treatments for the immobilization and the revaluation of hazardous waste are increasing in R & D plans. The overall object of the project is based on the design of a strategy of functionalization of highly charged swelling phyllosilicates and their later transformation on bionanocomposite for the effective retention and immobilization of hazardous waste, both cationic and anionic. This object represents a qualitative change in the work that is being nowdays developed in the field of model adsorbents systems with environmental applications that will improve the quality life of the population and the environmental conservation, because the designed functionalization of the synthetic silicates will allow the adsorption of a wide range of adsorbents in different oxidation states, cationic or anionic. The objectives are conformed to the Focus Area WASTE of the H2020 program and it is developed on the 2nd and 5th challenge of the H2020 program and on the 5th and 3rd challenge of the national research program.

The project has attracted interest from various observers companies, EPOs, (ENRESA and the Water and Local Energy Agency and Sustainability of the City of Seville), the public-private collaboration being promoted. Therefore, the research combines the basic principles of the National Strategy of Science and Technology: Putting the R&D&I at the service of citizens, social welfare and sustainable development, making the R&D&I a factor of improving business competitiveness (transfer of results to the private sector, see interest of EPOs) and recognize and promote R&D&I as an essential element for the generation of new excellence knowledge.

The viability of the proposal is ensured, first, because the research team, RT, has accomplished the synthesis of hydratable high charged phyllosilicates, with a novel and original method that allows setting the material desired charge, and, later, has successfully achieved their organofunctionalization (patent ES 2 362 597 B1). Second, the RT has developed the required methodology for the development of this project in closed scientific collaboration with other well recognized international groups (i.e. CNRS-University of Lille, University of Cambridge...). The RT enhances the clustering of their capabilities and scientific-technical skills which are essential to address this proposal with a remarkable transverse character.

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Filtros Bio-Cerámicos para Partículas en Motores Diesel / Bio-ceramics for diesel engine particulate filters

Código/Code:	MAT2013-41233-R (Programa Retos)
Periodo/Period:	01-01-2014 / 31-12-2016
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	163.265,30 €
Investigador responsable/Research head:	Julián Martínez Fernández /Ricado Chacartegui
Componentes/Research group:	José Antonio Becerra Villanueva, Alfonso Bravo León, Manuel Jiménez Melendo, Antonio Ramírez de Arellano López, Joaquín Ramirez Rico, Francisco Varela Feria

RESUMEN / ABSTRACT

La importancia del control de las emisiones de partículas en motores diésel es fundamental dados el volumen global de los mismos y el impacto medioambiental y económico asociado. Los sistemas de control de partículas basados en modificaciones del proceso de combustión en el motor no son suficientes para alcanzar las exigencias normativas actuales, y menos las futuras, y por tanto necesariamente hay que emplear sistemas de postratamiento como los filtros. Existe un amplio margen de mejora de los mismos tanto en fiabilidad, control de la degradación de prestaciones, durabilidad, operación multicombustible y reducción de costes.

En el presente proyecto se abordará el desarrollo y fabricación de un filtro de partículas regenerativo en motores diésel que mejore las especificaciones de los sistemas actuales, basado en una nueva generación de materiales cerámicos bioderivados, y que integre sistemas para la combustión de partículas. Para ello se aprovecharán las sinergias integrando investigadores del:
i) Grupo Maquinas y Motores Térmicos, GMTS, especialistas en motores de combustión interna
ii) Grupo Materiales Biomiméticos y Multifuncionales, MBM, especialistas en la obtención de cerámicos porosos bioderivados, así como en caracterización físico-química y microestructural.
El proyecto se completa con la colaboración de empresas en la evaluación de la tecnología y su aplicabilidad industrial.

Se trabajará en las siguientes líneas:

- Determinación de rutas de procesado para el desarrollo de elementos filtrantes con propiedades físico-químicas idóneas, en base a los conocimientos previos en materiales bioderivados y nuevas tecnologías relativas al uso de geles de SiO2.
- Determinación de catalizadores idóneos y sistemas para su deposición.
- Fabricación de los elementos filtrantes constituidos de soporte poroso más catalizador.
- Caracterización exhaustiva de las propiedades microstructurales y físico-químicas de interés para la aplicación.
- Desarrollo de sistemas de activación para la regeneración del filtro.
- Diseño y fabricación de los filtros con geometría idónea y dimensiones prototipo.
- Diseño de la unidad piloto y estudio de la integración y operación sobre el motor de referencia.
- Diseño final del filtro para su instalación industrial.

Estudios previos desarrollados por MBM en estos materiales bioderivados han demostrado su potencialidad como elementos filtrantes de gas a altas temperaturas en plantas de gasificación de carbón, lo que avala el éxito de este proyecto, que abordará las mejoras necesarias para desarrollar la tecnología en las condiciones de combustión de los motores diésel, bajo condiciones dinámicas en vehículos y filtros regenerativos.

Una reducción de contaminantes en las emisiones de los motores diésel tendría un gran impacto medioambiental, para la salud y económico, debido a los cerca de 100 millones de vehículos diésel circulando en Europa y una industria vinculada con más de 2 millones de empleos directos y tendencia creciente en el mercado. Este proyecto aborda el Reto Social 3 del Horizonte 2020, Energía segura, limpia y eficiente. Además el uso de materiales biocerámicos permite la sustitución de los elementos metálicos empleados en la actualidad, por lo que también se alinea con el Reto Social 5 del Horizonte 2020 en la búsqueda de alternativas a las materias primas esenciales en aplicaciones ya existentes reduciendo la dependencia de importaciones y sostenibilidad de las aplicaciones.

The importance of controlling particulate emissions from diesel engines is essential given its volume and the associated environmental and economic impact. Control systems based on modifications of the combustion process in the engine are not sufficient to meet the requirements of current regulations, less future ones, and therefore it must necessarily be employed post treatment systems such as filters. There is considerable scope for improving them both in reliability, degradation of control performance, durability, multifuel operation and cost reduction.

This project will assess the development and manufacturing of regenerative particulate filter for diesel engines to improve the current system specifications, based on a new generation of ceramic bio-derivated materials, with integrated systems for particle combustion. This objectives will be achieved integrating researchers synergies from: i) Thermal Engines and Machines Group, GMTS , specialists in internal combustion engines ii) Multifunctional Biomimetic Materials Group, MBM, specialists in obtaining bio-derivated porous ceramic as well as physical, chemical and microstructural characterization. In addition, the project is completed with the collaboration of companies in assessing technology and its industrial applicability.

The following research lines will be addressed:

- Determination of processing routes that enable the development of filter elements with suitable physical, and chemical properties, based on prior knowledge in bio-derivated materials and new technologies regarding the use of SiO2 gels.
- Identification of suitable catalysts and systems for its deposition.
- Manufacture of the filter elements consisting of porous support and catalyst.
- Thorough characterization of the physical, chemical and microstructural properties of interest for the application.
- Development of activation systems for the filter regeneration.
- Design and manufacturing of the filters with suitable geometry and prototype dimensions.
- Pilot unit design and study of the integration and operation of engine.
- Final design of the filter for industrial facility.

Previous studies developed by MBM in these bio-derivated materials have demonstrated their potential as gas filter elements at high temperatures in coal gasification plants, which supports the likelihood of success of this project, which will address the improvements needed to develop the technology in the combustion conditions of diesel engines, under dynamic conditions in vehicles and regenerative filters.

A reduction of pollutant emissions from diesel engines would have a great environmental impact, health and economic development, with about 100 million diesel vehicles circulating in Europe and a related industry with over 2 million direct jobs and growing trend in market. This project addresses the Social Challenge 3 Horizon 2020, Secure, clean and efficient energy. In addition, using bioceramics allows replacement of metal components used today, which also aligns with the Social Challenge 5 of the Horizon 2020 in search of alternatives to essential raw materials in existing applications by reducing dependence on imports and sustainability of applications.

Estudio de mecanismo de adsorción de contaminantes aniónicos peligrosos por aluminosilicatos de diseño / Adsorption mechanisms study of harmful anionic pollutants by tailor-made aluminosilicates

Código/Code:	TAHUB-082. Programa Talent HUB
Periodo/Period:	01-02-2015 / 28-02-2017
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	142642,80 €
Investigador responsable/Research head:	Esperanza Pavón González

RESUMEN / ABSTRACT

El desarrollo científico, tecnológico e industrial en la últimas décadas del siglo pasado ha causado un incremento en la contaminación del medio ambiente. Debido a ello, la comunidad internacional reconoce la necesidad de desarrollar nuevas tecnologías y estrategias para el control de la contaminación. El objective principal de este Proyecto cumple con este propósito: el diseño de silicatos laminares expansibles de alta carga y su posterior modificación superficial para que se conviertan en materiales adecuados para la retención e inmobilización de contaminantes tóxicos aniónicos.

La metodología del Proyecto se basas en la síntesis de micas de alta carga expansibles con sustituciones isomórficas de Si^{+4} por Al^{+3} y con una densidad de carga en el rango de las micas frágiles pero con una capacidad de intercambio e hinchamiento inusuales en estos materiales. Para potenciar su capacidad de adsorción, la superficie de estos materiales se funcionalizará con magnetica por un lado y con la inclusion de cationes de alquilamonio, por otro.

Además, se establecerá un protocolo de inmobilizacion de productos aniónicos altamente tóxicos como son AsO_4^{2-}, SO_4^{2-}, en función de la estructura y la funcionalización de las micas de alta carga expansibles. Más tarde, la aplicabilidad de estas reacciones de adsorción se comprobará en suelos reales contaminados de Chile y España.

The scientific, technological and industrial development carried out in the second half of last century has caused an increasing pollution in the natural environment. Consequently, a widespread recognition of the need to develop technologies and strategies for pollution control has arisen in the recent times. The main objective of this Project is to design swelling layered silicates of high charge and their surface modification for an effective activity with respect to the retention and immobilization of toxic and dangerous anionic wastes.

The proposed methodology consists on the synthesis of high charge swelling mica with isomorphic substitution of Si^{4+} by Al^{3+} with a charge density in the range of brittle mica but with a cation exchange and swelling capacities unusual in these silicates. In order to enhance the anionic adsorption capacity, the mica will be functionalized in the surface with magnetite and with the inclusion of alkylammonium cations in their interlayer space.

An immobilization protocol of harmful anionic products like AsO_4^{2-}, SO_4^{2-} will be established, using the best adsorbent in function of both the structure and the funcionalization of the highly charged swelling mica. Afterwards, the applicability of these adsorption reactions will be tested in actual contaminated soils from Chili and Spain.

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Estudio de la inmovilización de metales pesados por micas de alta carga sintéticas organofuncionalizadas: pruebas a escala de laboratorio / Inmobilization of heavy metals by synthetic high-charged organomicas: Test at laboratory scale

Código/Code:	P12-FQM-567
Periodo/Period:	16-05-2014 / 16-02-2019
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	174.455 €
Investigador responsable/Research head:	María Dolores Alba Carranza

RESUMEN / ABSTRACT

El tema central del proyecto aborda la exigencia tecnológica mediambiental de desarrollar metodologías avanzadas para la eliminación de agentes contaminantes. El interés y los esfuerzos encaminados al desarrollo de nuevas tecnologías orientadas a tratamientos más eficientes en la inmovilización y revalorización de los residuos peligrosos es crecientes en los planes de I + D + i de los últimos años. Es en este escenario donde debe encuadrarse el presente proyecto y en concreto en el marco de la gestión de cationes de metales pesados, tema de elevado interés social en la presente década.

Desde la segunda mitad del siglo XX la Humanidad se ha enfrentado a un enorme desarrollo científico y tecnológico que es el responsable de un incremento de la contaminación mediambiental. Como ejemplo podemos mencionar dos problemas que en la actualidad son moti-

vos de preocupación y actuación de la Junta de Andalucía: contaminación de los litorales andaluces y las aguas residuales urbanas. Por tanto, estamos ante un problema complejo en el que los agentes contaminantes son variados, las fuentes de procedencia son diversas y las vías o rutas seguidas por los distintos contaminantes, frecuentemente, escapan al control necesario para evitar efectos indeseados sobre el entorno natural y urbano. Es por ello, que se demanda una investigación a nivel básico y aplicado de los mecanismos necesarios para la inmovilización de dichos cationes nocivos.

Los objetivos y alcance de este proyecto se basan en los avances llevados a cabos por otros grupos de investigación de la gestión de estos tipos de contaminantes y en los últimos resultados de la investigación llevada a cabo por el equipo de investigación que han permitido el diseño de silicatos laminares expansibles de alta carga con especiales propiedades como precursores para la retención de residuos nocivos. Por tanto, se propone en este proyecto la organofuncionalización de dichas micas sintéticas con grupos tioles o con cationes de alquilamonio de longitud de cadena variable y la evaluación de su capacidad de adsorción y retención irreversible de metales pesados.

The focus of the project addresses the environmental technological requirement to develop advanced methods for removing pollutants. The interest and efforts to develop new technologies aimed at more efficient treatment in detention and revaluation of hazardous waste is increasing in R & D plans. It is in this scenario where this project should be framed and in particular in the framework of the management of heavy metal cations, issue of high public interest in this decade.

Since the second half of the twentieth century, humanity has faced a huge scientific and technological development that is responsible for increased environmental pollution. As an example, we can mention two problems that are currently of concern and action of the Andalusian: Andalusian coastal pollution and urban wastewater. Therefore, this is a complex problem that pollutants sources are varied of origin and routes followed by various pollutants are diverse and, frequently, it is beyond the control necessary to avoid urban undesirable effects on the natural environment and. Therefore, a basic level research is demaned to implement the necessary mechanisms for the immobilization of such harmful cations.

The objectives and scope of this project are based on advances made by other research groups in the management of these types of contaminants and the latest research conducted by the research team that allowed design expandable high-charged layered silicates with special properties as precursors for the retention of harmful residues. Therefore, it is proposed in this project the organofunzionalization of such synthetic micas with thiol groups or alkylammonium cations of varying chain length and evaluation of its adsorption capacity and irreversible retention of heavy metals.

Obtención de bioplásticos tipo polihidroxialcanoato (PHA) de cadena larga a partir de desechos de epidermis de frutos comerciales / The obtention of fatty polyhydroxyalcanoate (PHA) bioplastics from peels residues of commercial fruits

Código/Code:	P11-TEP-7418 (Proyecto de Excelencia)
Periodo/Period:	16-05-2013 / 15-05-2016
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	174.455 €
Investigador responsable/Research head:	José Jesús Benítez Jiménez
Componentes/Research group:	Antonio Heredia Bayona, Miguel Angel San Miguel Barrera, Jaime Oviedo López, J. Alejandro Heredia Guerrero, Santiago Domínguez Meister, Daniel Aguilera Puerto, Francisco Javier Navas Martos, José Manuel de la Torre Ramírez

RESUMEN / ABSTRACT

El presente proyecto de investigación tiene por objeto el estudio de la viabilidad de la implementación de un proceso a escala planta piloto que permita obtener un material bioplástico a partir de una materia prima asequible y de bajo coste como son los desechos de pieles (epidermis) de frutos. La oportunidad de la propuesta parte de proponer un nuevo tipo de material polimérico completamente inocuo, biodegradable y ecológico como sustituto de plásticos tradicionales obtenidos a partir del petróleo que conllevan un serio problema medioambiental, tanto en su producción industrial como en su posterior desecho. Por otro lado, el concepto de sostenibilidad medioambiental se extiende no sólo a la biodegradabilidad del producto final y al bajo impacto del proceso de producción propuesto, también a su obtención a partir de un recurso vegetal que no se retrae de la cadena alimenticia animal y humana, como es el caso de los bioplásticos qu<e se vienen fabricando actualmente a partir de maíz o patata. Por otro lado, y en nuestro ámbito territorial andaluz, la materia prima es especialmente accesible dado el volumen de la actividad agroalimentaria. En este mismo sentido, el nuevo bioplástico podría paliar un importante problema de eliminación de residuos plásticos en explotaciones agrarias ya que se concibe como plenamente compostable y, por tanto, capaz de generar biomasa asimilable por la siguiente generación de plantas. El producto propuesto viene, además, a completar la gama de polihidroxialcanoatos (PHA) a los miembros de la serie obtenidos a partir de polihidroxiácidos de cadena larga. Las propiedades singulares de esta nueva familia puede ser un buen complemento de los ya conocidos y podría dar lugar a aplicaciones novedosas y de mayor valor añadido.

The main objective of this project is to evaluate the feasibility of scaling up a procedure to obtain fatty polyhydroxyalcanoate (PHA) bioplastics from a low-cost and abundant source like peels residues of commercial fruits. The strength of the proposal relies on the introduction of a new non-toxic and fully biodegradable polymeric material as a substitute for environmental-

hostile petroleum-based plastics. The overall sustainability is extended to the use of a low-impact synthetic route and to the processing of a plant residue rather than crops intended for human or cattle feeding. The project is considered of additional interest in regions with an agricultural based economy like Andalusia and with an important environmental impact arising from the greenhouse activity. The proposal also covers the study of new and more specific applications of such bio-based fatty polyhydroxyalcanoates.

Otros Proyectos / Other Projects

Bases genéticas de la composición y propiedades biofísicas de la cutícula del fruto del tomate. Aprovechamiento de la variabilidad natural

Código/Code:	AGL2015-65246-R
Periodo/Period:	01-01-2016 / 31-12-2018
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	187.550 €
Investigador responsable/Research head:	Rafael Fernández Muñoz (IHSM)
Componentes/Research group:	José Jesús Benítez, Fernando Gallardo Alba (UMA), Antonio Heredia Bayona (IHSM)

Convenios y Contratos / Contracts and Agreements

Análisis comparativo de la retención de Cesio e yodo por barreras reactivas de arcillas: Escala Prepiloto

Periodo/Period:	01-07-2015 / 30-06-2017
Organismo Financiador/Financial source:	ENRESA
importe total/Total amount:	139.755 €
Investigador responsable/Research head:	Miguel Angel Castro Arroyo
Componentes/Research group:	María del Mar Orta Cuevas, M. Dolores Alba Carranza

Patentes / Patents

Poliester obtenido a partir de residuos de tomate mediante policondensación no catalizada en estado fundido

Inventores: José Alejandro Heredia Guerrero, José Jesús Benítez Jiménez, Antonio Heredia Bayona
Ambito Geográfico: Patente Española, España
Patente Solicitada. Número de solicitud: 201630194
Fecha de solicitud: 19/febrero/2016

Entidades Titulares: Consejo Superior de Investigaciones Científicas. Universidad de Málaga. Fondazione Instituto Italiano di Tecnologia

Artículos Publicados en Revistas SCI / Papers In SCI Journals

Electrochemical Energy Storage Applications of CVD Grown Niobium Oxide Thin Films

Fiz, Raquel; Appel, Linus; Gutierrez-Pardo, Antonio; Ramirez-Rico, Joaquin; Mathur, Sanjay
ACS Applied Materials & Interfaces, **8** (2016) 21423-21430
Agosto, 2016 | **DOI:** 10.1021/acsami.6b03945

We report here on the controlled synthesis, characterization, and electrochemical properties of different polymorphs of niobium pentoxide grown by CVD of new single-source precursors. Nb_2O_5 films deposited at different temperatures showed systematic phase evolution from low-temperature tetragonal (TT-Nb_2O_5, T-Nb_2O_5) to high temperature monoclinic modifications (H–Nb_2O_5). Optimization of the precursor flux and substrate temperature enabled phase-selective growth of Nb_2O_5 nanorods and films on conductive mesoporous biomorphic carbon matrices (BioC). Nb_2O_5 thin films deposited on monolithic BioC scaffolds produced composite materials integrating the high surface area and conductivity of the carbonaceous matrix with the intrinsically high capacitance of nanostructured niobium oxide. Heterojunctions in Nb_2O_5/BioC composites were found to be beneficial in electrochemical capacitance. Electrochemical characterization of Nb_2O_5/BioC composites showed that small amounts of Nb_2O_5 (as low as 5%) in conjunction with BioCarbon resulted in a 7-fold increase in the electrode capacitance, from 15 to 104 F g–1, while imparting good cycling stability, making these materials ideally suited for electrochemical energy storage applications.

Manganese Dioxide Supported on Porous Biomorphic Carbons as Hybrid Materials for Energy Storage Devices

Gutierrez-Pardo, A; Lacroix, B; Martinez-Fernandez, J; Ramirez-Rico, J
ACS Applied Materials & Interfaces, **8** (2016) 30890-30898
Noviembre, 2016 | **DOI:** 10.1021/acsami.6b09361

A facile and low-cost method has been employed to fabricate MnO2/C hybrid materials for use as binder-free electrodes for supercapacitor applications. Biocarbon monoliths were obtained through pyrolysis of beech wood, replicating the microstructure of the cellulosic precursor, and serve as 3D porous and conductive scaffolds for the direct growth of MnO, nanosheets by a solution method. Evaluation of the experimental results indicates that a homogeneous and uniform composite material made of a carbon matrix exhibiting ordered hierarchical porosity and MnO, nanosheets with a layered nanocrystalline structure is obtained. The tuning of the MnO2 content and crystallite size via the concentration of KMnO4 used as impregnation solution allows to obtain composites that exhibit enhanced electrochemical behavior, achieving a capacitance of 592 F g(-1) in electrodes containing 3 wt % MnO2 with an excellent cyclic stability. The electrode materials were characterized before and after electrochemical testing.

Influence of temperature and time on the Eu3+ reaction with synthetic Na-Mica-n (n=2 and 4)

Garcia-Jimenez, MJ; Cota, A; Osuna, FJ; Pavon, E; Alba, MD
Chemical Engineering Journal, **284** (2016) 1174-1183
Enero, 2016 | **DOI:** 10.1016/j.cej.2015.09.077

Bentonite is accepted as the best clay material for the engineered barrier of Deep Geological Repositories (DGRs). The performance of clay as the main component of the engineered barrier in the DGR has been intensively studied and the structure of the selected clay mineral play a crucial role. In this sense, a new family of synthetic swelling silicates, Na-Mica-n, with tuned layer charge (n) values between 2.0 and 4.0 per unit cell has recently been synthesized and a general synthetic method has been reported. These swelling high-charge micas could be highly valuable for the decontamination of harmful cations. The ability of these micas to immobilize Eu3+ under subcritical conditions has been probed. The adsorption was in both non-specific sites (cation exchange mechanism) and specific sites (chemical reaction or surface defects adsorption). Moreover, its adsorption capacity, under the same conditions is higher than in saponite and far superior to the bentonites.

Permeability and mechanical integrity of porous biomorphic SiC ceramics for application as hot-gas filters

Gomez-Martin, A.; Orihuela, M. P.; Becerra, J. A.; Martinez-Fernandez, J.; Ramirez-Rico, J.
Materials & Design, **107** (2016) 450-460
Octubre, 2016 | **DOI:** 10.1016/j.matdes.2016.06.060

Biomorphic SiC is a biotemplated material fabricated by Si melt-infiltration of carbon preforms from wood pyrolysis. In this work, porous bioSiC ceramics from five different wood precursors, with porosities between 45 and 72% were studied for their feasibility in filtering applications.
Gas permeability and mechanical stability were investigated as a function of the microstructure of the starting wood precursor. Air-permeation performance at room temperature was measured for a range of flow rates, and the permeability constants were assessed by fitting of Forchheimer's equation to the experimental data. Darcian permeabilities were achieved in the range 10− 11–10− 12 m2, while inertial terms were in the range 10− 7–10− 8 m, showing a correlation with the average pore size and orientation of the larger channels. Regarding the mechanical stability, maximum compressive strength values were reached in the range of 3–115 MPa.
These results improve our understanding of the ways in which the microstructure influences permeability and mechanical robustness, enabling the device requirements to be tailored by selecting the wood precursor. It was also shown that these materials are promising for hot-gas filtering applications.

Thermal conductivity of Fe graphitized wood derived carbon

Ramirez-Rico, J; Gutierrez-Pardo, A; Martinez-Fernandez, J; Popov, VV; Orlova, TS
Materials & Design, **99** (2016) 528-534
Junio, 2016 | **DOI:** 10.1016/j.matdes.2016.03.070

Graphitic porous carbon materials from pyrolysis of wood precursors were obtained by means of a nanosized Fe catalyst, and their microstructure and electrical and thermal transport properties investigated. Thermal and electrical conductivity of graphitized carbon materials increase with the pyrolysis temperature, indicating a relationship between the degree of graphitization and thus in crystallite size with transport properties in the resulting carbon scaffolds. Evaluation of the experimental results indicate that thermal conductivity is mainly through phonons and increases with the temperature in Fe-catalyzed carbons suggesting that the mean free path of phonons in the material is small and defect scattering dominates over phonon-phonon interactions in the range from room temperature to 800 °C.

Biological strategy for the fabrication of highly ordered aragonite helices: the microstructure of the cavolinioidean gastropods

Checa, AG; Macias-Sanchez, E; Ramirez-Rico, J
Scientific Reports, **6** (2016) 25989
Mayo, 2016 | **DOI:** 10.1038/srep25989

The Cavolinioidea are planktonic gastropods which construct their shells with the so-called aragonitic helical fibrous microstructure, consisting of a highly ordered arrangement of helically coiled interlocking continuous crystalline aragonite fibres. Our study reveals that, despite the high and continuous degree of interlocking between fibres, every fibre has a differentiated organic-rich thin external band, which is never invaded by neighbouring fibres. In this way, fibres avoid extinction. These intra-fibre organic-rich bands appear on the growth surface of the shell as minuscule elevations, which have to be secreted differentially by the outer mantle cells. We propose that, as the shell thickens during mineralization, fibre secretion proceeds by a mechanism of contact recognition and displacement of the tips along circular trajectories by the cells of the outer mantle surface. Given the sizes of the tips, this mechanism has to operate at the subcellular level. Accordingly, the fabrication of the helical microstructure is under strict biological control. This mechanism of fibre-by-fibre fabrication by the mantle cells is unlike that any other shell microstructure.

Active metal brazing of silicon nitride ceramics using a Cu-based alloy and refractory metal interlayers

Fernandez, JM; Asthana, R; Singh, M; Valera, FM
Ceramics International, **42** (2016) 5447-5454
Marzo, 2016 | **DOI:** 10.1016/j.ceramint.2015.12.087

Silicon nitride/silicon nitride joints with refractory metal (W and Mo) interlayers were vacuum brazed using an active braze, Cu-ABA (Cu-3Si-2Al-2.25Ti, wt%), and two interlayer arrangements in a double-lap offset configuration: Si3N4/Cu-ABA/W/Cu-ABA/Mo/Cu-ABA/Si3N4 and Si3N4/Cu-ABA /Si3N4. Titanium segregated at the Si3N4/Cu-ABA and Mo/Cu-ABA interfaces, but not at the W/Cu-ABA interface. The room-temperature compression-shear strength values of Si3N4/Cu-ABA/Si3N4 and Si3N4/Cu-ABA/W/Cu-ABA/Mo/Cu-ABA/Si3N4 joints were 118 +/- 24 MPa and 22 +/- 5 MPa, respectively. Elevated-temperature compression tests showed that Si3N4/Cu-ABA/Si3N4 joints had strength of 31 +/- 6 MPa at 1023 K and 17 +/- 3 MPa at 1073 K. Likewise, Si3N4/Cu-ABA/W/Cu-ABA/Mo/Cu-ABA/Si3N4 joints had strength of 19 +/- 4 MPa at 1023 K and 13 +/- 3 MPa at 1073 K. Knoop microhardness profiles revealed hardness gradients across the joints. The effect of joint microstructure and test configuration on the mechanical behavior is discussed.

Thermal conductivity of porous biomorphic SiC derived from wood precursors

Gomez-Martin, A; Orihuela, MP; Ramirez-Rico, J; Chacartegui, R; Martinez-Fernandez, J
Ceramics International, **42** (2016) 16220-16229
Noviembre, 2016 | **DOI:** 10.1016/j.ceramint.2016.07.151

Biomorphic SiC is a SiC/Si composite made by the reactive infiltration of molten silicon by capillarity into a carbon preform from high-temperature pyrolysis of a wood porous precursor. When excess silicon is removed, a hierarchically porous SiC material with highly interconnected porosity is obtained. By choosing different wood precursors, different pore size distributions can be obtained thus tailoring the resulting properties.
We study the thermal conductivity of porous biomorphic SiC from five different precursors, including a recycled wood product, in order to determine the microstructure-conductivity correlation. Here, we remove the excess of silicon by a high temperature capillary extraction-evaporation method. We used the laser flash technique to measure thermal diffusivity in the range similar to 300 K to 973 K, in order to determine the thermal conductivity. Thermal conductivities in the range 4-88 W/m K were achieved. The temperature, porosity, pore shape and orientation, and the treatment used to remove the remaining silicon all have a significant impact in the resulting thermal conductivity. We explain this influence in a first approximation in terms of a geometrical model.

Stress measurement using area detectors: a theoretical and experimental comparison of different methods in ferritic steel using a portable X-ray apparatus

Ramirez-Rico, J; Lee, SY; Ling, JJ; Noyan, IC
Journal of Materials Science, **51** (2016) 5343-5355
Junio, 2016 | **DOI:** 10.1007/s10853-016-9837-3

Using area detectors for stress determination by diffraction methods in a single exposure greatly simplifies the measurement process and permits the design of portable systems without complex sample cradles or moving parts. An additional advantage is the ability to see the entire or a large fraction of the Debye ring and thus determine texture and grain size effects before analysis. The two methods most commonly used to obtain stress from a single Debye ring are the so-called cosαcos□αand full-ring fitting methods, which employ least-squares procedures to determine the stress from the distortion of a Debye ring by probing a set of scattering vector simultaneously. The widely applied sin2ψsin2□ψ method, in contrast, requires sample rotations to probe a different subset of scattering vector orientations. In this paper, we first present a description of the different methods under the same formalism and using a unified set of coordinates that are suited to area detectors normal to the incident beam, highlighting the similarities and differences between them. We further characterize these methods by means of in situ measurements in carbon steel tube samples, using a portable detector in reflection geometry. We show that, in the absence of plastic flow, the different methods yield basically the same results and are equivalent. An analysis of possible sources of errors and their impact in the final stress values is also presented.

Enhancement of dielectric barrier layer properties by sol-gel and PECVD stacks

Lopez-Lopez, C; Menendez, MF; Menendez, LA; Menendez, A; Sanchez, P; Alba, MD; Sanchez-Cortezon, E; Delgado-Sanchez, JM
Surface and Coatings Technology, **305** (2016) 36-40

Noviembre, 2016 | **DOI:** 10.1016/j.surfcoat.2016.07.085

Thin-film PV modules grown on flexible, light weight, thermally stable and low cost substrates such as stainless steel foil, are an attractive product for solar market applications. When metal foils are used as substrate, it is essential to deposit a dielectric barrier layer to isolate electrically and chemically the thin-film solar cells from the substrate. In this work, SiOx stacks deposited on 'rough' stainless steel by a combination of a new sol-gel formulation and a Plasma Enhanced Chemical Vapor Deposition (PECVD) deposition step are reported as a suitable dielectric barrier layer candidate. Using these SiOx multilayers, a smooth and homogeneous film was achieved. X-ray diffraction (XRD) analysis showed that back contact of the solar cell (based on Molybdenum) is not affected by the presence of the barrier layer. Moreover, according to X-ray photoelectron spectroscopy (XPS) and Secondary Ion Mass Spectrometry (SIMS) measurements, this approach led to excellent barrier layer properties against the diffusion of impurities from the stainless steel. A complete electrical characterization of these dielectric barrier layers was also carried out showing good electrical insulation.

Biodegradable polyester films from renewable aleuritic acid: surface modifications induced by melt-polycondensation in air

Benitez, JJ; Heredia-Guerrero, JA; de Vargas-Parody, MI; Cruz-Carrillo, MA; Morales-Florez, V; de la Rosa-Fox, N; Heredia, A

Journal of Physics D-Applied Physics, **49** (2016) 175601

Mayo, 2016 | **DOI:** 10.1088/0022-3727/49/17/175601

Good water barrier properties and biocompatibility of long-chain biopolyesters like cutin and suberin have inspired the design of synthetic mimetic materials. Most of these biopolymers are made from esterified mid-chain functionalized.-long chain hydroxyacids. Aleuritic (9,10,16-trihydroxypalmitic) acid is such a polyhydroxylated fatty acid and is also the major constituent of natural lac resin, a relatively abundant and renewable resource. Insoluble and thermostable films have been prepared from aleuritic acid by melt-condensation polymerization in air without catalysts, an easy and attractive procedure for large scale production. Intended to be used as a protective coating, the barrier's performance is expected to be conditioned by physical and chemical modifications induced by oxygen on the air-exposed side. Hence, the chemical composition, texture, mechanical behavior, hydrophobicity, chemical resistance and biodegradation of the film surface have been studied by attenuated total reflection-Fourier transform infrared spectroscopy (ATR-FTIR), atomic force microscopy (AFM), nanoindentation and water contact angle (WCA). It has been demonstrated that the occurrence of side oxidation reactions conditions the surface physical and chemical properties of these polyhydroxyester films. Additionally, the addition of palmitic acid to reduce the presence of hydrophilic free hydroxyl groups was found to have a strong influence on these parameters.

Sliding wear resistance of porous biomorphic sic ceramics

Lopez-Robledo, MJ; Gomez-Martin, A; Ramirez-Rico, J; Martinez-Fernandez, J

International Journal of Refractory Metals and Hard Materials, **59** (2016) 26-31

Septiembre, 2016 | **DOI:** 10.1016/j.ijrmhm.2016.05.004

Porous biomorphic SiC ceramics were fabricated from four different wood precursors, three natural woods and one recycled wood product, by reactive infiltration of molten silicon into a carbon preform obtained from wood pyrolysis. Sliding wear resistance when sliding against a Si_3N_4 ball in air was studied. Tribological experiments were done with a pin-on-disk apparatus, under normal loads of 1 and 2 N, at a sliding velocity of 100 mm/s. The wear properties and the volume fraction of porosity were correlated. A commercial sintered SiC ceramic was also tested for comparison. The measured values of friction coefficient were in the range reported in literature for monolithic SiC ceramics under similar dry contact conditions. Two concurrent wear mechanisms are taking place: abrasion from the SiC debris and soft ploughing. The presence of an oxide tribolayer was assessed using energy dispersive X-ray analysis. Wear rates were found to scale with the composite porosity.

Microstructure, elastic, and inelastic properties of biomorphic carbons carbonized using a Fe-containing catalyst

Orlova, TS; Kardashev, BK; Smirnov, BI; Gutierrez-Pardo, A; Ramirez-Rico, J
Physics of the Solis State, **58** (2016) 2481-2487
Diciembre, 2016 | **DOI:** 10.1134/S1063783416120234

The microstructure and amplitude dependences of the Young's modulus E and internal friction (logarithmic decrement δ), and microplastic properties of biocarbon matrices BE-C(Fe) obtained by beech tree carbonization at temperatures Tcarb = 850–1600°C in the presence of an iron-containing catalyst are studied. By X-ray diffraction analysis and transmission electron microscopy, it is shown that the use of Fe-catalyst during carbonization with Tcarb ≥ 1000°C leads to the appearance of a bulk graphite phase in the form of nanoscale bulk graphite inclusions in a quasi-amorphous matrix, whose volume fraction and size increase with Tcarb. The correlation of the obtained dependences E(Tcarb) and δ(Tcarb) with microstructure evolution with increasing Tcarb is revealed. It is found that E is mainly defined by a crystalline phase fraction in the amorphous matrix, i.e., a nanocrystalline phase at Tcarb < 1150°C and a bulk graphite phase at Tcarb > 1300°C. Maximum values E = 10–12 GPa are achieved for samples with Tcarb ≈ 1150 and 1600°C. It is shown that the microplasticity manifest itself only in biocarbons with Tcarb ≥ 1300°C (upon reaching a significant volume of the graphite phase); in this case, the conditional microyield stress decreases with increasing total volume of introduced mesoporosity (free surface area).

Thermal conductivity of partially graphitized biocarbon obtained by carbonization of medium-density fiberboard in the presence of a Ni-based catalyst

Orlova, TS; Parfen'eva, LS; Smirnov, BI; Gutierrez-Pardo, A; Ramirez-Rico, J
Physics of the Solis State, **58** (2016) 208-214
Enero, 2016 | **DOI:** 10.1134/S1063783416010236

The thermal conductivity k and resistivity rho of biocarbon matrices, prepared by carbonizing medium-density fiberboard at T (carb) = 850 and 1500A degrees C in the presence of a Ni-based catalyst (samples MDF-C(Ni)) and without a catalyst (samples MDF-C), have been measured for the first time in the temperature range of 5-300 K. X-ray diffraction analysis has revealed that the bulk graphite phase arises only at T (carb) = 1500A degrees C. It has been shown that the temperature dependences of the thermal conductivity of samples MDFC- 850 and MDF-C-850(Ni) in the range of 80-300 K are to each other and follow the law of k(T) similar to T (1.65), but the use of the Ni-catalyst leads to an increase in the thermal conductivity by a factor of approximately 1.5, due to the formation of a greater fraction of the nanocrystalline phase in the presence of the Ni-catalyst at T (carb) = 850A degrees C. In biocarbon MDF-C-1500 prepared without a catalyst, the dependence is k(T) similar to T (1.65), and it is controlled by the nanocrystalline phase. In MDF-C-1500(Ni), the bulk graphite phase formed increases the thermal conductivity by a factor of 1.5-2 compared to the thermal conductivity of MDF-C-1500 in the entire temperature range of 5-300 K; k(T = 300 K) reaches the values of similar to 10 W m(-1) K-1, characteristic of biocarbon obtained without a catalyst only at high temperatures of T (carb) = 2400A degrees C. It has been shown that MDF-C-1500(Ni) in the temperature range of 40aEuro'300 K is characterized by the dependence, k(T) similar to T (1.3), which can be described in terms of the model of partially graphitized biocarbon as a composite of an amorphous matrix with spherical inclusions of the graphite phase.

Strength and microplasticity of biocarbons prepared by carbonization in the presence of a catalyst

Shpeizman, VV; Orlova, TS; Smirnov, BI; Gutierrez-Pardo, A; Ramirez-Rico, J
Physics of the Solis State, **58** (2016) 703-710
Abril, 2016 | **DOI:** 10.1134/S1063783416040223

The microdeformation has been investigated under uniaxial compression of beech-derived biocarbons partially graphitized during carbonization in the presence of a Ni- or Fe-containing catalyst. The strength and ultimate fracture strain have been determined at different temperatures of carbonization of the samples in the absence or in the presence of a catalyst. It has been shown using high-precision interferometry that the deformation of biocarbon samples under uniaxial loading occurs through jumps (in magnitude and rate of deformation) with axial displacements in the nanometer and micrometer ranges. The use of a catalyst leads to a decrease in the size of nanometer-scale jumps and in the number of micrometer-scale jumps. The standard deviations of the strain rate on loading steps from the smooth average dependence of the strain rate on the displacement have been calculated for micrometer-scale jumps. A similar characteristic for nanometer- scale jumps has been determined from the distortion of the shape of beats in the primary interferogram. It has been shown that the variation in the standard deviation of the strain rate with a change in the carbonization temperature is similar to the corresponding dependence of the ultimate fracture strain.

■ CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

XI Encuentro Facultad de Ciencias-UPTC Y III Encuentro Nacional La Ciencia para el Desarrollo Sostenible del País

5 – 7 octubre [Tunja, Colombia]

Estudio in situ de la en capacidad de hidratación micas de alta carga expansible
E. Pavón, A. Cota, F.J. Osuna, M.D. Alba
Conferencia Invitada

Estructura y reactividad de silicatos para la vitrificación de residuos radiactivos
A. Cota, F.J. Osuna, E. Pavón, M.D. Alba
Conferencia Invitada

Funcionalización superficial de micas de diseño para la adsorción de metales pesados
F.J. Osuna, A. Cota, E. Pavón, M.D. Alba
Conferencia Invitada

FORMACION / TRAINING

FORMACIÓN DE GRADUADOS / MASTER DEGREE THESIS

Título:	**Desarrollo de materiales basados en aluminosilicatos para usos medioambientales**
Autor:	Noelia Muñoz Ceballos
Directores:	María Dolores Alba Carranza
Grado:	Trabajo Fin de Grado
Centro:	Universidad de Sevilla
Año Académico:	2015-2016 (16 septiembre 2016)

DOCENCIA / TEACHING

Maestría en Química (III y IV cohorte) y Doctorado en Ciencias Químicas (I cohorte)
Curso intensivo de caracterización estructural del orden a largo y corto alcance: Desde los sólidos policristalinos hasta los amorfos
Dra. M. Dolores Alba Carranza
Dra. Esperanza Pavón González
Lugar: Universidad Pedagógica y Tecnológica de Colombia (UPTC, Tunja)

EQUIPAMIENTO CIENTÍFICO AVAILABLE EQUIPMENT

- Horno tubular hasta 1200º C
- Horno tubular en atmósfera controlada hasta 1700º C
- Horno de Cámara hasta 1650º C
- Horno de Cámara hasta 1200 ºC
- Estufa hasta 400º C
- Estufa hasta 200º C
- Caja de guantes

- Reactores hidrotermales
- Línea de vacío
- 3 microscopios AFM.
- Microscopio STM en aire.
- Calorímetro diferencial de barrido (DSC).
- Analizador mecánico dinámico (DMA).
- Máquina de ensayos mecánicos por tracción.
- Analizador de ángulo de contacto.
- Balanza Langmuir-Blodgett (LB).
- Spin coater.

MECANOQUÍMICA Y REACTIVIDAD DE MATERIALES

MECHANOCHEMISTRY AND REACTIVITY OF MATERIALS

PERSONAL / PERSONNEL

Investigadores Científicos

- Dr. Angel Justo Erbez
- Dr. Luis Allan Pérez Maqueda
- Dra. Concepción Real Pérez
- Dr. Pedro José Sánchez Soto

Científicos Titulares

- Dra. María Jesús Diánez Millán
- Dr. Francisco José Gotor Martínez
- Dra. María Jesús Sayagués de Vega

Profesores Titulares

- Dra. María Dolores Alcalá González

Profesor Vinculado “Ad Honorem”

- Dr. José Manuel Criado Luque

Investigadores Contratados

- Dra. Mónica Benítez Guerrero
- Dr. José Manuel Córdoba Gallego
- Dr. Antonio Perejón Pazo
- Dr. Pedro E. Sánchez Jiménez

Profesor Contratado Doctor

- Dra. María Regla Ayala Espinar

Becarios Predoctorales

- Lda. Cristina García Garrido
- Lda. Eva Gil González
- Lda. Beatriz Sarrión Aceytuno

PROYECTOS DE INVESTIGACIÓN / RESEARCH PROJECTS

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Almacenamiento TErmoQuímico Híbrido de energía SOLAR concentrada SOLARTEQH Hybrid thermochemical storage of concentrated solar energy SOLARTEQH

Código/Code:	CTQ2014-52763-C2-1-R
Periodo/Period:	01-01-2015 / 31-12-2017
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	82.280 €
Investigador responsable/Research head:	Luis Allan Pérez Maqueda
Componentes/Research group:	María Jesús Diánez Millán, José Manuel Criado Luque

RESUMEN / ABSTRACT

Actualmente existen proyectos dentro de los prgramas Sunshot (USA) y FP7 (UE) en los que se analiza la viabilidad de lechos fluidizados de sólidos granulados para el almacenamiento químico de energía solar concentrada. Uno de los materiales considerados es la caliza natural ($CaCO_3$), abundante y barata. Usando una mezcla CO_2/aire en porcentajes relativos adecuados a las temperaturas de trabajo (600ºC-900ºC) se descarbonataría el $CaCO_3$ mediante reacción endotérmica en períodos de elevada irradiación o se carbonataría el CaO liberando calor cuando la temperatura descendiese por debajo de un cierto valor. Mediante la variación del %CO_2 en el gas de fluidización se provocarían las reacciones de descarbonatación-carbonatación según se desee reducir o aumentar la temperatura del lecho en función de la intensidad de radiación solar y de la demanda. Este control ayudaría a paliar el efecto de la variabilidad de la intensidad de radiación solar sobre la transferencia de calor al ciclo de vapor para la producción de corriente eléctrica. Además de tratarse de un almacenamiento de energía sin pérdidas, la densidad energética del $CaCO_3$ (~1 MWhr/m^3) es mayor que la de las sales fundidas actualmente empleadas en plantas comerciales (0.25-0.40 MWhr/m^3), siendo además la caliza un material no corrosivo, no degradable y que permitiría operar a mayores temperaturas y aumentar así la eficiencia de conversión termoeléctrica. No obstante, la fluidización de la caliza es altamente heterogénea, formándose canales de gas y agregados no fluidizables en el lecho que reducirían en gran medida la transferencia térmica, la eficacia de contacto sólido/gas y por tanto el grado de conversión. Por otra parte, se han puesto en marcha plantas piloto basadas en almacenamiento térmico en lechos fluidizados de sólidos granulados inertes de alta capacidad calorífica como la arena o el carburo de silicio que presentan un estado de fluidización uniforme con alta transferencia térmica. Estos sistemas poseen inevitables pérdidas térmicas y son necesarios grandes volúmenes para garantizar el suministro de calor al ciclo de vapor en períodos de baja radiación. Nuestro proyecto se basa en complementar de manera sinérgica las ventajas del almacenamiento térmico en lechos fluidizados de sólidos inertes con el químico mediante lechos fluidizados de mezclas de sólidos inertes fluidizables con otros basados en CaO (arena y caliza naturales por ejemplo). En nuestro trabajo evaluaremos la transferencia y almacenamiento de energía

solar concentrada de estos sistemas híbridos. El plan de trabajo contemplará acotar las condiciones óptimas de concentración de CO_2 en el gas de fluidización y proporción de arena/caliza en función de la temperatura para las que la eficacia de almacenamiento se viera optimizada. Estudiaremos las propiedades físicas y químicas de mezclas de arena/caliza y los parámetros físicos que favorezcan la transferencia y almacenamiento de calor en función de la intensidad de la radiación solar. Así mismo se explorarán métodos de estabilización térmica del CaO con el objeto de incrementar la reversibilidad de carbonatación/calcinación en condiciones prácticas. De manera paralela se desarrollará un modelo termodinámico que incluya aquellos procesos que afectan a la eficiencia energética del mismo y sirva para establecer parámetros óptimos de operación con el objetivo final de transferencia al sector tecnológico para lo que se contará con el apoyo de Abengoa Solar.

There are current projects within the Sunshot (USA) initiative and UE FP7 program in which the feasibility of fluidized beds for permanent chemical storage of concentrated solar energy is analyzed. One of the materials considered is the cheap and abundantly available natural limestone ($CaCO_3$). Using a CO_2/air mixture in suitable relative proportions according to the operating temperatures (600-900ºC), $CaCO_3$ would be decarbonated by endothermic reaction in periods of high irradiation or the CaO would be carbonated releasing heat when the temperature falls below a certain value. By varying the %CO_2 in the fluidization gas, either decarbonation or carbonation would be provoked as desired to reduce or increase the bed temperature based on the intensity of solar radiation and electricity demand. This control would help to alleviate the effect of the variability of sunlight intensity. Besides of the permanent storage of energy, the energy density of $CaCO_3$ (about 1 MWhr/m^3) is greater than that of molten salts currently used in commercial plants (0.25-0.40 MWhr /m^3). Furthermore, natural limestone is non-corrosive material, not degradable and would allow operation at higher temperatures thus increasing the thermoelectric conversion efficiency. However, the fluidization of limestone is typically very heterogeneous, being characterized by the formation of gas channels and large unfluidizable aggregates in the bed which greatly reduce the effectiveness of solid/gas contact and thus the heat transfer efficiency of the reaction. On the other hand, other projects have leaded to the development of successful small-scale pilot plants based on the thermal storage in fluidized beds of inert solids with high heat capacity such as fine silica sand or silicon carbide with good fluidization properties and thus characterized by a high heat transfer. However, these systems present unavoidable heat losses and large volumes are needed to ensure a supply of heat to the power cycle in periods of low solar irradiation. Our project is based on synergistically combine the heat storage in fluidized beds of fluidizable inert solids (such as sand) with the permanent chemical storage of CaO precursors (such as natural limestone) by the use of fluidized beds of mixtures of both granular materials. Experimental measurements will allow characterizing the behavior of the sand/natural limestone mixtures for the transfer and storage of concentrated solar energy. The working plan shall limit the optimum concentration of CO_2 in the fluidizing gas and proportion of sand/limestone as a function of temperature for optimizing the energy storage efficiency. The physic-chemical properties of mixtures of sand/limestone that favor heat transfer and storage according to the intensity of solar radiation will be delimited. Also thermal and chemical stabilization methods will be explored in order to increase the reversibility of the carbonation/calcination reaction under practical conditions. In parallel, a thermodynamic modeling work will be carried out that includes processes that affect the energy efficiency and serve

as a starting point to establish optimum operating parameters with the ultimate goal of transferring the knowledge to the technology sector. For this final purpose the project has the support of Abengoa Solar.

Desarrollo de cermets con aleaciones de alta entropía de mezcla como fase ligante para aplicaciones de mecanizado Development of cermets with high entropy alloys as binder phase for machining applications

Código/Code:	MAT2014-52407-R
Periodo/Period:	01-01-2015 / 31-12-2018
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	90.750 €
Investigador responsable/Research head:	Francisco José Gotor Martínez
Componentes/Research group:	José Manuel Córdoba Gallego, María Dolores Alcalá González, Pedro José Sánchez Soto, Concepción Real Pérez, María Jesús Sayagués de Vega

RESUMEN / ABSTRACT

El mecanizado es parte esencial de los procesos de fabricación empleados en muchos sectores industriales y posee una importante implicación económica, al representar una proporción significativa del coste total de fabricación. El éxito del mecanizado depende de múltiples factores, entre los que destaca la herramienta de corte utilizada. El mecanizado de alta velocidad y de los materiales denominados difíciles de mecanizar, como las superaleaciones empleadas en la fabricación de motores a reacción, imponen unas condiciones extremas de trabajo caracterizadas por altas temperaturas, presiones y tensiones, que pueden provocar el fallo prematuro en servicio de la herramienta de corte. Además, el deterioro de la herramienta, debido a un excesivo desgaste y deformación, hace que resulte difícil mantener las tolerancias y la integridad de la superficie mecanizada, lo que compromete seriamente las propiedades de fatiga de la pieza y, por tanto, su aplicabilidad y vida útil. La importante implicación económica de este tipo de mecanizados hace que la industria europea se haya marcado como objetivo primordial mejorar la productividad de estos procesos e incrementar su precisión y calidad, promoviendo la búsqueda de nuevos materiales para herramienta que se adapten mejor a estos nuevos requerimientos.

De los materiales para herramienta que se emplean en la actualidad, los cermets son los que mejor se adaptarían a las exigencias de estos mecanizados, ya que poseen una alta resistencia al desgaste, una estabilidad química elevada y una resistencia mecánica que se mantiene a alta temperatura. Pero, sería necesario mejorar ostensiblemente la tenacidad de fractura y la tolerancia al daño hasta valores próximos a los que presentan los carburos cementados. Durante los últimos años se ha producido un continuado proceso de optimización de los cermets, modi-

ficando principalmente la microestructura y la composición química de las fases cerámicas empleadas. En el proyecto MAT2011-22981 demostramos que los cermets denominados de solución sólida completa, caracterizados por poseer una única fase cerámica homogénea formada por un carbonitruro complejo, permiten alcanzar una buena combinación de dureza y tenacidad y una alta resistencia a la oxidación.

En el presente proyecto, que puede considerarse como complementario al MAT2011-22981, se pretende mejorar aún más las propiedades de los cermets, pero actuando en este caso sobre la fase ligante, que es en última instancia la principal causante de la cohesión y la tenacidad del material. Las aleaciones de alta entropía de mezcla se postulan como candidatas idóneas para sustituir a las fases ligantes actuales, ya que presentan una alta resistencia, una buena ductilidad y un excelente comportamiento mecánico a elevadas temperaturas. El objetivo general del presente proyecto se centra en el desarrollo de cermets de solución sólida completa con una fase metálica ligante formada por aleaciones de alta entropía de mezcla. Los materiales que se desarrollarán poseerán una microestructura sencilla, similar a la que presentan los actuales carburos cementados, pero con una elevada complejidad composicional, ya que ambas fases constituyentes (cerámica y metálica) serán soluciones sólidas con un número importante de componentes, al menos cinco. Con estos nuevos cermets, se pretenden mantener las propiedades óptimas que presentan actualmente y mejorar aquellas que limitan su uso potencial en los mecanizados más exigentes.

Machining is an essential part of the manufacturing processes in many industries and has significant economic implications, as it represents an important proportion of the total manufacturing cost. The success of machining depends on many factors, among which the correct choice of the cutting tool. High-speed machining and difficult-to-cut materials, such as superalloys employed in the fabrication of aircraft engines, impose extreme working conditions to cutting-tools, which are characterized by high temperatures, pressures and tensions that can lead to the premature failure in service. Furthermore, the deterioration of the cutting-tool due to an excessive wear and deformation makes it difficult to maintain the tolerances and the surface integrity of the workpiece, severely compromising the fatigue properties and, therefore, its applicability and lifetime. The European industry has as a main objective to improve the productivity, accuracy and quality of these highly-demanding machining processes, stimulating the search for new cutting-tool materials that are better suited to these new requirements.

Cermets have properties, such as high wear resistance, high chemical stability and good mechanical strength at high temperature, well-adapted to the requirements of these machining processes. But for a realistic application, it is necessary to significantly increase the fracture toughness and damage tolerance to values close to those of cemented carbides. In the last few years, there has been an ongoing process of cermets optimization, mainly by modifying the microstructure and chemical composition of the ceramic phase. In a previous project (MAT2011-22981), we have shown that the so-called complete solid solution cermets, characterized by single phase ceramic particles consisting of a complex carbonitride, allow achieving a good combination of hardness and fracture toughness.

In this new project, which can be considered as complementary to MAT2011-22981, we propose to further improve the properties of cermets, also acting on the binder phase as it is ultimately responsible for the cohesion and toughness of the material. High entropy alloys (HEAs), which are composed of at least five major metal elements in equal or near equal atomic percent (as opposed to traditional alloy systems that are typically based on only one or two major elements), can be postulated as suitable to replace current binder phase in cermets. These alloys often exhibit superior properties than conventional alloys, including high strength and ductility at high temperature and good wear and corrosion resistances. The main goal of this project focuses on the development of complete solid solution cermets with HEAs as the binder phase. The cermets to be developed will have a simple microstructure; similar to cemented carbides, but high compositional complexity, since the two constituent phases (ceramic and binder) will be complex solid solutions with a high number of components (at least five). With these new cermets, we try to maintain their current optimal properties, while improving those limiting their potential use in the most demanding machining processes.

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Preparación por molienda reactiva de nanocomposites de interés tecnológico
Preparation of technically interesting nanocomposites by mechanochemistry

Código/Code:	P11-TEP-7858 (Proyecto de Excelencia)
Periodo/Period:	16-05-2013 / 15-05-2017
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	198.739 €
Investigador responsable/Research head:	Luis A. Pérez Maqueda
Componentes/Research group:	José Manuel Criado Luque, María Jesús Diánes Millán, José Luís Pérez Rodríguez, Juan Poyato Ferrera, Pedro Enrique Sánchez Jiménez, Antonio Perejón Pazo

RESUMEN / ABSTRACT

Los materiales compuestos nanométricos o nanocomposites presentan un gran interés tecnológico y académico. Estos materiales están constituidos por dos o más fases diferentes con un grado de interacción nanométrico pues una de las fases presenta al menos una dimensión menor de 100 nm. Ello les confiere unas propiedades que no se pueden conseguir con composites o materiales convencionales. En el presente proyecto se propone el uso de la molienda reactiva para obtener nanocomposites de diversa naturaleza. Esta estrategia es sostenible desde el punto de vista medioambiental a la vez que sencilla de aplicar y escalar. Para este propósito se pretende desarrollar, con la imprescindible colaboración de la empresa andaluza de ingeniería mecánica MC2, un molino planetario de alta energía con características específicas y únicas con respecto a los equipos actualmente disponibles en el mercado. Dicha empresa llevará a cabo además un estudio de las fuerzas que operan sobre la muestra en función de las variables operacionales del molino, lo que contribuirá a una mjor comprensión del mecanismo de las reacciones mecanoquímicas. Se llevará a cabo la síntesis por molienda reactiva de dos tipos de nanocomposites: a) nanocomposites de cobre reforzado tanto con inclusiones cerámicas como por precipitación a partir de aleaciones en base de cobre preparadas por aleado mecánico y b) nanocomposites tanto de zirconia parcialmente estabilizada (PSZ) como de zirconia totalmente estabilizada (TSZ) reforzada con alúmina. En este último caso, se propone para la sinterización de los polvos cerámicos, preparados por molienda reactiva, un método novedoso que implica calentar la muestra a la vez que se somete a un campo eléctrico. Con este procedimiento se espera una reducción drástica de la temperatura requerida para la compactación total de la zirconia. El estudio de la cinética de sinterización bajo los efectos de un campo eléctrico se llevará a cabo mediante un equipo a desarrollar en el proyecto a partir de un dilatómetro que se modificará de modo que permita seguir la evolución de la dimensión de la muestra en función de la temperatura estando ésta sometida a un campo eléctrico.

Nanocomposites are of the most academic and technical interest. Those materials consist of two or more different phases being the dimension of one of the phases smaller than 100 nm. Thus, those materials have outstanding properties as compared with conventional ones. In this project, mechanochemistry is proposed for the preparation of different nanocomposites. This preparation procedure is sustainable from the environmental point of view and easy to scale-up. In the frame of the project, a unique high energy planetary ball mill will be developed in collaboration with the MC2 enterprise. Moreover, a study of the forces as a function of the milling conditions will be performed in order to get a better understanding of the processes involved in the mechanochemical reactions. Two types of nanocomposites will be prepared: a) copper reinforced nanocomposites and b) partially and totally stabilized zirconia. In the latter case, powders will be sintered using a kind of field assisted sintering procedure. It is expected a reduction in the sintering temperature of the zirconia. The kinetics of the sintering process with and without electric field will be performed within the project using an new dilameter that will be constructed for this purpose.

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Síntesis y caracterización de materiales cerámicos no oxídicos obtenidos por descomposición de precursores poliméricos Synthesis and characterization of non oxide ceramic obtained by the thermal decomposition of polymeric precursors

Código/Code:	TEP-1900
Periodo/Period:	16-05-2014 / 15-05-2016
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	11.760 €
Investigador responsable/Research head:	Pedro E. Sánchez Jiménez
Componentes/Research group:	Antonio Perejón Pazo, Cristina García Garrido

RESUMEN / ABSTRACT

En los últimos años se ha incrementado sustancialmente el interés por las cerámicas derivadas de polímeros debido al amplísimo abanico de potenciales propiedades que presentan. Este tipo de cerámicas son más conocidas por las siglas PDC (polymer derived ceramics). Estos materiales se obtienen como producto de la descomposición térmica de un precursor polimérico, que deja como residuo una cerámica, habitualmente de naturaleza no oxídica, de tipo SiC, Si3N4, BN, etc. Las PDCs presentan una serie de propiedades termomecánicas y eléctricas de gran interés, así como una elevada resistencia a la temperatura y a la oxidación que los hacen muy adecuados para aplicaciones en condiciones extremas. Así, se han propuesto numerosas aplicaciones que abarcan desde la nanotecnología a la aeronáutica. Una importante ventaja es que dichas propiedades dependen en gran medida de las características químicas del precursor polimérico de partida así como del procedimiento de conversión en cerámica. Por tanto, es posible dirigir las propiedades de la cerámica final seleccionando cuidadosamente el precursor y las condiciones experimentales de ceramización. Además, las temperaturas necesarias para obtener materiales cerámicas por esta vía son relativamente suaves si se comparan con las necesarias mediante procesado cerámico convencional por consolidación de polvos cerámicos. Sin embargo, estos materiales presentan una limitación para ciertas aplicaciones debido a que durante la transformación en cerámica se producen defectos o fracturas que pueden llegar a hacer inservible el material. A pesar de su importancia, existen pocos estudios sistemáticos en los que se haya abordado la influencia de las condiciones de preparación en las propiedades finales de las cerámicas. En concreto, se planea utilizar los métodos de control inteligente de temperatura para el procesado de estos precursores poliméricos precerámicos. Esta metodología permite controlar con gran precisión las condiciones experimentales y ha demostrado ser muy útil para de controlar la estructura y microestructrura de productos preparados a partir de transformaciones térmicas de precursores. Así mediante el uso de los métodos de control inteligente de temperatura pretendemos obtener PDC libres de defectos, estudiar la influencia de las condiciones de la preparación en la nanoestructura de los productos y ahondar en el conocimiento de los procesos de conversión polímero-cerámica. Los productos obtenidos se caracterizarán en cuanto a su nanoestructura y propiedades, en particular la piezoresistividad, porosidad, capacidad de inserción de litio y la resistencia a la oxidación.

There has been a substantial interest during the last years in polymer derived ceramics due to the wide array of interesting properties they exhibit. This type of ceramic, best known by the acronym PDCs, are obtained by the thermal decomposition of a polymeric precursor and are mainly non oxidic, such as SiC, Si3N4, BN, etc. PDCs exhibit a wide array of thermomechanical and electrical properties of great interest, as well as a high thermal and oxidation resistance which make them promising candidates for working under extreme environmental conditions. Thus, several potential applications ranging from nanotechnology to aeronautics have been proposed. A big advantage of these materials is that their properties depend on both the chemical properties of the original polymeric precursor and the processing conditions. Therefore, by carefully selecting the precursor and the experimental degradation conditions it would be possible to tailor the properties of the final ceramic. Moreover, the temperatures needed to prepare these ceramics are much milder than those required by means of conventional ceramic processing or powder consolidation techniques. However, there is an important disadvantage that has severely limited their usability in that cracks are formed during the transformation into a ceramic so that the final pieces might be rendered unusable. Despite the important of processing, there are few systematic studies assessing the influence of ceramification conditions on the final properties. In this proposal, we plan to use smart temperature controlled methods to study the synthesis of different types of PDC. This methodology allows for great precision in the control of experimental conditions such as temperature and gas pressure and has been previously proved useful to help control the microstructure of materials synthesized by thermal transformations from precursors. Thus, we plan to use this methodology to synthesise defect-free PDCs and to study the influence of experimental conditions on the nanostructure and properties of the final ceramic material. At the same time, the information provided by the systematic study will help to better comprehend the underlying physics of the as yet poorly understood polymer-ceramic transformation. The prepared powders will be characterised in terms of nanostructure and properties such of piezoresistivity, porosity, lithium insertion capability and oxidation resistance.

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Nanogeneradores ferroeléctricos basados en polímeros para aplicaciones en generación de energía y sensores Ferroelectric polymer-based piezoelectric nanogenerators for energy harvesting and sensor applications

Código/Code:	TAPOST-134. Programa Talent HUB
Periodo/Period:	01-10-2014 / 30-09-2016
Organismo Financiador/Financial source:	Junta de Andalucía /Unión Europea
Importe total/Total amount:	152.498 €
Investigador responsable/Research head:	Pedro E. Sánchez Jiménez

RESUMEN / ABSTRACT

La generación de energía a partir de fuentes ambientales ha generado un enorme interés pues ofrece una slución energética para aplicaciones de bajo consumo tales como sensores inalámbricos, dispositivos portátiles, implantes biomédicos o dispositivos de monitorización estructural o medioambiental. Por ejemplo, se considera que el número de dispositivos de uso diario conectados a internet se estima 50.000 millones para el año 2020. La mayoría de estos dispositivos ("internet of things") son de un tamaño muy reducido o se encuentran integrados en otros equipos mayores. La manera más sostenible de proporcionar energía a estos dispositivos es la autogeneración, de tal manera que no sea necesario recargarlos durante toda su vida útil. En este sentido, la generación de energía a partir de vibraciones ambientales es particularmente atractiva pues es una fuente de disponibilidad casi ilimitada y extraordinariamente barata al producirse por fuentes tales como las partes móviles de equipos, fluidos o incluso personas. Los generadores piezoeléctricos de escala nanométrica, también conocidos como nanogeneradores, son capaces de convertir vibraciones a pequeña escala en energía eléctrica, y por tanto son candidatos para reemplazar baterías que requieran una recarga constante, las cuales no se redimensionan con facilidad a muy pequeño tamaño La generación de energía mediante nanogeneradores piezoeléctricos es una tecnología emergente y esta propuesta se basa en la preparación de materials novedosos polímero-cerámica con propiedades piezoeléctricas que puedan utilizarse para diseñar dispositivos baratos, medioambientalmente limpios y que se puedan integrar fácilmente como nanogeneradores en dispositivos electrónicos.

Harvesting energy from ambient sources in our environment has generated tremendous interest as it offers a fundamental energy solution for small-power applications including, but not limited to, ubiquitous wireless sensor nodes, portable, flexible and wearable electronics, biomedical implants and structural/environmental monitoring devices. As an example, consider that the number of smart devices linking everyday objects via the internet is estimated to grow to 50 billion by the year 2020. Most of these “Internet of Things” devices will be extraordinarily small and in many cases embedded, and will wirelessly provide useful data that will make our lives easier, better and more energy-efficient. The only sustainable way to power them is using ambient energy harvesting that lasts through the lifetime of the product, and hence the need for commercially viable small scale energy harvesters that can operate in any environment. In this context, energy harvesting from ambient vibrations is particularly attractive, as these are ubiquitously available and easily accessible, originating from ever-present sources such as the moving parts of devices and machines, fluid flow and even body movements. Nanoscale piezoelectric energy harvesters, also known as nanogenerators2, are capable of converting small-scale vibrations into electrical energy, thus offering a means of superseding batteries that require constant replacing/recharging, and that do not scale easily with size. Nanogenerators can thus pave the way for the realization of the next generation of self-powered electronic devices, with profound implications in disciplines as far-reaching as biomedicine, robotics, smart environmental monitoring and resource management, to name a few. Nano-piezoelectric energy harvesting is an emerging technology and this proposal is designed to tackle the challenge of developing novel materials with enhanced piezoelectric properties that are cheap, environment-friendly, bio-compatible and easily integrated as nanogenerators into electronic devices.

CONVENIOS Y CONTRATOS / CONTRACTS AND AGREEMENTS

Analizador térmico para estudios en condiciones realistas de funcionamiento de materiales

Código/Code:	CSIC15-CE-3316
Periodo/Period:	01-01-2016 / 31-12-2017
Organismo Financiador/Financial source:	Ministerio de Economía y Çompetitividad
Importe total/Total amount:	138.789,35 €
Investigador responsable/Research head:	Luis A. Pérez Maqueda
Componentes/Research group:	Pedro E. Sánchez Jiménez, M. Jesús Diánez Millán, José Manuel Criado Luque, Enrique Jiménez Roca, M. Carmen Jiménez de Haro

PATENTES / PATENTS

Implemento mecánico para cuchara bivalva y su uso en la ejecución de pozos verticales de gran diámetro

Inventores: M. Cano, E. Garzón, I. Pulido, P.J. Sánchez Soto
Ambito Geográfico: Patente Española, España
Patente Solicitada. Número de solicitud: P201500466
Fecha de solicitud: 31/junio/2016
Entidades Titulares: CSIC - Universidad de Huelva - Universidad de Almería

Procedimiento térmoquímico de almacenamiento de energía solar concentrada a partir de escoria de acería
Inventores: L. Pérez Maqueda, A. Perejón Pazo, J. Miranda Pizarro, P.E. Sánchez Jiménez
Ambito Geográfico: Patente Española, España
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Template-Assisted Hydrothermal Growth of Aligned Zinc Oxide Nanowires for Piezoelectric Energy Harvesting Applications

Ou, C; Sanchez-Jimenez, PE; Datta, A; Boughey, FL; Whiter, RA; Sahonta, SL; Kar-Narayan, S
ACS Applied Materials & Interfaces, **8** (2016) 13678-13683
Junio, 2016 | **DOI:** 10.1021/acsami.6b04041

A flexible and robust piezoelectric nanogenerator (NG) based on a polymer-ceramic nanocomposite structure has been successfully fabricated via a cost-effective and scalable template assisted hydrothermal synthesis method. Vertically aligned arrays of dense and uniform zinc oxide (ZnO) nanowires (NWs) with high aspect ratio (diameter similar to 250 nm, length similar to 12 mu m) were grown within nanoporous polycarbonate (PC) templates. The energy conversion efficiency was found to be similar to 4.2%, which is comparable to previously reported values for ZnO NWs. The resulting NG is found to have excellent fatigue performance, being relatively immune to detrimental environmental factors and mechanical failure, as the constituent ZnO NWs remain embedded and protected inside the polymer matrix.

The Calcium-Looping technology for CO2 capture: On the important roles of energy integration and sorbent behavior

Perejon, A; Romeo, LM; Lara, Y; Lisbona, P; Martinez, A; Valverde, JM
Applied Energy, **162** (2016) 787-807
Enero, 2016 | **DOI:** 10.1016/j.apenergy.2015.10.121

The Calcium Looping (CaL) technology, based on the multicyclic carbonation/calcination of CaO in gas-solid fluidized bed reactors at high temperature, has emerged in the last years as a potentially low cost technology for CO2 capture. In this manuscript a critical review is made on the important roles of energy integration and sorbent behavior in the process efficiency. Firstly, the strategies proposed to reduce the energy demand by internal integration are discussed as well as process modifications aimed at optimizing the overall efficiency by means of external integration. The most important benefit of the high temperature CaL cycles is the possibility of using high temperature streams that could reduce significantly the energy penalty associated to CO2 capture. The application of the CaL technology in precombustion capture systems and energy integration, and the coupling of the CaL technology with other industrial processes are also described. In particular, the CaL technology has a significant potential to be a feasible CO2 capture system for cement plants. A precise knowledge of the multicyclic CO2 capture behavior of the sorbent at the CaL conditions to be expected in practice is of great relevance in order to predict a realistic capture efficiency and energy penalty from process simulations. The second part of this manuscript will be devoted to this issue. Particular emphasis is put on the behavior of natural limestone and dolomite, which would be the only practical choices for the technology to meet its main goal of reducing CO2 capture costs. Under CaL calcination conditions for CO2 capture (necessarily implying high CO2 concentration in the calciner), dolomite seems to be a better alternative to limestone as CaO precursor. The proposed techniques of recarbonation and thermal/mechanical pretreatments to reactivate the sorbent and accelerate calcination will be the final subjects of this review.

Hot-pressing of (Ti,Mt)(C,N)-Co-Mo2C (Mt = Ta,Nb) powdered cermets synthesized by a mechanically induced self-sustaining reaction

Chicardi, E; Gotor, FJ; Medri, V; Guicciardi, S; Lascano, S; Cordoba, JM
Chemical Engineering Journal, **292** (2016) 51-61
Mayo, 2016 | **DOI:** 10.1021/acsami.6b00232

A mechanically induced self-sustaining reaction (MSR) has been successfully employed for manufacturing powdered cermets based on Ti, Ti–Ta and Ti–Nb carbonitrides using Co as the binder phase and Mo2C as the sintering additive. The powders were sintered by hot-pressing, and complete chemical, microstructural and mechanical characterizations were performed on the densified cermets. When elemental Ta, Nb and/or Mo2C were added to the initial raw mixture submitted to the MSR process, smaller ceramic grains were observed after sintering, which suggested that ceramic particle growth was hindered by the presence of Ta, Nb and/or Mo in the host titanium carbonitride structure. Nanoindentation measurements enabled the determination of the hardness of the ceramic and binder phases, and values in the range of 26–29 GPa and 14–16 GPa were found, respectively. The high hardness values of the binder were related to the formation of intermetallic phases.

A hybrid silver-magnetite detector based on surface enhanced Raman scattering for differentiating organic compounds

Caro, C; Sayagues, MJ; Franco, V; Conde, A; Zaderenko, P; Gamez, F
Sensors and Actuators B: Chemical, **228** (2016) 124-133
Junio, 2016 | **DOI:** 10.1016/j.snb.2016.01.003

In this work a cheap detector of organic molecules is developed. It comprises a cellulose fiber doped with a mixture of magnetite and reduced silver nanoparticles, the latter ones synthesized anew. The nanoparticles and the fiber were characterized with well-established spectroscopic, microscopic and magnetic techniques, namely infrared, UV–vis spectroscopies, vibrating sample magnetometry and electronic microscopies. The so-obtained doped-fibers were tested as surface enhanced Raman spectroscopy detector in aqueous samples with a diluted mixture of two pollutant models (rhodamine 6G and picric acid), being able to differentiate between both organic compounds. Hence, the nanoparticle-impregnated fiber is proposed as a reliable preliminary qualitative and semiquantitative test of the presence of specific organic molecules in solutions. Moreover, the magnetite nanoparticles provide the detector with a saturation magnetization value that enables the separation of the fiber from the solution with the aid of a commercial magnet.

Kinetics of high-temperature oxidation of (Ti,Ta)(C,N)-based cermets

Chicardi, E; Cordoba, JM; Gotor, FJ
Corrosion Science, **102** (2016) 168-177
Enero, 2016 | **DOI:** 10.1016/j.corsci.2015.10.006

The kinetics of the high-temperature oxidation of titanium–tantalum carbonitride-based cermets with different Ti/Ta ratios was studied. Isothermal oxidation tests were conducted under static air for 48 h at temperatures between 700 °C and 1200 °C. The oxidation satisfied the parabolic kinetics, characteristic of the existence of a protective oxide layer. The apparent activation energy suggests the rate-controlling process during oxidation is the simultaneous inward and outward diffusion of oxygen and titanium, respectively, through the formed protective layer, consisting mainly of a rutile phase. A higher Ta(V) content in the rutile decreased the oxygen diffusivity due to the reduction of oxygen vacancy concentration.

High temperature oxidation resistance of (Ti,Ta)(C,N)-based cermets

Chicardi, E; Cordoba, JM; Gotor, FJ
Corrosion Science, **102** (2016) 125-136
Enero, 2016 | **DOI:** 10.1016/j.corsci.2015.10.001

Cermets based on titanium-tantalum carbonitride were oxidized in static air between 800 degrees C and 1100 degrees C for 48 h. The thermogravimetric and microstructural study showed an outstanding reduction in the oxidation of more than 90% when the Ta content was increased. In cermets with low Ta content, the formation of a thin CoO/Co3O4 outer layer tends to disappear by reacting with the underlying rutile phase, which emerges at the surface. However, in cermets with higher Ta content, the formation of an external titanate layer, observed even at a low temperature, appears to prevent the oxygen diffusion and the oxidation progression.

On the relevant role of solids residence time on their CO_2 capture performance in the Calcium Looping technology

Perejon, A; Miranda-Pizarro, J; Perez-Maqueda, LA; Valverde, JM
Energy, **113** (2016) 160-171
Octubre, 2016 | **DOI:** 10.1016/j.energy.2016.07.028

The multicycle CO_2 capture performance of CaO derived from natural limestone and dolomite has been investigated by means of thermogravimetry under realistic Calcium-Looping conditions, which necessarily involve high CO_2 concentration and high temperatures in the calcination stage and fast transitions between the carbonation and calcination stages. Natural dolomite allows reducing the calcination temperature as compared to limestone while high calcination efficiency is maintained. This could help reducing the energy penalty of the CaL process thus further enhancing the industrial competitiveness for the integration of this technology into fossil fuel power plants. Importantly, the CO_2 capture capacity of the sorbents is critically affected by the solids residence time in the carbonation and calcination stages within the feasible range in practice. Thus, carbonation/calcination residence times play a critical role on the multicycle CO_2 capture performance, which has been generally dismissed in previous studies. A main observation is the enhancement of carbonation in the solid-state diffusion controlled phase, which is against the commonly accepted conception that the only relevant phase in the carbonation stage is the fast reaction-controlled stage on the surface of the solids. Thus, the CO_2 capture efficiency may be significantly enhanced by increasing the solids residence time in the carbonator.

Study by DSC and HRTEM of the aging strengthening of Cu-Ni-Zn-Al alloys

Dianez, MJ; Donoso, E; Criado, JM; Sayagues, MJ; Diaz, G; Olivares, L
Materials & Design, **92** (2016) 184-188
Febrero, 2016 | **DOI:** 10.1016/j.matdes.2015.12.030

The structural changes of a Cu-12 wt.% Ni-17 wt.% Zn-1.7 wt% Al alloy as a function of the aging temperature have been studied by means of Differential Scanning Calorimetry (DSC), high resolution transmission electron microscopy (HRTEM) and hardness measurements. It has been proposed a hardening mechanism that implies the crystallization of a Ll(0) Cu2NiZn phase coherent with the matrix a phase followed, firstly, by its transformation into a Ll(2) coherent phase and, secondly, by the precipitation of this phase. It has been shown that aluminum play an important role in the precipitation hardening process because Cu2NiZn precipitates are not formed by aging a ternary Cu-Ni-Zn alloy of similar composition. It has been shown by the first time that DSC could be a powerful tool for discriminating the whole set of phase transformations undergone by alloys as a function of the annealing temperature from a single heating run.

Effect of dolomite decomposition under CO2 on its multicycle CO2 capture behaviour under calcium looping conditions

Martos, AD; Valverde, JM; Sanchez-Jimenez, PE; Perejon, A; Garcia-Garrido, C; Perez-Maqueda, LA
Physical Chemistry Chemical Physics, **18** (2016) 16325-16336
Junio, 2016 | **DOI:** 10.1039/c6cp01149g

One of the major drawbacks that hinder the industrial competitiveness of the calcium looping (CaL) process for CO2 capture is the high temperature (~930–950 °C) needed in practice to attain full calcination of limestone in a high CO2 partial pressure environment for short residence times as required. In this work, the multicycle CO2 capture performance of dolomite and limestone is analysed under realistic CaL conditions and using a reduced calcination temperature of 900 °C, which would serve to mitigate the energy penalty caused by integration of the CaL process into fossil fuel fired power plants. The results show that the fundamental mechanism of dolomite decomposition under CO2 has a major influence on its superior performance compared to limestone. The inert MgO grains resulting from dolomite decomposition help preserve a nanocrystalline CaO structure wherein carbonation in the solid-state diffusion controlled phase is promoted. The major role played by the dolomite decomposition mechanism under CO2 is clearly demonstrated by the multicycle CaO conversion behaviour observed for samples decomposed at different preheating rates. Limestone decomposition at slow heating rates yields a highly crystalline and poorly reactive CaCO3 structure that requires long periods to fully decarbonate and shows a severely reduced capture capacity in subsequent cycles. On the other hand, the nascent CaCO3 produced after dolomite half-decomposition consists of nanosized crystals with a fast decarbonation kinetics regardless of the preheating rate, thus fully decomposing from the very first cycle at a reduced calcination temperature into a CaO skeleton with enhanced reactivity as compared to limestone derived CaO.

Combined TGA-MS kinetic analysis of multistep processes. Thermal decomposition and ceramification of polysilazane and polysiloxane preceramic polymers

Garcia-Garrido, C; Sanchez-Jimenez, PE; Perez-Maqueda, LA; Perejon, A; Criado, JM
Physical Chemistry Chemical Physics, **18** (2016) 29348-29360
Noviembre, 2016 | **DOI:** 10.1039/c6cp03677e

The polymer-to-ceramic transformation kinetics of two widely employed ceramic precursors, 1,3,5,7-tetramethyl-1,3,5,7-tetravinylcyclotetrasiloxane (TTCS) and polyureamethylvinylsilazane (CERASET), have been investigated using coupled thermogravimetry and mass spectrometry (TG-MS), Raman, XRD and FTIR. The thermally induced decomposition of the preceramic polymer is the critical step in the synthesis of polymer derived ceramics (PDCs) and accurate kinetic modeling is key to attaining a complete understanding of the underlying process and to attempt any behavior predictions. However, obtaining a precise kinetic description of processes of such complexity, consisting of several largely overlapping physico-chemical processes comprising the cleavage of the starting polymeric network and the release of organic moieties, is extremely difficult. Here, by using the evolved gases detected by MS as a guide it has been possible to determine the number of steps that compose the overall process, which was subsequently resolved using a semiempirical deconvolution method based on the Frasier-Suzuki function. Such a function is more appropriate that the more usual Gaussian or Lorentzian functions since it takes into account the intrinsic asymmetry of kinetic curves. Then, the kinetic parameters of each constituent step were independently determined using both model-free and model-fitting procedures, and it was found that the processes obey mostly diffusion models which can be attributed to the diffusion of the released gases through the solid matrix. The validity of the obtained kinetic parameters was tested not only by the successful reconstruction of the original experimental curves, but also by predicting the kinetic curves of the overall processes yielded by different thermal schedules and by a mixed TTCS-CERASET precursor.

Influence of Ball Milling on CaO Crystal Growth During Limestone and Dolomite Calcination: Effect on CO2 Capture at Calcium Looping Conditions

Sanchez-Jimenez, PE; Valverde, JM; Perejon, A; de la Calle, A; Medina, S; Perez-Maqueda, LA
Cryst. Growth Des., **16** (2016) 7025–7036
Diciembre, 2016 | **DOI:** 10.1021/acs.cgd.6b01228

Under Calcium-Looping “realistic” operation conditions (high CO_2 concentration and temperature), the multicycle CO_2 capture capacity performance of CaO derived from limestone and dolomite is inversely related to its crystallite size. Ball-milling the raw sorbents results in the formation of larger nascent CaO nanocrystals during the calcination. Constraint sintering effect due to inert compounds such as dolomitic MgO mitigates the inactivation.

Assessment of the performance of commonly used DFT functionals vs. MP2 in the study of IL-Water, IL-Ethanol and IL-(H2O)(3) clusters

López-López, A., Ayala, R.
Journal of Molecular Liquids, **220** (2016) 970-982
Agosto, 2016 | **DOI:** 10.1016/j.molliq.2016.05.037

We present a comparative study of the accuracy of different DFT approaches vs. MP2 for evaluating ionic liquids (ILs) + cosolvent. Namely, we are interested in [XBmim] + cosolvent (X being Cl-, BF4-, PF6-, and CH3SO3- anions and cosolvent being water or ethanol) and [XBmim] + (H2O)(3) clusters. In this study the B3LYP, B3LYP-D3, M06, M06-2X and M06-HF functionals with Pople and Dunning basis sets are considered. We find that the influence of the basis sets is a factor to take into consideration. As already seen for weakly bonded systems when the basis set quality is low the uncorrected counterpoise (unCP) or averaging counterpoise (averCP) energies must be used due to cancellation errors. Besides, the inclusion of extra diffuse functions and polarization is also required specially in the case of ILs interacting with water clusters. The B3LYP functional does not reproduce either the structure or the interaction energies for ILs + H2O and ILs + EtOH aggregates, the energetic discrepancies being more significant than the structural ones. Among the dispersive corrected functionals, M06-2X results resemble to a great extent the reference data when the unCP interaction energies are considered for both water and ethanol. In turn, M06 and B3LYP-D3 functionals are the best option for ILs containing polar and non-polar anions, respectively, whether the averCP interactions energies are taking into consideration. From the structural point of view, B3LYP and M06 functionals describe more open structures whereas B3LYP-D3, M06-2X and M06-HF structures resemble quite well MP2 results. When the number of water molecules increases the H bonding motif gains importance and the effect depends on the underlying functional. Only M06-2X and M06-HF behaviour is similar to that observed for one water molecule. This is important because to describe ILs-cosolvent solutions is not only necessary to take into account the ILs-cosolvent interactions but also the cosolvent-cosolvent ones in the ensemble of the system.

Magnesium hydride for energy storage applications: The kinetics of dehydrogenation under different working conditions

Perejon, A; Sanchez-Jimenez, PE; Criado, JM; Perez-Maqueda, LA
Journal of Alloys and Compounds, **681** (2016) 571-579
Octubre, 2016 | **DOI:** 10.1016/j.jallcom.2016.04.191

A new approach to the kinetics of magnesium hydride dehydrogenation is considered. A model able to predict the dehydrogenation under different experimental conditions has been proposed. A new combined kinetic analysis method, which considers the thermodynamic of the process according to the microreversibility principle, has been used for performing the kinetic analysis of data obtained under different thermal schedules at hydrogen pressures ranging from high vacuum up to 20 bar.

The kinetic analysis shows that the dehydrogenation mechanism of magnesium hydride depends on the experimental conditions. Thus, the reaction follows a first order kinetics, equivalent to an Avarmi-Erofeev kinetic model with an Avrami coefficient equal to 1, when carried out under high vacuum, while a mechanism of tridimensional growth of nuclei previously formed (A3) is followed under hydrogen pressure. An explanation of the change of mechanism is given. It has been shown that the activation energy is closed to the Mg-H bond breaking energy independently of the hydrogen pressure surrounding the sample, which suggests that the breaking of this bond would be the rate limiting step of the process. The reliability of the calculated kinetic parameters is tested by comparing simulated and experimental curves.

The calorimetric analysis as a tool for studying the aging hardening mechanism of a Cu-10wt%Ni-5.5wt%Sn alloy

Dianez, MJ; Donoso, E; Sayagues, MJ; Perejon, A; Sanchez-Jimenez, PE; Perez-Maqueda, LA; Criado, JM

Journal of Alloys and Compounds, **688** (2016) 288-294
Diciembre, 2016 | **DOI:** 10.1016/j.jallcom.2016.07.021

The transformations of a Cu-10wt%Ni-5.5wt%Sn alloy as a function of the aging time in the range from room temperature up to 600 degrees C have been followed by Differential Scanning Calorimetry (DSC). The results obtained have shown that this alloy undergone two overlapping exothermic phase transitions with DSC peaks at 208 degrees C and 305 degrees C, respectively, followed by an endothermic phase transformation with a DSC peak at 526 degrees C. The structural analysis by TEM, ED, EDX and XRD of the intermediates phases previously discriminated by DSC suggests that the first exothermic peak is associated to the spinodal decomposition of the sample, while the second one is associated to the segregation of a DO22 (Cu-x-Ni1-x)(3)Sn tetragonal phase coherent with the alpha-Cu structure of the starting alloy. The endothermic peak has been associated to the precipitation of cubic DO3 nanocrystals from the DO22 phase previously formed. The microhardness measurements carried out in combination with the structural characterization demonstrate that the aging hardening of the alloy under study is exclusively due to the formation of the coherent DO22 phase. The DO22/DO3 transition leads to a dramatic drop of the hardness of the alloy.

Use of steel slag for CO_2 capture under realistic calcium-looping conditions

Miranda-Pizarro, J; Perejon, A; Valverde, JM; Sanchez-Jimenez, PE; Perez-Maqueda, LA
RSC Advances, **6** (2016) 37656-37663
Junio, 2016 | **DOI:** 10.1039/C6RA03210A

In this study, CaO derived from steel slag pretreated with diluted acetic acid has been tested as a dry sorbent for CO_2 capture under realistic Ca-Looping (CaL) conditions, which necessarily implies calcination under high CO_2 partial pressure and fast transitions between carbonation and calcination stages. The multicycle capture performance of the sorbent has been investigated by varying the precalcination time, carbonation/calcination residence times and with the introduction of a recarbonation stage. Results show that the sorbent can be regenerated in very short residence times at 900 °C under high CO_2 partial pressure, thus reducing the calciner temperature by about 30–50 °C when compared to limestone. Although the introduction of a recarbonation stage to reactivate the sorbent, as suggested in previous studies for limestone, results in a slightly enhanced capture capacity, the sorbent performance can be significantly improved if the main role of the solid-state diffusion-controlled carbonation is not dismissed. Thus, a notable enhancement of the capture capacity is achieved when the carbonation residence time is prolonged beyond just a few minutes, which suggests a critical effect of solids residence time in the carbonator on the CO_2 capture efficiency of the CaL process when integrated into a power plant.

Targeted multifunctional tannic acid nanoparticles

Aguilera, J. R.; Venegas, V.; Oliva, J. M.; Sayagues, M. J.; de Miguel, M.; Sanchez-Alcazar, J. A.; Arevalo-Rodriguez, M.; Zaderenko, A. P.
RSC Advances, **9** (2016) 108611-108620
Junio, 2016 | **DOI:** 10.1039/c5ra19405a

Tannic acid (TA) has multiple effects against cancer, being especially promising in those types that overexpress the epidermal growth factor receptor (EGFR), as TA modulates its activation and downstream signaling pathways, triggering apoptosis. Nonetheless, despite the important role of this receptor in the pathogenesis and progression of a wide variety of tumors, no TA systems targeted to this receptor have been described yet. In this work, we synthesize, characterize by physico-chemical techniques and study the cytotoxic effect and cell uptake of TA nanoparticles targeted to EGFR in both tumoral and normal human skin cells. Our nanoparticles exhibited an extremely high entrapment efficiency, and were only toxic for the tumoral cells. The uptake assay demonstrated that nanoparticles are able to enter the cells through a receptor-mediated mechanism. Furthermore, we have included fluorescent markers in these nanoparticles to combine imaging and therapeutic applications, thus building effectively a multifunctional tool for biomedicine.

Constant rate thermal analysis of a dehydrogenation reaction

Perejon, A; Perez-Maqueda, LA; Sanchez-Jimenez, PE; Criado, JM; Murafa, N; Subrt, J
RSC Advances, **6** (2016) 81454-81460
Julio, 2016 | **DOI:** 10.1039/C6RA10157G

The Constant Rate Thermal Analysis (CRTA) procedure has been employed for the first time to study the kinetics of MgH_2 dehydrogenation by thermogravimetry under high vacuum. CRTA implies controlling the temperature in such a way that the decomposition rate is maintained constant all over the process, employing the mass change as the experimental signal proportional to the reaction rate. The CRTA curves present a higher resolution power to discriminate the kinetic model obeyed by the reaction in comparison with conventional heating rate curves. The combined kinetic analysis has been applied to obtain the kinetic parameters, which show that MgH_2 decomposition under high vacuum obeys first-order kinetics (F1). It has been proposed that the dehydrogenation of MgH_2 under high vacuum takes place by instantaneous nucleation in the border line of the bidimensional crystallites followed by the growth of the nuclei.

Effect of lime on stabilization of phyllite clays

Garzon, E; Cano, M; O'Kelly, BC; Sanchez-Soto, PJ
Applied Clay Science, **123** (2016) 329-334
Abril, 2016 | **DOI:** 10.1016/j.clay.2016.01.042

This paper represents a new advance in the study of engineering properties and material applications of phyllite clays. Considering their potential use as construction materials for structures subjected to low stress levels, this laboratory research investigated the stabilization and improvement in engineering properties of a Spanish phyllite clay achieved by the addition of 3, 5 and 7 wt.% lime. Geotechnical properties investigated include the consistency limits, compaction, California Bearing Ratio, swelling potential and water-permeability. The phyllite clay–lime mixtures had good compaction properties and very to extremely low permeability-coefficient values, with a semi-logarithmic correlation between increasing permeability and increasing proportion of lime additive. The addition of 3 wt.% lime was sufficient to reach the index of capacity amble specified in the Sheet of Technical General Prescriptions for Works of Roads and Bridges PG–3 (Spanish Highways Agency, 2008), significantly reducing the plasticity index value, with the compacted mixture undergoing no swelling under soakage. The required pavement thicknesses for the raw phyllite–clay material and the phyllite clay–lime mixtures are compared and discussed. Potential applications for phyllite clay–lime mixtures include for pavements/road subgrade, earth construction, building materials and for impermeabilization purposes.

Ceramics from clays and by-product from biodiesel production: Processing, properties and microstructural characterization

Martinez-Martinez, S; Perez-Villarejo, L; Eliche-Quesada, D; Carrasco-Hurtado, B; Sanchez-Soto, PJ; Angelopoulos, GN
Applied Clay Science, **121-122** (2016) 119–126
Marzo, 2016 | **DOI:** 10.1016/j.day.2015.12.003

The production of biodiesel generates a by-product called glycerine which contains glycerol that cannot be reintegrated into the same manufacturing process. The ceramic bricks are an interesting option to set in their structure a wide range of by-products and residues materials and composites, sometimes serving only as a reservoir for the inert residue, and other, having a positive effect on the ceramic material or process. In the present work, the incorporation of this waste in raw clays has been studied. The raw materials: clay and glycerine was characterized by XRD, XRF, CNHS analysis, higher heating value and thermal analysis and after, using conventional moulding and sintering processing methods to prepare clay-glycerine composites, the influence of the amount of waste added to clay has been evaluated. To do this, percentages of glycerine were added to the clay from 5% to 20% and evaluated by a series of technological properties such as compressive strength, absorption and suction of water, bulk density, the study of porosity generated by adsorption-desorption isotherms of N-2, thermal conductivity and finally by the compressive strength after freezing-thaw, it was considered as the maximum permissible rate of addition of glycerol 10-15% in weight, because higher additions have a strong effect on the properties of the obtained materials such as compression strength and bulk density which descending dramatically due to the large amount of porosity generated as reflected by high values of absorption and suction experiments. It was concluded that adding 5% glycerol to the ceramic paste generated plasticity in clay to achieve be moulded, with values of compressive strength of 84 MPa while gets to reduce the density apparent by almost.

Correlation between chemical and mineralogical characteristics and permeability of phyllite clays using multivariate statistical analysis

Garzon, E; Romero, E; Sanchez-Soto, PJ
Applied Clay Science, **129** (2016) 92-101
Agosto, 2016 | **DOI:** 10.1016/j.clay.2016.05.008

Phyllite clays are applied as a layer on a surface to be waterproofed and subsequently compacted. For this purpose, phyllite clays deposits can be grouped by their chemical and mineralogical characteristics, and these characteristics can be connected with their properties, mainly permeability, in order to select those deposits with the lowest permeability values. Several deposits of phyllite clays in the provinces of Almeria and Granada (SE Spain) have been studied. The results of applying a multivariate statistical analysis (MVA) to the chemical data analysed from 52 samples determined by XRF, mineralogical analysis by XRD and permeability are reported. Permeability, a characteristic physical property of phyllite clays, was calculated using the results for experimental nitrogen gas adsorption and nitrogen adsorption-desorption permeability dependence. According to the results, permeability values differentiated two groups, i.e. group 1 and group 2, with two subgroups in the latter. The influence of chemical as well as mineralogical characteristics on the permeability values of this set of phyllite clays was demonstrated using a multiple linear regression model. Two regression equations were deduced to describe the relationship between adsorption and desorption permeability values, which support this correlation. This was an indication of the statistical significance of each chemical and mineralogical variable, as it was added to the model. The statistical tests of the residuals suggested that there was no serious autocorrelation in the residuals.

On the Multicycle Activity of Natural Limestone/Dolomite for Thermochemical Energy Storage of Concentrated Solar Power

Sarrion, B; Valverde, JM; Perejon, A; Perez-Maqueda, L; Sanchez-Jimenez, PE
Energy Technology, **4** (2016) 1013-1019
Agosto, 2016 | DOI: 10.1002/ente.201600068

Cheap, efficient, and non-toxic energy storage technologies are urgently needed to handle the rapidly increasing penetration of intermittent renewable energies into the grid. This work explores the use of limestone and dolomite for energy storage in concentrated solar power (CSP) plants by means of the calcium looping (CaL) process based on the multicycle carbonation/calcination of CaO. An efficient integration of the CaL process into CSP plants involves high temperature carbonation and calcination at moderate temperatures in a close CO2 cycle for power generation. These conditions differ from those of the CaL process for CO2 capture, which lead to CaO deactivation as extensively reported in recent years. In contrast, we show that limestone- and dolomite-derived CaO give rise to a high residual conversion at CaL-CSP conditions and in short residence times, which would facilitate the development of a competitive and clean CSP technology with permanent energy storage.

Synthesis and characterization of CuInS2 nanocrystalline semiconductor prepared by high-energy milling

Dutkova, E; Sayagues, MJ; Briancin, J; Zorkovska, A; Bujnakova, Z; Kovac, J; Kovac, J; Balaz, P; Ficeriova, J
Journal of Materials Science, **51** (2016) 1978-1984
Febrero, 2016 | **DOI:** 10.1007/s10853-015-9507-x

Nanocrystalline CuInS2 particles have been synthesized from copper, indium, and sulfur powders by high-energy milling in a planetary mill in an argon atmosphere. Structural characterization of the prepared nanoparticles, including phase identification, Raman spectroscopy, specific surface area measurement, and particle size analysis were performed. The optical properties were studied using UV-Vis absorption and photoluminescence (PL) spectroscopy. The production of CuInS2 (JCPDS 027-0159) particles with a crystallite size of about 17.5-23.5 nm was confirmed by X-ray diffraction. The crystal structure has a tetragonal body-centered symmetry belonging to the I-42d space group. The Raman spectra also proved the formation of pure CuInS2 nanoparticles. TEM and HRTEM measurements revealed the presence of nanoparticles of different dimensions (10-20 nm) and their tendency to form agglomerates. The nanoparticles tend to agglomerate due to their large specific surface area. The average size of the synthesized particles was determined by photon cross-correlation spectroscopy to be in the range of 330-530 nm (bimodal size distribution). The band gap of the CuInS2 particles is 2 eV which is wider than that in bulk materials. The decrease in size leads to the blue-shift of the PL spectra. Therefore, CuInS2 nanoparticles are promising candidates for optical applications, and they have high potential in solar energy conversion.

Mechanochemically synthesized nanocrystalline ternary CuInSe2 chalcogenide semiconductor

Dutkova, E; Sayagues, MJ; Kovac, J; Kovac, J; Bujnakova, Z; Briancin, J; Zorkovska, A; Balaz, P; Ficeriova, J

Materials Letters, **173** (2016) 182-186

Junio, 2016 | **DOI:** 10.1016/j.matlet.2016.03.051

The synthesis of nanocrystalline ternary CuInSe2 particles prepared by high-energy milling in a planetary mill in an argon atmosphere from copper, indium and selenium was reported. CuInSe2 particles crystallize in the tetragonal structure with the crystallite size of about 30.5 nm. The Raman spectrum of CuInSe2 nanoparticles shows a strong peak at 176 cm−1 corresponds to the A1 phonon mode of tetragonal CuInSe2 chalcopyrite. HRTEM measurements also revealed the presence of nanocrystals with the size of 10–20 nm with the tendency to form agglomerates. The optical absorption study shows that nanoparticles have direct optical band gap energy of 1.8 eV. The quantum size effect of the particles was confirmed also by PL measurement.

A new approach to the determination of the synthetic or natural origin of red pigments through spectroscopic analysis

Franquelo, ML; Perez-Rodriguez, JL

Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy, **166** (2016) 103-111

Agosto, 2016 | **DOI:** 10.1016/j.saa.2016.04.054

This work suggests a way of differentiation between the natural or synthetic origin of inorganic materials that were historically used in the Cultural Heritage field. An exhaustive review of different reported procedures of synthesis of pigments was conducted, as well as a review of the accompanying minerals in case of natural pigments. The natural or synthetic origin of the pigments studied in this work was performed through the characterization of the accompanying minerals, in the case of the natural pigments, or the trace elements that are present as part of synthesis by-products or washing/purifying reagents and/or reactants that have only been partly removed in the final steps of these processes. This work characterized red pigments due to their wide variety, complexity and possibility of use in different mixtures. The following pigments were studied: cinnabar-vermilion, red lead and iron pigments. Also mixtures of these pigments between them and with red lake were also studied. Natural cinnabar was accompanied by silicon oxide (opal, chalcedony or quartz), calcite, clay minerals and, sometimes, pyrite. K together with S indicated a synthetic pigment (vermilion) obtained through the wet method. Nevertheless, K has not been found in layers containing only vermilion in our samples. The presence of Sn in some cases indicated vermilion that came from the dry process. K from the synthesis always appeared in the red lead pigment. The red natural ochre was confirmed by presence of clay minerals and iron. It should be said that Ca and S, and sometimes Al and K, were usually found in Mars red pigment. The presence of Al and Ca allowed the identification of carmine lake.

Surface functionalizing of a lipid nanosystem to promote brain targeting: step-by-step design and physico-chemical characterization

Cozar-Bernal, MJ; Garcia-Esteban, E; Sanchez-Soto, PJ; Rabasco, AM; Gonzalez-Rodriguez, ML

Pharmaceutical Development and Technology, **21** (2016) 823-831
Noviembre, 2016 | **DOI:** 10.3109/10837450.2015.1063651

The use of lipid nanosystems as drug delivery to the central nervous system may be advantageous over the current strategies. The aim of this study was to develop and characterize functionalized liposomes for treatment of brain diseases. The covalent method of coupling IgG to liposomes via the derivatized lipid 1,2-dioleoyl-sn-glycero-3-phosphoethanolamine-N-[4-(p-maleimidophenyl)butyramide](MPB-PE) was investigated. Optimized coupling conditions are shown to result in the efficient conjugation of IgG to liposomes containing low concentrations of MPB-PE (3/1 SH:IgG). The qualitative analysis has shown that after the extrusion process, more homogeneous populations of vesicles have been obtained with a nanometric size suitable to be effective to further anchor the protein. Negative values of zeta potential demonstrate that they are stable systems. Lyophilization was used to maintain the stability of the formulation. These very interesting results encourage further investigations to formulate peptide- and protein-loaded immunoliposomes, making targeting of liposomes as an attractive approach for brain drug delivery.

An innovative combination of non-invasive UV–Visible-FORS, XRD and XRF techniques to study Roman wall paintings from Seville, Spain

Garofano, Isabel; Luis Perez-Rodriguez, Jose; Dolores Robador, Maria; Duran, Adrian
Journal of Cultural Heritage, **21** (2016) 1028-1039
Noviembre, 2016 | **DOI:** 10.1016/j.culher.2016.07.002

This study attempts to establish the advantages and limitations of the combined use of portable UV–Vis-FORS and XRF-XRD portable equipment for the non-invasive characterisation of pigments from Roman wall paintings from Seville, Spain, dated to the first and second century AD. XRD revealed the presence of calcite, dolomite and aragonite, indicating the colour white. Egyptian blue was identified using FORS and XRF, and additional information was obtained with XRD. For the colour green, FORS and mainly FTIR and colorimetry enabled the distinction between glauconite and celadonite, although other techniques were necessary to classify all components of the green areas by determining the presence of cuprorivaite, chlorite and chromium. For the colours yellow and red, the presence of goethite, yellow ochre, cinnabar and haematite was confirmed using FORS and XRF in some cases; the results were corroborated by XRD. Chromatic characterisation and the values of inflection points of FORS spectra enabled a better differentiation between reddish colours (orange, brown, purple and pink). The XRD and XRF techniques revealed that violet was created by mixing red haematite and Egyptian blue and slight variations in FORS spectra confirmed this.

The Structure and Chemical Composition of Wall Paintings From Islamic and Christian Times in the Seville Alcazar

Robador, MD; De Viguerie, L; Perez-Rodriguez, JL; Rousseliere, H; Walter, P; Castaing, J
Archaeometry, **58** (2016) 255-270
Abril, 2016 | **DOI:** 10.1111/arcm.12218

Wall paintings from the Islamic epoch (10th to 12th centuries) and the Christian monarchy (14th to 16th centuries) have been recovered in discarded materials and on walls after reconstruction works in the Seville Alcazar. These paintings have spent centuries underground or under a plaster coat. Portable X-ray fluorescence (XRF) and combined XRF/X-ray diffraction (XRD) were employed in situ, as well as scanning electron microscopy (SEM-EDX), grazing angle incidence XRD and micro-Raman spectroscopy, on cross-section samples to fully characterize the materials in the wall paintings. Using these methods, the fresco technique was demonstrated, and many kinds of pigments were identified in accordance with the various periods of the history of the Alcazar, thus assessing the authenticity of all the wall paintings studied here.

Fabrication and characterization of CeO_2 pellets for simulation of nuclear fuel

Garcia-Ostos, C; Rodriguez-Ortiz, JA; Arevalo, C; Cobos, J; Gotor, FJ; Torres, Y
Nuclear Engineering and Design, **298** (2016) 160-167
Marzo, 2016 | **DOI:** 10.1016/j.nucengdes.2015.12.026

Cerium Oxide, CeO_2, has been shown as a surrogate material to understand irradiated Mixed Oxide (MOX) based matrix fuel for nuclear power plants due to its similar structure, chemical and mechanical properties. In this work, CeO_2 pellets with controlled porosity have been developed through conventional powder-metallurgy process. Influence of the main processing parameters (binder content, compaction pressure, sintering temperature and sintering time) on porosity and volumetric contraction values has been studied. Microstructure and physical properties of sintered compacts have also been characterized through several techniques. Mechanical properties such as dynamic Young's modulus, hardness and fracture toughness have been determined and connected to powder-metallurgy parameters. Simulation of nuclear fuel after reactor utilization with radial gradient porosity is proposed.

Preparation of calcium carbonate as nanoparticles from inorganic precursors and sucrose as additive with potential application as biomaterial

Takabait, F; Mahtout, L; Villarejo, LP; Hurtado, BC; Soto, PJS
Boletin de la Sociedad Española de Cerámica y Vidrio, **55** (2016) 179-184
Septiembre, 2016 | **DOI:** 10.1016/j.bsecv.2016.01.006

En esta comunicación se presentan unos primeros resultados de interés relevante sobre la obtención de carbonato de calcio precipitado como nanopartículas de los polimorfos vaterita y calcita. Se parte de precursores inorgánicos, nitrato de calcio tetrahidratado y bicarbonato de sodio, en presencia de sacarosa empleada como aditivo orgánico en disolución acuosa. Las fases cristalinas formadas se estudian mediante difracción de rayos X con un método cuantitativo y la morfología de las partículas obtenidas, mediante microscopia electrónica de barrido. Cuando no se emplea el aditivo orgánico se consigue la precipitación de calcita, polimorfo más estable termodinámicamente, como fase nanocristalina predominante (83%) mezclada con vaterita. Con una alta concentración del aditivo (67%) se obtiene vaterita como fase mayoritaria (> 98%). La utilización del aditivo en distinta proporción produce la formación de los 2 polimorfos de carbonato de calcio, siendo vaterita la fase predominante. La morfología de las partículas obtenidas muestra la formación de partículas nanoesféricas uniformes con contornos irregulares que se asocian a vaterita, así como partículas romboédricas de calcita cuando está presente, con potencial interés por su biocompatibilidad para su aplicación como biomateriales en implantes óseos.

Influence of tin additions on the precipitation processes in a Cu-Ni-Zn alloys

Donoso, EC; Dianez, MJ; Criado, JM; Espinoza, R; Mosquera, E

Revista de Metalurgia, **55** (2016) e060
Enero, 2016 | **DOI:** 10.3989/revmetalm.060

Influence of tin additions on the precipitation processes in a Cu-Ni-Zn alloys. The influence of 1.1 wt% tin additions on the precipitation hardening of Cu-11 wt% Ni-20 wt% Zn alloy was studied by Differential Scanning Calorimetry (DSC), microhardeness measurements and High Resolution Transmission Electron Microscopy (HRTEM). The calorimetric curves, in the range of temperatures analyzed, show the presence of two exothermic reactions in the ternary alloy, associated to the short-range-order development assisted by migration of excess vacancies. On the other hand, one exothermic and one endothermic reaction are observed in the quaternary alloy, associated to the formation and dissolution of Cu2NiZn precipitates, respectively. It has been show that an addition of 1.1% tin plays an important role in the formation of Cu2NiZn precipitates, responsible for the precipitation hardening of the ternary alloy.

ARTICULOS PUBLICADOS EN REVISTAS (NO SCI) / PAPERS IN NON-SCI JOURNALS

Electro Conductive Alumina Nanocomposites from different Alumina-Carbides Mixtures
L.A. Diaz, A. Fernandez, A. Okunkova, W. Solis, P. Peretyagin, F.J. Gotor, R. Torrecillas
2016 The International Conference on Nanomaterial, Semiconductor and Composite Materials (ICNSCM 2016) Book Series MATEC Web of Conferences. Vol. 65 (2016) Art. N. UNSP 02003
DOI: 10.1051/matecconf/20166502003

Study of tin amalgam mirrors by ^{119}Sn Mössbauer spectroscopy and other analytical methods
A. Lerf, F.E. Wagner, L.K. Herrera, A. Justo, A. Muñoz-Páez, J.L. Pérez-Rodríguez
Hyperfine Interactions, (2016) 237: 55
DOI: 10.1007/s10751-016-1279-4

TiTaCN-Co cermets prepared by mechanochemical technique: microstructure and mechanical properties
M. Fides, P. Hvizdos, J. Balko, E. Chicardi, F.J. Gotor
International Conference on Manufacturing Engineering and Materials. ICMEM 2016, 149 (2016) 87-93
DOI: 10.1016/j.proeng.2016.06.642

Processing and Characterization of Ti-6Al-4V Samples Manufactured by Selective Laser Melting
P. Sarria, Y. Torres, F. J. Gotor, E. Gutiérrez, M. Rodríguez, R. González, L. Hernández, E. Peón, H.
Key Engineering Materials, Vol. 704, pp. 260-268, 2016

Placa de Honor de la AEC-2015 concedida a Vicente Rives Arnau
P.J. Sánchez-Soto
Acta Científica y Tecnológica, 26 (2016) pags. 29-30

Congreso Bienal de la Sociedad Española de Cerámica y Vidrio (SECV)

P.J. Sánchez-Soto y F.J. Gotor Martínez
Químicos del Sur, 104 (2016) pags. 18-19

LIBROS Y OTRAS PUBLICACIONES / BOOKS AND OTHER PUBLICATIONS

"Cerámica y Vidrio 2016: Tradición, Modernidad y Retos para el Futuro", Libro de Resúmenes del LV Congreso de la Sociedad Española de Cerámica y Vidrio (SECV), I Congreso Bienal de la SECV, Sevilla 5-7 de octubre de 2016 (España). Edita: Sociedad Española de Cerámica y Vidrio (SECV) (2016). Editores: D. Gómez García, F.J. Gotor Martínez, J. Ramírez Rico, F. Gutiérrez Mora, B. Malmal Moshtaghion Entezari y P.J. Sánchez-Soto
ISBN: 978-84-617-5255-3

V. Moreno Megías , P.J. Sánchez-Soto, F.J. García Fernández, A. Ruiz-Conde
"Anexo 1. Análisis Arqueométrico de Materiales Cerámicos" en: La Influencia Púnica en las Mesas Turdetanas: Cerámica de Tipo Kuass en el Bajo Valle del Guadalquivir, Autor: Violeta Moreno Megías, Editorial Diputación de Sevilla (2016) pags. 219-239
ISBN: 978-84-7798-383-5

E. Garzón, F. González-Miranda, J.A. Sánchez-Garrido, P.J. Sánchez-Soto
"Bioengineering applied to the stabilization of slopes of soils in Guatemala", en: 20th International Congress on Project Management and Engineering, Editorial AEIPRO, Cartagena (España) (2016) pags. 1316-1325
ISBN: 978-84-617-4180-9

E. Garzón, L. Pérez-Villarejo, S. Martínez-Martínez, P.J. Sánchez-Soto
"Natural aging process of a film used as a cover material for greenhouses", en: 20th International Congress on Project Management and Engineering, Editorial AEIPRO, Cartagena (España) (2016) pags. 495-507
ISBN: 978-84-617-4180-9

S. Martínez Martínez, D. Eliche D, L. Pérez-Villarejo, G.N. Angelopoulos, P.J. Sánchez-Soto
"Valorización de chamota residual derivada de la producción de ladrillos de construcción cerámicos para el desarrollo de un nuevo tipo de Clinker de cemento belítico", en: Libro de las comunicaciones técnicas al Congreso Nacional de Medio Ambiente (CONAMA 2016), Editorial Fundación CONAMA, España (2016) pags. 1-10
ISBN: 978-84-7390-

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

The International Conference on Nanomaterial, Semiconductor and Composite Materials ICNSCM2016

21 - 22 mayo [República de Singapur]

Electro conductive alumina nanocomposites from different alumina-carbides mixtures
L.A. Díaz, A. Fernández, A. Okunkova, W. Solís, P. Peretyagin, F.J. Gotor, R. Torrecillas
Comunicación oral

International Conference on Manufacturing Engineering and Materials ICMEM 2016

6 - 10 junio [Nový Smokovec, República Eslovaca]

TiTaCN-Co Cermets Prepared by Mechanochemical Technique: Microstructure and Mechanical Properties
M. Fides;P. Hvizdoš;J. Balko;E. Chicardi;F. J. Gotor
Comunicación oral

XXV International Materials Research Congress

14 - 19 agosto [Cancún, México]

Influence of milling conditions on the amorphization degree of titanium alloys
E. Chicardi, F.J. Gotor, M.J. Sayagués, C. Aguilar
Poster

11th International Mining History Congress-XI Congreso Internacional de Historia de la Minería

6 - 11 septiembre [Linares, España]

Arcillas sericíticas, pizarras alumínicas o tierras blancas: desarrollo histórico de las investigaciones realizadas sobre diversos yacimientos pensinsulares-Sericitic clays, aluminium shales of "white earths": Historic development of the research conducted on several Peninsular Spanish deposits
S. Martínez-Martínez, J.Ma. Rincón López, L. Pérez-Villarejo, J.M. Galdón-Requena, P.J. Sánchez-Soto
Comunicación oral

Don Manuel Saldaña y Pineda, Marqués de San Antonio: un noble utrerano como Subdelegado del Superintendente del Rey en lamina de azogues de Huancavelica (Perú) y su labor en el

periodo 1743-1748-Don Manuel Saldaña y Pineda, I Marquis of San Antonio: A noble from Utrera (Sevilla) as Subdelegate of the Superintendent of the Spanish King at the "Royal Mine of Azogue" of Huancavelica (Perú) and his labour in the 1743-1748 period
S. Martínez-Martínez S, P. Sánchez Núñez, L. Pérez-Villarejo, P.J. Sánchez-Soto
Comunicación oral

Yacimientos de Vermiculitas en Andalucía: Desarrollo histórico de las investigaciones realizadas-Deposits of vermiculite in Andalousie: Historic development of the previous performed research
S. Martínez-Martínez, M.A. Avilés Escaño, L. Pérez-Villarejo, D. Eliche Quesada, P.J. Sánchez-Soto
Comunicación oral

Yacimientos minerales de arcillas y otras materias primas de interés cerámico de Andalucía: una perspectiva histórica de las investigaciones realizadas-Clay minerals deposits and other raw materials of ceramic interest in Andalousie: a Historical perspective of investigations
S. Martínez-Martínez, J.M. Galdón-Requena, L. Pérez-Villarejo, D. Eliche Quesada, P.J. Sánchez-Soto
Comunicación oral

6th EuCheMS Chemistry Congress
11 - 15 septiembre [Sevilla, España]

Chemical microcharacterisation of high entropy alloys acting as binder phase in Ti(C,N)-based cermets
A.G. de la Obra, M.A. Avilés, M.J. Sayagués, F.J. Gotor
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CONGRESOS Y REUNIONES NACIONALES / NATIONAL CONGRESS AND MEETINGS

PARTICIPACIÓN EN LA ORGANIZACIÓN DE CONGRESOS Y REUNIONES / PARTICIPATION IN ORGANISING AND MEETINGS

LV Congreso de la Sociedad Española de Cerámica y Vidrio (SECV) y I Congreso Bienal de la SECV
5 – 7 octubre [Sevilla, España]
Diego Gómez García [Miembro del Comité Organizador)
Joaquín Ramírez Rico [Miembro del Comité Organizador)
Pedro José Sánchez Soto {Miembro del Comité Organizador)
Francisco José Gotor Martínez [Miembro del Comité Organizador)
Felipe Gutiérrez Mora [Miembro del Comité Organizador)

COMUNICACIONES / COMMUNICATIONS

XIV Congreso Nacional de Materiales

8 - 10 junio [Gijón, España]

Outstanding improvement of the high temperature oxidation resistance of cermets based on (Ti,Ta)(C,N)
A.G. de la Obra, M.A. Avilés, E. Chicardi, C. Real, M.D. Alcalá, P.J. Sánchez-Soto, M.J. Sayagués, J.M. Córdoba, F.J. Gotor
Comunicación oral

Síntesis por aleado mecánico de aleaciones de alta entropía de mezcla de interés como fase cementante en cermets basados en carbonitruro de titanio
A.G. de la Obra, M.A. Avilés, E. Chicardi, C. Real, M.D. Alcalá, P.J. Sánchez-Soto, M.J. Sayagués, J.M. Córdoba, F.J. Gotor
Comunicación oral

Estudio de los procesos de difusión en multicapas de materiales compuestos cerámica-metal y sus efectos en la adherencia entre las capas
J.M. Córdoba, F.J. Gotor, M.A. Avilés, A.G. de la Obra
Poster

Aplicación de óxidos mixtos con estructura perovskita para electrodos de pilas cerámicas de combustible obtenidos por vía mecanoquímica
M.J. Sayagués, R. Moriche, M. Pueyo, F.J. García, F.J. Gotor
Poster

LV Congreso de la Sociedad Española de Cerámica y Vidrio (SECV) Y I Congreso Bienal de la SECV

5 - 7 octubre [Sevilla, España]

Influencia de la velocidad de molienda en la homogeneidad composicional de un carbonitruro complejo de Ti, Ta y Nb, sintetizado mediante reacción de auto-propagación inducida por energía mecánica
E. Chicardi Augusto, A. García de la Obra, F. J. Gotor Martínez
Comunicación oral

Pigmentos con mercurio y plomo en pinturas murales medievales de iglesias de Eslovenia
A. Kriznar, P.J. Sánchez-Soto
Comunicación oral

Valorización de escorias de aluminio de segunda fusión para la fabricación de materiales cerámicos, refractarios y vitrocerámicos
L. Pérez-Villarejo, E. Bonet-Martínez, D. Eliche, S. Martínez, P.J. Sánchez-Soto
Comunicación oral

Actividades de la Escuela "Della Robbia" de Artes y Artesanías de Andalucía, Gelves (Sevilla), dentro del Patrimonio Cultural de Cerámica y Vidrio
J.J. Lupión Alvarez, P.J. Sánchez-Soto
Poster

Aplicación de RCD en la fabricación de morteros y hormigones
J.L. Martínez Jurado, L. Pérez Villarejo, D. Eliche Quesada, P.J. Sánchez-Soto
Poster

Aplicación de subproductos procedentes de la industria cerámica de ladrillos al desarrollo de nuevos cementos
S. Martínez Martínez, L. Pérez-Villarejo, D. Eliche Quesada, E. Garzón, J.M. Galdón Requena, P.J. Sánchez-Soto
Poster

Ánodos para pilas cerámicas de combustible: Sr0.9La0.1Ti1-xFexO3 obtenido por vía mecano-química
M.J. Sayagués, F.J. García, R. Poyato, F.J. Gotor
Poster

Arqueometría de materiales cerámicos vidriados (siglos XI-XII) procedentes de intervenciones arqueológicas en los Reales Alcázares de Sevilla
P.J. Sánchez-Soto, R. Moreno Rosa, G. Durán-Domínguez, L. Pérez-Villarejo
Poster

Bloques cerámicos sostenibles silico-calcáreos obtenidos a partir de cenizas de biomasa y polvo de filtros de depuración de gases
D. Eliche Quesada, J. Sánchez Martínez, S. Martínez Martínez, L. Pérez Villarejo, P.J. Sánchez-Soto
Poster

Caracterización arqueométrica y por tratamiento de imagen fotográfica de dos obras singulares del pintor Bartolomé Esteban Murillo (1617-1682): estudio del soporte de naturaleza vítrea y pigmentos inorgánicos utilizados
R. Moreno, G. Durán, P.J. Sánchez-Soto
Poster

Evolución térmica de una filita de interés como materia prima cerámica
P.J. Sánchez-Soto, S. Martínez Martínez, L. Pérez-Villarejo, D. Eliche Quesada, E. Garzón Garzón
Poster

Influencia de los parámetros de molienda en la síntesis sólido-gas de TiCxN1-x por una reacción auto-sostenida inducida mecánicamente
E. Chicardi Augusto, F.J. Gotor, M.D. Alcalá, M.J. Sayagués, M.A. Avilés, J.M. Córdoba
Poster

La ausencia de Talleres Alfareros en la Comarca de la Sagra Toledana durante la Reconquista: estudio arqueométrico de materiales procedentes de intervenciones arqueológicas en la iglesia de la Natividad de Nuestra Señora en Cedillo del Condado (Toledo)
E. Domínguez, J.M. Martínez-Blanes, L. Pérez-Villarejo, P.J. Sánchez-Soto
Poster

La cerámica creativa a vista de Metaverso: caracterización tridimensional de muestras cerámicas como influencia estética para obras artísticas digitales
G. Durán Domínguez G, P.J. Sánchez-Soto
Poster

La ciudad de Sevilla como sede de Congresos de la Sociedad Española de Cerámica y Vidrio: una perspectiva histórica con hechos y personalidades desde la tradición a la modernidad con retos para el futuro
P.J. Sánchez-Soto, F.J. Gotor Martínez
Poster

Materiales compuestos filita-cemento con aplicaciones en construcción
E. Garzón Garzón, B.C. O'Kelly, L. Pérez-Villarejo, P.J. Sánchez-Soto
Poster

Obtención de Si mesoporoso a partir de cascarilla de arroz por tratamiento mecánico
M.D. Alcalá, C. González, C. Real
Poster

Propiedades tecnológicas de ladrillos de arcilla que incorporan cenizas volantes de combustión del orujillo
D. Eliche Quesada, J. Sánchez Martínez, S. Martínez, L. Pérez-Villarejo, P.J. Sánchez-Soto
Poster

Valorización de escorias de acería de arco eléctrico (EAF) para la fabricación de materiales cerámicos y vitrocerámicos
E. Bonet Martínez, L. Pérez Villarejo, D. Eliche Quesada, B. Carrasco Hurtado, J.Ma. Rincón López, P.J. Sánchez-Soto
Poster

FORMACION / TRAINING

FORMACIÓN DE GRADUADOS / MASTER DEGREE THESIS

Título:	**Obtención y caracterización de laminados Cermet/WC-Co**
Autor:	Luis Manuel González Becerra
Directores:	Francisco José Gotor Martínez
Grado:	Trabajo Fin de Carrera
Año Académico:	2015-2016 (14 marzo 2016)

Título: **Caracterización microestructural y tribo-mecánica de muestras de Ti6Al4V fabricadas combinando la fusión selectiva por láser y tratamientos termo-químicos de modificación superficial**
Autor: Rita Pérez Martínez
Directores: Francisco José Gotor Martínez
Grado: Trabajo Fin de Grado
Año Académico: 2015-2016 (4 julio 2016)

Título: **Sintesis y caracterizacion de aleaciones de alta entropia de mezcla basadas en Fe, Co y Ni**
Autor: Inmaculada García Trigo
Directores: María Dolores Alcalá González, Concepción Real Pérez
Grado: Trabajo Fin de Grado
Año Académico: 2015-2016 (5 julio 2016)

Título: **Nueva familia de cermets basada en aleaciones de alta entropía (CoFeMnNiV)**
Autor: Francisco Moreno Rodríguez
Directores: Francisco José Gotor Martínez
Grado: Trabajo Fin de Grado
Año Académico: 2015-2016 (22 septiembre 2016)

Título: **Evolución estructural del sistema LaMn1-xFexO3+y con estructura perovskita por análisis Rietveld.**
Autor: Manuel Fernández Ponce
Directores: María Jesús Sayagués de Vega
Grado: Trabajo Fin de Grado
Año Académico: 2015-2016 (23 septiembre 2016)

Título: **Obtención y Caracterización de laminados WC-Co/WC-Co**
Autor: Daniel Ordoñez Morillo
Directores: Francisco José Gotor Martínez
Grado: Trabajo Fin de Grado
Año Académico: 2015-2016 (6 octubre 2016)

Título: **Desarrollo de aleaciones de alta entropía de base FeCoNiCrV como fase cementante para cermets**
Autor: María del Carmen López González
Directores: Francisco José Gotor Martínez
Grado: Trabajo Fin de Máster
Año Académico: 2015-2016 (9 diciembre 2016)

DOCENCIA / TEACHING

Máster en Biotecnología Sanitaria

Caracterización de materiales nanoestructurados
María José Sayagués de Vega
Lugar: Universidad Pablo de Olavide

EQUIPAMIENTO CIENTÍFICO AVAILABLE EQUIPMENT

- Microdurómetro FM700 Future-Tech
- Máquina de ensayo universal AGS-J, Shimadzu
- 3 Termobalanzas CI Electronics
- Dilatómetro diferencial ADAMEL LHOMARGY DI-24
- 2 molinos planetarios PULVERISETTE 7 (Fritsch)
- 1 molino planetario PULVERISETTE 7 Premium Line (Fritsch)
- 1 molino planetario PULVERISETTE 4 (Fritsch)
- 1 molino planetario PM100 (Retsch)
- 1 molino vibratorio MM301 (Retsch)
- 1 molino vibratorio SPEX 8000
- 1 molino centrífugo PULVERISETTE (Fritsch)
- 1 molino de atrición O1HD (Union Process)
- Calorímetro SETARAM differential scanning DSC 111
- Horno horizontal alta temperatura 1800ºC AGNI
- Horno horizontal alta temperatura 1600ºC Carbolite
- Horno horizontal 1200ºC Carbolite
- Microcortadora metalográfica manual EVOLUTION (REMET)
- Microcortadora metalográfica MICROMET (REMET)
- Prensa automática metalográfica IPA30 (REMET)
- Pulidora automática LS2 (REMET)
- Analizador Termomecánico TMA 1000 (Linseis)
- Calorímetro diferencial de barrido DSC (TA instruments Q200)

MATERIALES FUNCIONALES NANOESTRUCTURADOS

NANOSTRUCTURED FUNCTIONAL MATERIALS

PERSONAL / PERSONNEL

Profesor de Investigación

- Dr. Juan Pedro Espinós Manzorro
- Dr. Hernán R. Míguez García
- Dr. Manuel Ocaña Jurado
- Dr. Agustín Rodríguez González-Elipe

Catedráticos

- Dr. José Cotrino Bautista

Investigadores Científicos

- Dr. Francisco Yubero Valencia

Científicos Titulares

- Dr. Angel Barranco Quero
- Dra. Ana Isabel Becerro Nieto
- Dra. Ana Isabel Borrás Martos
- Dra. María Aránzazu Díaz Cuenca
- Dra. Nuria O. Núñez Álvarez
- Dr. Alberto Palmero Acebedo

Profesor Visitante

- Dr. Richard M. Lambert

Investigadores Contratados

- Dra. María Alcaire Martín
- Dr. Francisco J. Aparicio Rebollo
- Dr. Mauricio E. Calvo Roggiani
- Dra. Sol Carretero Palacios
- Dr. Alberto Escudero Belmonte
- Dr. Francisco J. García García
- Dra. Dongling Geng
- Dra. Ana María Gómez Ramírez
- Dr. Yuelong Li
- Dr. Victor López Flores
- Dra. M. Carmen López Santos
- Dr. Gabriel Lozano Barbero
- Dr. Pedro A. Salazar Carballo
- Dr. Juan Ramón Sánchez Valencia

Becarios Predoctorales

- Ldo. Miguel Anaya Martín
- Lda. Sara Borrego González
- Lda. Victoria Esteso Carrizo
- Ldo. Aurelio García Valenzuela
- Ldo. Daniel González Mancebo
- Ldo. Alberto Jiménez Solano
- Ldo. José María Miranda Muñoz
- Ldo. Manuel Oliva Ramírez
- Ldo. Noe Orozco Corrales
- Lda. Liliam Beatriz Romero Sánchez
- Ldo. Andrea Rubino

Personal Contratado

- Dr. Rafael Álvarez Molina
- Dr. Juan F. Galisteo López
- Dra. Ana García Navarro
- Dr. Jorge Gil Rostra
- Lda. M. Carmen Gutiérrez Lázaro
- Ldo. Mariano Laguna Moreno
- Lda. Ana Mora Boza

Proyectos de Investigación / Research Projects

Materiales ópticos basados en nanofósforos para la próxima generación de dispositivos emisores de luz Nanophosphor-based photonic materials for next generation light-emitting devices NANOPHOM

Código/Code:	H2020-ERC-STG/0259
Periodo/Period:	12-12-2016 / 31-03-2022
Organismo Financiador/Financial source:	Unión Europea. ERC Starting Grant
Importe total/Total amount:	1.499.739 €
Investigador responsable/Research head:	Gabriel S. Lozano Barbero

RESUMEN / ABSTRACT

El desarrollo de fuentes de luz eficientes y respetuosas con el medio ambiente constituye una parte esencial de la estrategia mundial para reducir el consumo mundial de electricidad. Los diodos emisores de luz (LED de sus siglas en inglés) emergen como la alternativa a la iluminación convencional, debido a su alta eficiencia de conversión de energía, larga vida útil, conmutación rápida, robustez y tamaño compacto. Sin embargo, su implementación en la industria electrónica de consumo se ve obstaculizada por el control limitado sobre el brillo, la calidad del color y la direccionalidad de la emisión de LED que proporcionan los elementos ópticos convencionales cuyo funcionamiento se basa en la óptica geométrica.

Este proyecto explora nuevas formas de controlar las propiedades de emisión de nanomateriales luminiscentes, superando los límites impuestos por la óptica convencional, mediante el uso de arquitecturas nanofotónicas. El desarrollo de materiales ópticos fiables y escalables basados en nanofósforos permitirá un control espectral y angular fino sobre la emisión de luz, abordando las deficiencias que los LED actuales presentan. El nuevo diseño óptico de estos dispositivos estará basado en la integración de multicapas ópticas, texturas superficiales, y nano dispersores de composición, tamaño y forma controlados, para obtener materiales que posean propiedades ópticas que faciliten un control preciso de la radiación visible.

Nanophom permitirá mejorar nuestra comprensión sobre fenómenos fundamentales como la formación de modos fotónicos en medios ópticos complejos a los que se puede acoplar la luz, así como avanzar en el desarrollo de dispositivos de iluminación de estado sólido de alta eficiencia.

Energy-efficient and environmentally friendly light sources are an essential part of the global strategy to reduce the worldwide electricity consumption. Light-emitting diodes (LEDs) emerge as a key alternative to conventional lighting, due to their high power-conversion efficiency, long lifetime, fast switching, robustness, and compact size. Nonetheless, their implementation in the consumer electronic industry is hampered by the limited control over brightness, colour quality and directionality of LED emission that conventional optical elements relying on geometrical optics provide.

This project exploits new ways of controlling the emission characteristics of nanophosphors, surpassing the limits imposed by conventional optics, through the use of nanophotonic concepts. The development of reliable and scalable nanophosphor-based photonic materials will allow ultimate spectral and angular control over the light emission properties, addressing the critical shortcomings of current LEDs. The new optical design of these devices will be based on multilayers, surface textures and nano-scatterers of controlled composition, size and shape, to attain large-area materials possessing photonic properties that will enable a precise management of the visible radiation.

Nanophom will significantly advance our comprehension of fundamental phenomena like the formation of photonic modes in complex optical media to which light can couple, as well as advancing the state of the art of high-efficiency solid-state lighting devices.

Láminas flexibles nanoestructuradas de polímeros inorgánicos para el control de la luz Polymer-Inorganic Flexible Nanostructured Films for the Control of Light (POLIGHT)

Código/Code:	307081
Periodo/Period:	01-01-2012 / 30-11-2017
Organismo Financiador/Financial source:	Unión Europea. ERC Starting Grant
Importe total/Total amount:	1.497.000 €
Investigador responsable/Research head:	Hernán R. Míguez García

RESUMEN / ABSTRACT

El proyecto POLIGHT se centra en la integración de una serie de materiales inorgánicos nanoestructurados que poseen propiedades plasmónicas y/o fotónicas en películas poliméricas, los cuales constituyen un avance significativo con respecto al estado actual de la técnica en materiales fotónicos flexibles. Estas láminas, altamente adaptables, pueden actuar como espejos o filtros pasivos, selectivos en frecuencia en el rango espectral UV-visble-infrarrojo cercano, así como matrices en las que incluir especies absorbentes de luz u ópticamente activas capaces de adaptar su respuesta óptica.

El objetivo de este proyecto es doble. Por un lado se va a llenar un agujero existente en la actualidad en el campo de los materiales para la protección de radiación, que es la ausencia de láminas flexibles y adaptables en las que se puedan seleccionar de una manera drástica y precisa los rangos de longitudes de onda que se deseen bloquear o dejar pasar, según las necesidades de cada aplicación concreta.

Por otro lado el proyecto POLIGHT pretende ir un paso más allá en la integración de nanomateriales absorbentes o emisores de luz dentro de matrices poliméricas flexibles, mediante la fabricación de capas fotónicas jerárquicamente estructuradas que permiten el ajuste fino de las propiedades ópticas del conjunto. Esto se consigue como resultado de las interacciones materia-radiación, intensificadas a raíz de los efectos de localización del campo en los modos resonantes específicos.

El proyecto surge como resultado del reciente desarrollo de una serie de robustas estructuras fotónicas inorgánicas conformadas por redes porosas interconectadas capaces de alojar polímeros y heredando así las propiedades mecánicas características de los mismos.

The POLIGHT project will focus on the integration of a series of inorganic nanostructured materials possessing photonic or combined photonic and plasmonic properties into polymeric films, providing a significant advance with respect to current state of the art in flexible photonics. These highly adaptable films could act either as passive UV-Vis-NIR selective frequency mirrors or filters, or as matrices for light absorbing or optically active species capable of tailoring their optical response. The goal of this project is two-fold. In one aspect, the aim is to fill a currently existing hole in the field of materials for radiation protection, which is the absence of highly flexible and adaptable films in which selected ranges of the electromagnetic spectrum wavelengths can be sharply blocked or allowed to pass depending on the different foreseen applications. In another, the POLIGHT project seeks to go one step beyond in the integration of absorbing and emitting nanomaterials into simple flexible polymeric matrices by including hierarchically structured photonic lattices that provide fine tuning of the optical properties of these hybrid ensembles. This will be achieved by means of enhanced matter-radiation interactions that result from field localization effects at specific resonant modes. The opportunity arises as a result of the recent development of a series of robust inorganic photonic structures that present interconnected porous networks susceptible of hosting polymers and thus inheriting their mechanical properties.

Integración de Nanoestructuras Fotónicas en Celdas Solares de Colorante Integration of Photonic Nanostructures in Flexible Dye Solar Cells

Código/Code:	FP7-PEOPLE-2013-IIF Marie Curie Actions
Periodo/Period:	01-09-2014 / 31-08-2016
Organismo Financiador/Financial source:	Unión Europea
Importe total/Total amount:	173.370 €
Investigador responsable/Research head:	Hernán Míguez García
Componentes/Research group:	Yuelong Li

RESUMEN / ABSTRACT

El proyecto INPHOFLEX se enmarca en la búsqueda de ese aumento de eficiencia sin alterar completamente las propiedades de transparencia y flexibilidad. El grupo liderado por el Dr. Míguez en el Instituto de Ciencia de Materiales de Sevilla ha conseguido recientemente un aumento significativo sin perder la transparencia, mediante la introducción de estructuras fotónicas en la celda. Este proyecto continúa en esa senda de investigación y se basa en la hipótesis de que insertando nuevas estructuras ópticas flexibles en la celda se conseguirá el aumento deseado de eficiencia sin perder transparencia ni flexibilidad. Es en este contexto que se incorpora al grupo el Dr. Yuelong Li, experto en el desarrollo de celdas solares flexibles y autor de los principales trabajos en ese campo. El objetivo general del proyecto se afrontará a través de los siguientes objetivos y líneas de investigación integradas en el presente proyecto:

- Objetivo 1. Preparación de las celdas solares flexibles altamente eficientes y transparentes a través de la integración de nuevas estructuras fotónicas flexibles porosas sobre la capa de recolección de luz. Se prepará la nanoestructura óptica sobre el electrodo flexible. Las propiedades mecánicas de la estructura han de ser tales que la celda resultante debe ser estable contra la flexión y estiramiento.
- Objetivo 2. Preparación de las celdas solares flexibles altamente eficientes y transparentes a través de la integración de nanoestructuras fotónicas flexibles de polímero para que actúen como espejos traseros. Se fabricará una nanoestructura fotónica flexible para ser transferida sobre la parte trasera de la celda de manera que actúe como retroreflector. Se trata de un espejo de bajo peso y capaz de resistir la flexión de la misma sin perder calidad óptica.
- Objetivo 3. Preparación de las celdas solares altamente eficientes y flexibles mediante la integración de dispersores aleatoriamente distribuidos: diseño de una estructura desordenada que maximice la absorción de la luz y la captura electrónica a la vez que preserve parcialmente la transparencia. Se introducirán en el electrodo centros dispersores, desordenadamente distribuidos y de un tamaño y forma bien definidos, con el propósito de controlar el transporte difuso de luz a través de la celda para maximizar la probabilidad de absorción así como la eficiencia de captura electrónica. Se trabajará también para que los diseños de dispersión propuestos preserven una buena parte de la transmisión de la luz de forma difusa, de manera que la celda solar resultante deje pasar parte de la claridad.

El presente proyecto comprende el desarrollo de tanto del diseño teórico óptico de las nanoestructuras fotónicas optimizado para la obtención de la máxima eficiencia, así como la realización experimental de las nanoestructuras y de los ulteriores dispositivos fotovoltaicos.

It is the main goal of this project to bring to the host institution and the European Research Area the knowledge and technology to prepare current record flexible dye sensitized photovoltaic devices, previously developed by the candidate in South Korea and then the USA, in order to be able to further improve them, while endowing them with semi-transparency, using stretchable and bendable optical materials. The candidate has demonstrated that several key materials and processes provide better performance of bendable dye solar cells, i.e., enhanced efficiency and flexibility, by allowing the preparation of electrodes in which the electron diffusion length is longer and charge collection efficiency is consequently enhanced. However, highly efficient dye solar cells are opaque as a consequence of the particular diffuse scattering design employed to improve light absorption, which limits their application in building or automotive integrated photovoltaics. This proposal seeks to solve such drawback by introducing photonic nanostructures in different configurations, yielding both light harvesting enhancement and preserving transparency, hence placing Europe at the forefront of research in this specific area within the field of renewable energy. This final goal will be attempted through different approaches, each one challenging from the materials science perspective. Preparation of such highly efficient and transparent devices will combine the flexible solar cell processing tools previously developed by the candidate with the versatile optical material preparation techniques pioneered by the host institution. More specifically, integration of novel porous flexible photonic structures into the light harvesting layer, use of flexible mirrors attached to the back of the counter-electrode, and designed distribution of scatterers will be employed to reach the target.

PhoLED – Nanoestructuras fotónicas para dispositivos emisores de luz PhoLED – Photonic Nanostructures for Light-Emitting Devices

Código/Code:	Marie S. Curie – 2014 – IF (G.A. nº 657434)
Periodo/Period:	01-09-2015 / 31-08-2017
Organismo Financiador/Financial source:	Unión Europea
Importe total/Total amount:	158.122€
Investigador responsable/Research head:	Hernán Míguez García
Componentes/Research group:	Dongling Geng

RESUMEN / ABSTRACT

Este proyecto ha recibido financiación del Programa H2020 de la Unión Europea para la investigación, desarrollo tecnológico y demostración en virtud del acuerdo de subvención número 657434.

El proyecto PHOLED pretende superar en gran medida el rendimiento óptico de los emisores de luz concebidos para aplicaciones de iluminación que existen en el mercado actualmente, y contribuir a resolver algunas de las principales limitaciones técnicas que presenta la tecnología actual. Este proyecto tiene como objetivo la integración de nanoestructuras novedosas y emisores ópticos, tales como puntos cuánticos coloidales o nanofósforos, para producir la próxima generación de dispositivos emisores de luz en los que se dispondrá de un control espectral y angular completo sobre las propiedades de emisión. El enfoque se centra en el desarrollo de: i) nuevas rutas de síntesis para lograr nanofósforos eficientes, y ii) estrategias de preparación y transformación, basadas en texturas superficiales y dispersores coloidales, para alcanzar nanoestructuras ópticas de gran área que posean propiedades fotónicas que permitan un control preciso sobre la intensidad, la distribución angular y la calidad del color de la emisión de luz. Los resultados obtenidos en este proyecto proporcionarán un avance significativo tanto en la comprensión de fenómenos fundamentales, como en el desarrollo de dispositivos de iluminación de estado sólido versátiles de eficiencia optimizada, con el objetivo de superar los obstáculos técnicos y maximizar el rendimiento. El resultado del proyecto es doble: una expansión sustancial de la preparación de nanoestructuras ópticos para controlar la interacción luz-materia, y la realización práctica de dispositivos emisores de luz nanoestructurados con propiedades sin precedentes.

This project has received funding from the European Union's H2020 Programme for research, technological development and demonstration under grant agreement no 657434.

The PhoLED project seeks to largely surpass the optical performance of state-of-the-art light emitters devised for illumination applications and contribute to solve some of the main technical limitations that the current technology presents. This project aims at integrating novel optical nanostructures and emitters, such as colloidal quantum dots or nanophosphors, to yield the next generation of light-emitting devices in which full spectral and angular control over the emission properties will be possible. The approach focuses on the development of: i) new synthetic routes to achieve efficient nanophosphors, and ii) preparation and processing strategies, based on surface textures and colloidal scatterers, to attain large area optical nanostructures possessing photonic properties that will allow a precise control on the intensity, angular distribution and color quality of light emission. Results achieved within this project will provide significant advance both in the comprehension of fundamental phenomena as well as in the development of versatile solid-state lighting devices of optimized efficiency, aiming to overcome technical barriers and maximize performance. The project's outcome is twofold: a substantial expansion of the preparation of optical nanostructures to control light-mater interaction, and the practical realization of nanostructured lightemitting devices with unprecedented properties.

Un proceso completo integrado de vacío y plasma para la síntesis de celdas solares de perovskita planares y en 1D A full plasma and vacuum integrated process for the synthesis of high efficiency planar and 1D conformal perovskite solar cells

Código/Code:	H2020-MSCA-IF-2014, Project ID: 661480
Periodo/Period:	01-01-2016 / 31-12-2017
Organismo Financiador/Financial source:	Unión Europea
Importe total/Total amount:	158.122 €
Investigador responsable/Research head:	Angel Barranco Quero
Componentes/Research group:	Juan Ramón Sánchez Valencia

RESUMEN / ABSTRACT

Las celdas solares (CS) –dispositivos que transforman luz en electricidad- han sido objeto de numerosos estudios en las últimas décadas ya que representan una prometedora vía para aprovechar la energía solar. Recientemente, las CS basadas en perovskita están recibiendo una gran atención debido a su bajo coste y alta eficiencia. Son muy prometedoras como alternativa a las actuales, pero aún necesitan avanzar para alcanzar más alta eficiencia, durabilidad y reproducibilidad, a la vez que requieren métodos de síntesis compatibles con la producción actual de dispositivos microelectrónicos a escala de oblea de silicio. Estas recientes CS son fabricadas usualmente por métodos húmedos que presentan desventajas como contaminaciones o reacciones químicas en las intercaras que pueden llevar a un deterioro del funcionamiento de la CS.

PlasmaPerovSol tiene como objetivo principal la fabricación de una celda solar de perovskita completa mediante un proceso integrado de vacío y plasma llevado a cabo bajo el concepto de un solo reactor. Los diferentes componentes de la CS se depositarán secuencialmente en un reactor de vacío evitando la exposición de los materiales e intercaras al aire o disolventes. La tecnología de deposición asistida por plasma desarrollada por el grupo receptor permite la fabricación de películas altamente conformales sobre una amplia variedad de templates.Esta

aproximación se propone para fabricar multicapas conformales sobre materiales unidimensionales, con el que se mejorarán numerosos aspectos de las CS. Los procesos de vacío y plasma presentan como ventajas una alta reproducibilidad, pureza y control estequiométrico en la deposición. La síntesis propuesta es compatible con la producción a escala industrial y permite la fabricación de CS en sustratos procesables y flexibles. Al mismo tiempo, las bajas temperaturas utilizadas la hacen compatible con la tecnología actual de dispositivos microelectrónicos, y mediante el uso de máscaras permiten su integración en dispositivos preformados.

Photovoltaic or solar cells (SC) devices –that transform light into electricity- have been extensively studied in the last decades since they represent a promising way to exploit the sun energy. Currently, perovskite-based solar cells(SC) are receiving increasing attention due to their low cost and high efficiency. They are very promising as an alternative for the existing ones, but still need to advance to reach higher efficiency and durability and require synthesis methods compatible with the industrial production of CMOS devices at wafer scale. These recent SC are mostly fabricated via wet methods in planar architecture. Inherent to the nature of the wet approaches, usually appear several drawbacks as contaminations and chemical reactions on the interfaces that might result deterioration of the SC performance.

PlasmaPerovSol main objective is the fabrication of a complete perovskite solar cell device by a full plasma and vacuum integrated process carried out under the premises of the “one reactor concept”. Thus, the different components of the solar cell will be deposited sequentially within a vacuum reactor avoiding exposition of the materials and interfaces to air or solvents. The technology developed by the hosting group combine vacuum deposition assisted by plasma that permits the fabrication of conformal layers over a large variety of templates. This approach is also proposed here to fabricate conformal multilayers over 1D scaffold that will demonstrate the advantages of 1D-SC. Plasma and vacuum processes present as advantage the high purity and stoichiometric control on the deposition within an ample range of materials compositions. The synthesis approach is compatible with large scale industrial production and allows the fabrication of SC on processable and flexible substrates. At the same time, the low temperatures used make the approach compatible with current CMOS technology and by using masks permits their integration on preformed devices.

Superficies super-hielofóbicas para prevenir la formación de hielo en aeroplanos Super-IcePhobic surfaces to prevent ice formation on aircraft

Código/Code: H2020-TRANSPORT/0149
Periodo/Period: 01-02-2016 / 31-01-2019
Organismo Financiador/Financial source: Unión Europea
Importe total/Total amount: 86.579,04 €
Investigador responsable/Research head: Agustín R. González-Elipe

RESUMEN / ABSTRACT

La acumulación de hielo representa un grave problema para los aviones, ya que la presencia incluso de una capa apenas visible puede limitar seriamente la funcionalidad de las alas, las hélices, los parabrisas, las antenas, las rejillas de ventilación, las tomas de aire y las cubiertas. El Proyecto PHOBIC2ICE tiene como objetivo desarrollar tecnologías y herramientas de simulación predictiva para evitar o mitigar este fenómeno.

El proyecto PHOBIC2ICE, mediante la aplicación de un enfoque innovador de simulación y modelado, permitirá el diseño y fabricación de superficies hielofóbicas con funcionalidades mejoradas. Se desarrollarán varios tipos de recubrimientos poliméricos, metálicos e híbridos usando diferentes métodos de deposición. Se prepararán superficies tratadas con láser y anodizadas. En consecuencia, el proyecto se centra en la recopilación de conocimientos fundamentales sobre los fenómenos asociados con los problemas de repulsión de hielo. Este conocimiento dará una mejor comprensión del proceso de acreción de hielo en diferentes superficies modificadas y recubiertas. La infraestructura de investigación certificada (túnel de viento de hielo) y las pruebas de vuelo previstas ayudarán a desarrollar soluciones integrales para abordar la cuestión de la formación de hielo y elevarán el nivel de innovación del Proyecto.

La solución propuesta será respetuosa con el medio ambiente, contribuirá a la reducción del consumo de energía y ayudará a eliminar la necesidad de procedimientos frecuentes de deshielo sobre suelo. Esto contribuirá a la reducción del coste, la contaminación y el retraso de vuelo.

The accretion of ice represents a severe problem for aircraft, as the presence of even a scarcely visible layer can severely limit the function of wings, propellers, windshields, antennas, vents, intakes and cowlings. The PHOBIC2ICE Project aims at developing technologies and predictive simulation tools for avoiding or mitigating this phenomenon.

The PHOBIC2ICE project, by applying an innovative approach to simulation and modelling, will enable the design and fabrication of icephobic surfaces with improved functionalities. Several types of polymeric, metallic and hybrid coatings using different deposition methods will be developed. Laser treated and anodized surfaces will be prepared. Consequently, the Project focuses on collecting fundamental knowledge of phenomena associated with icephobicity issues. This knowledge will give better understanding of the ice accretion process on different coatings and modified surfaces. Certified research infrastructure (ice wind tunnel) and flight tests planned will aid in developing comprehensive solutions to address ice formation issue and will raise the Project's innovation level.

The proposed solution will be environment-friendly, will contribute to the reduction of energy consumption, and will help eliminate the need for frequent on-ground de-icing procedures. This in turn will contribute to the reduction of cost, pollution and flight delay.

Arquitecturas SOFC innovadoras basadas en operación "Triodo"
Innovative SOFC Architecture based on Triode Operation

Código/Code:	FCH-JU-2011-1
Periodo/Period:	01-09-2012 / 29-02-2016
Organismo Financiador/Financial source:	Unión Europea
Importe total/Total amount:	256.363 €
Investigador responsable/Research head:	Agustín R. González-Elipe
Componentes/Research group:	Francisco Yubero Valencia, Juan Pedro Espinós Manzorro, Angel Barranco Quero, Richard Lambert, Victor J. Rico, Ana Borrás Martos, José Cotrino, Jorge Gil, Pedro Castillero, Fran J. García, Alberto Palmero

RESUMEN / ABSTRACT

El desarrollo de celdas de combustible de óxido sólido (SOFCs) que operen con hidrocarburos como combustibles (gas natural, biocompustibles, LPG) es esencial para la comercialización a plazos cortos y medios. El desarrollo de HC SOFCs directas se enfrenta todavía a numerosos retos y problemas que surgen del hecho que los materiales del anodo operan bajo condiciones muy severas. Estas limitan el rendimiento con reacciones de reformado u oxidación, producen una desactivación rápida debido a la contaminación con carbón y generan inestabilidad asociada a la presencia de compuestos de azufre. Aunque la investigación en estos temas es intensa, no se han producido avances tecnológicos significativos respecto a mejorar la robustez del proceso, el incremento de su tiempo de vida y a la disminución de su costo.

T-CELL propone una aproximación electroquímica novedosa con el fin de abordar estos problemas mediante un esfuerzo integrado para definir, explorar, caracterizar, desarrollar y ejecutar una aproximación a la tecnología SOFC radicalmente nueva basada en una aproximación tipo “triodo”. Para ello se desarrollará una aproximación integrada basada en el desarrollo de materiales y en la aplicación de un diseño de celda innovador que permite el control efectivo de la actividad electrocatalítica bajo vapor o condiciones de reformado en seco.

La novedad del trabajo propuesto reside en el esfuerzo pionero para desarrollar nuevos materiales a base de Ni que actúen como ánodo y que presenten tolerancia al envenenamiento, así como en el desarrollo del concepto de triodo aplicado a celdas SOFC para incorporar una nueva variable en la operación de celdas de combustible.

Para demostrar la posibilidad de apilamiento en las celdas triodo, se desarrollará un apilamiento SOFC tipo triodo formado por cuatro unidades repetidas. Este sistema se evaluará con metano y vapor en presencia de una pequeña concentración de compuestos de azufre.

The development of Solid Oxide Fuel Cells (SOFCs) operating on hydrocarbon fuels (natural gas, biofuel,LPG) is the key to their short to medium term broad commercialization. The development of direct HC SOFCs still meets lot of challenges and problems arising from the fact that the anode materials operate under severe conditions leading to low activity towards reforming and oxidation reactions, fast deactivation due to carbon formation and instability due to the presence of sulphur compounds. Although research on these issues is intensive, no major technological breakthroughs have been so far with respect to robust operation, sufficient lifetime and competitive cost.

T-CELL proposes a novel electrochemical approach aiming at tackling these problems by a comprehensive effort to define, explore, characterize, develop and realize a radically new triode approach to SOFC technology means of an integrated approach based both on materials development and on the deployment of an innovative cell design that permits the effective control of electrocatalytic activity under steam or dry reforming conditions.

The novelty of the proposed work lies in the pioneering effort to apply Ni-modified materials electrodes of proven advanced tolerance, as anodic electrodes in SOFCs and in the exploitation of our novel triode SOFC concept which introduces a new controllable variable into fuel cell operation.

In order to provide a proof of concept of the stackability of triode cells, a triode SOFC stack consisting of at least 4 repeating units will be developed and its performance will be evaluated under methane and steam co-feed, in presence of a small concentration of sulphur compound.

Arquitecturas de multicapas nanostructuradas para el desarrollo de dispositivos optofluídicos sensores y procesos de funcionalización superficial avanzada (NANOFLOW) Nanostructured multilayered architectures for the development of optofluidic responsive devices, smart labers, and advanced Surface functionalization (NANOFLOW)

Código/Code:	MAT2016-79866-R
Periodo/Period:	31-12-2016 / 31-12- 2019
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	332.750 €
Investigador responsable/Research head:	Angel Barranco Quero / Francisco Yubero Valencia
Componentes/Research group:	Ana Isabel Borrás Martos, Juan Pedro Espinós Manzorro, Fabián Frutos Rayego (US), Germán de la Fuente Leis (ICTP), Fernando Lahoz Zamarro (IPNA), Ricardo Molina Mansilla (IQAC), Alberto Palmero Acebedo, Victor Joaquin Rico Gavira, Agustín R. González-Elipe

RESUMEN / ABSTRACT

NANOFlow es un projecto multidisciplinar que persigue el desarrollo de nuevos dispositivos optofluídicos mediante la integración de materiales nanostructurados multifuncionales. El proyecto está sólidamente fundamentado en la experiencia de los componentes del grupo de investigación en campos como la síntesis de películas multifuncionales, procesos avanzados de

modificación de superficies y en el desarrollo de dispositivos fotónicos multicapa. El objetivo principal de este proyecto de investigación es combinar e integrar los distintos métodos avanzados de síntesis y procesado disponibles en la fabricación de dispositivos optofluídicos singulares que sean capaces de responder a la presencia de líquidos mediante un cambio físico. La combinación de este tipo de procesos de integración junto con el desarrollo de nuevos métodos de de detección fotónica, el uso de microplasmas de gran área como fuentes de luz y de sustratos flexibles que incorporan elementos sensores definen un conjunto muy rico de posibilidades de desarrollo de aplicaciones prácticas que se explorarán en el proyecto. Además, en el proyecto también se llevarán a cabo estudios de simulación de crecimiento de películas delgadas que, en combinación con estudios de diagnosis, permitirán ajustar los procesos de crecimiento para conseguir la funcionalidades requeridas. De esta forma, el proyecto NANOFlow intenta cubrir toda la cadena tecnológica que va desde el desarrollo de nuevos materiales hasta la aplicación final incluyendo estudios de caracterización, rutas sintéticas flexibles, búsqueda de procesos alternativos de bajo costo y alto rendimiento (por ejemplo métodos de fabricación empleando plasmas a presión atmosférica), integración de dispostivos y test de éstos en condiciones reales.

Las actividades de desarrollo planteadas en NANOFlow culminan en el desarrollo de tres tipos de dispositivos innovadores como son: las etiquetas inteligentes con actividad sensora y posibilidad de usarse como sistemas de trazabilidad y anticopia (por ejemplo integrados en el empaquetado de productos alimentarios), un dispositivos optofluídico multisensor versátil y un sistema de limpieza optofluídico fotocatalítico que integra una microplasma emisor de luz, interruptores ópticos de luz UV y visible actuados por líquidos y una superficie fotocatalítica nanoestructurada. Los tres dispositivos funcionan sobre la base de una actuación o respuesta optofluídica y están diseñados para poder usarse de forma directa en sistemas de detección, manipulación y monitorización de líquidos.

Las actividades de investigación en los distintos paquetes de trabajo del proyecto y los dispositivos finales se han propuesto para responder al Reto nº 2 de la convocatoria referida a “Seguridad y calidad alimentaria”. Además, algunas de las actividades del proyecto, por ejemplo el tercer dispositivo, están también relacionados con el Reto nº 3 “Energía Segura, eficiente y limpia”. Es interesante indicar que las actividades propuestas en NANOFlow son de particular relevancia en el contexto geográfico de Andalucía donde la agricultura, la producción de alimentos y la energía son tres de los más relevantes sectores estratégicos.

NANOFlow is a multidisciplinary Project that aims the development of novel optofluidics sensing devices integrating advanced multifunctional nanostructured materials. The project is solidly grounded in the research group experience in the synthesis of nanoestructured functional thin films, advance surface treatments and development of planar photonic structures The main objective of the project is to combine and integrate the available synthetic and processing methodologies in the fabrication of optofluidic components capable of modifying their physical behavior when they are exposed to liquids. The integration of these optofluidic components together with accessory technologies based on new principles of photonic detection, large surface area microplasmas discharge as light sources or flexible substrates for the fabrication of sensing tags define an ambitious landscape of applications that will be explored in the project. Besides, the modeling of thin film growth in combination with advanced deposition diagnosis methodologies will be combined to adjust the thin film deposition processes to the desired functionalities.Therefore, NANOFlow aims to cover all the scientific-technological chain from the materials development to the final applications including advanced characterization, flexible synthetic routes, alternative low-cost and high throughput process (e.g. atmospheric plasma synthesis), device integration and testing of devices in real conditions.

The NANOFlow research activities will culminate in the development of three innovative devices, namely smart labels for sensing, traceability and anticounterfeiting applications (e.g. smart labels incorporated in food-packaging), a versatile optofluidic multisensing device and an optofluidic photocatalytic cleaning system that will integrate a large area microplasma source, liquid actuated UV/Visible optical switches and a photocatalytic nanostructured surface. All of these devices will operate under the basis of an optofluidic actuation and/or response and are designed to present clear potentialities for direct application in liquid sensing, manipulation and monitoring.

The NANOFlow research activities in the different work-packages and, particularly, the final devices are intended to have a direct impact in the Theme 2 (Seguridad and Calidad Alimentaria) of the “RETOS” defined in the call covering this project proposal.. Besides, some of the activities proposed, in particular the third device are also connected with the Theme 3 (Energía segura eficiente y limpia) of the call. It is very interesting to stress that these activities are of particular relevance in the geographical context of Andalucia where Agriculture, Food production and Energy are three of the most relevant strategic sectors.

Nuevas nanoestructuras 1d-híbridas multifuncionales para el desarrollo de nanosistemas autoalimentados New multifunctional 1D hybrid nanostructures for selfpowered nanosystems

Código/Code:	MAT2013-42900-P
Periodo/Period:	1-01-2014 / 31-12- 2016
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	54.326,53 €
Investigador responsable/Research head:	Ana Isabel Borrás Martos
Componentes/Research group:	José Cotrino Bautista, Ricardo Molina Mansilla, Juan Pedro Espinós Manzorro, Ana Isabel Borrás Martos, Angel Barranco Quero

RESUMEN / ABSTRACT

HYBR(1)D es un proyecto de carácter multidisciplinar en el que se persigue el desarrollo de nuevos materiales nanoestructurados multifuncionales que encuentren aplicación final en campos como las energías renovables, fotónica y la miniaturización de dispositivos. En el proyecto se plantea como principal objetivo el desarrollo de métodos de fabricación de nuevos materiales unidimensionales nanoestructurados como nanocables orgánicos e inorgánicos y sistemas unidimensionales heteroestructurados e híbridos. Haciendo especial hincapié en sistemas compuestos del tipo coaxial "core@shell/multi-shell" que integren componentes orgánicas, metales y óxidos. Estos materiales serán fabricados mediante una innovativa metodología que permiten su formación sobre sustratos procesables de distinta naturaleza involucrando tecnologías

escalables industrialmente. Además se propone de forma exploratoria la fabricación de membranas "compuestas" que permitan el uso de estas nanoestructuras embebidas de forma autosoportada. El segundo objetivo de este proyecto es probar la funcionalidad de estas nuevas nanoestructuras en distintas aplicaciones atendiendo al concepto de “nanosistema autoalimentado”: como sistemas de generación de energía (celdas solares y piezoelectricidad) y nanosensores. Cabe resaltar que aunque los materiales a fabricar son muy diversos, desde nanotubos de semiconductores inorgánicos (TiO2 y ZnO) a nanohilos orgánicos ("small-molecule single crystal nanowires) híbridos y heteroestruturados, los métodos de fabricación que se pretenden utilizar se basan en técnicas de vacío muy similares y facilmente acoplables. Así, las distintas nanoestructuras y heteroestructuras se fabricarán mediante cuatro técnicas principales y combinaciones de las mismas: deposición física desde fase vapor aplicada a moléculas orgánicas, deposición química desde fase vapor asistida por plasma de moléculas orgánicas y óxidos semiconductores, sputtering-dc de metales y "etching" por plasma de oxígeno. El IP y el grupo Nanotechnology on Surfaces del ICMS tienen un amplio background en la aplicación de estas técnicas para el desarrollo de sistemas del tipo láminas delgadas y recubrimientos funcionales, experiencia que se ha extendido en los últimos años al estudio de nanoestructuras 1D soportadas. El proyecto aborda toda la cadena de valor que lleva desde la síntesis de nuevos materiales a sus aplicaciones, incluyendo su caracterización avanzada e integración en dispositivos y prototipos a escala de laboratorio.

HYBR(1)D is a multidisciplinary Project that aims the development of novel multifunctional nanostructured materials for applications as renewable energy devices, photonics and device miniaturization. The main objective of the project is the development of original synthetic strategies for nanostructured 1D materials like organic and inorganic nanowires and other hybrid hetero-structured systems. Special attention will be paid to the development of coaxial “core@shell/multi-shell” structures integrating organic, metallic and oxide nanostructured components. These materials will be synthesized using an innovative methodology compatible with processable substrates of different nature that will be fully scalable to industrial production. In addition, the project also included exploratory studies about self-supported composite membranes where the nanostructured 1D materials will be embedded.

A second project objective is to probe the functionality of the novel 1D nanostructures in different applications under the global strategy that we defined as development of “selfpowered nanosystems”. These applications are: energy power generation devices (solar cells and piezoelectric nanogenerators) and nanosensors. It is worthy to notice that although the materials under study are relatively diverse, from semiconducting inorganic nanotubes (TiO2, ZnO) to organic single-crystal nanowires (“small molecules”) or hybrid heterostructures, the synthetic vacuum methodologies are, in all the cases, very similar and easily adaptable. These methodologies are physical vapor deposition (organic molecules), plasma assisted vacuum deposition (organic molecules and inorganic oxides), metal dc-sputtering and oxygen plasma etching. All of them can be used sequentially or in combination and are integrated in the same reactors. The project PI and the Nanotechnology on Surface group from the ICMS-CSIC have a solid background in the use of plasma and vacuum technology for the study of functional thin films and devices that is being extended to the field of 1D supported nanostructures in the recent years. HYBR(1)D project intend to cover all the scientific-technological chain from the materials development to the final applications including advanced characterization, flexible synthetic routes, device integration and testing at laboratory scale.

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Unidad altamente optimizada para un Sistema solar sostenible y mejorado

Highly optimized unit for a sustainable enhanced solar system HOUSESS

Código/Code:	RTC-2014-2333-3 (Programa Retos)
Periodo/Period:	3-02-2014 / 31-12- 2017
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	244.204,44 €
Investigador responsable/Research head:	Hernán Míguez García
Componentes/Research group:	Juan Francisco Galisteo López, José María Miranda Muñoz

RESUMEN / ABSTRACT

El objetivo del proyecto es el diseño, desarrollo, prototipado y validación de un sistema híbrido de cilindro parabólico termosolar- fotovoltaico que permita el almacenamiento y la gestionabilidad de la energía solar generada. Dicho sistema estará caracterizado por generar electricidad a menores costes que la tecnología estándar termosolar, de forma que el sistema integrado de concentración posea una mayor eficiencia.

El sistema híbrido consiste en un sistema cilindro parabólico termosolar y un receptor fotovoltaico de baja concentración. Entre estos dos componentes se sitúa un filtro dicroico de separación espectral, que recibe la luz reflejada del espejo primario del cilindro parabólico y permite la separación selectiva del espectro solar, dirigiendo una parte del mismo hacia el receptor fotovoltaico y el resto hacia el tubo absorbedor térmico. Dicho filtro dicroico se caracteriza por enviar al receptor fotovoltaico los fotones de luz con una longitud de onda a la cual dicho semiconductor opera más eficientemente, lo que conlleva a que el sistema integrado propuesto posee una mayor eficiencia que las tecnologías convencionales actuales, redundando en una mayor competitividad en costes. El sistema presenta además, por su parte termosolar, la capacidad de entrega de la energía de manera gestionable, permitiendo su almacenamiento para su introducción en la red eléctrica durante las franjas horarias en los que es más conveniente para el sistema.

The aim of the project is the design, development, prototyping and validation of a hybrid photovoltaic-thermosolar system that allows the storage and manageability of the generated solar energy. This integrated system will generate electricity at lower costs than standard thermosolar technology.

The hybrid system consists of a parabolic cylinder system and a low concentration photovoltaic solar receiver. Between these two components a dichroic filter is placed, which receives the reflected light from the parabolic cylinder primary mirror and allows the selective separation of the solar spectrum, letting pass a portion of the light to the photovoltaic receiver and reflecting the rest to the thermal tube receiver. Said dichroic filter sends to the photovoltaic receiver photons with wavelengths which are more efficiently absorbed by the solar cell. The thermal part of the system also shows the ability to controllably deliver power, allowing energy storage for its use in the most suitable moment of the day.

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Materiales Ópticos Avanzados para Dispositivos Optoelectrónicos más Eficientes Advanced optical materials for efficient optoelectronic devices

Código/Code: MAT2014-54852-R
Periodo/Period: 01-01-2015 / 31-12-2017
Organismo Financiador/Financial source: Ministerio de Economía y Competitividad
Importe total/Total amount: 242.000 €
Investigadores responsables/Research head: Hernán Míguez García. Manuel Ocaña Jurado
Componentes/Research group: Ana Isabel Becerro Nieto, Nuria Núñez Alvarez, Mauricio E. Calvo Roggiani, Gabriel Lozano Barbero, Juan Francisco Galisteo López, Miguel Anaya Martin

RESUMEN / ABSTRACT

El proyecto MODO se centrará en el desarrollo de materiales ópticos que optimicen el funcionamiento de dispositivos optoelectrónicos tales como celdas fotovoltaicas o dispositivos emisores de luz, mejorando así su eficiencia de conversión énergetica. El objetivo principal de esta propuesta es aumentar este rendimiento a través del control de los procesos de absorción y emisión de luz que tienen lugar en los materiales que forman estos dispositivos. Esto se logrará mediante el diseño e integración de nanoestructuras fotónicas cuyas propiedades sean, además, compatibles con los requisitos generales de fabricación y funcionamiento de estos sistemas, tales como estabilidad térmica, química y mecánica, durabilidad, procesado fácil o escalado.

En anteriores proyectos, el grupo dedicó sus esfuerzos a la realización de estudios de carácter fundamental y aplicado en los campos de diseño, preparación y caracterización de materiales ópticos, así como a la demostración experimental de la viabilidad de la integración de estos materiales en celdas solares para aumentar su eficiencia. Este proyecto tiene como meta ampliar el rango de dispositivos cuyo funcionamiento puede mejorar mediante la inclusión de estructuras que permitan controlar sus propiedades ópticas. El proyecto MODO tiene, por tanto, un marcado carácter tecnológico y pretende poner en práctica el conocimiento adquirido por el grupo solicitante durante los últimos años para mejorar el rendimiento de dispositivos optoelectrónicos de distinto tipo. Por un lado, se continuará investigando en nuevas estructuras fotónicas que otorguen alta eficiencia y más versatilidad y adaptabilidad a celdas solares de tercera generación basadas en colorantes, con énfasis en nuevos diseños que incrementen su funcionalidad. Proponemos además integrar estructuras fotónicas en celdas de perovskita de tipo haluro metal-orgánico, que acaparan la atención de investigadores y tecnólogos y para las cuales no se han realizado aún diseños ópticos específicos, con el objetivo de aumentar la recolección de luz en el rango espectral en que la celda absorbe menos y modificar su color controladamente.

Al mismo tiempo, se busca extender los conceptos estudiados previamente al campo de la iluminación para conseguir dispositivos en los que se pueda obtener un control fino de las propiedades fotocromáticas y direccionales de la luz emitida, mejorándose sus prestaciones y su eficiencia energética, aún lejos de lograrse con la tecnología actual. En este ámbito, creemos que la integración de estructuras ópticas puede permitir alcanzar el objetivo largamente perseguido de adecuar la curva espectral de emisión a la curva de percepción del ojo humano, maximizándose así el rendimiento energético del dispositivo, ya que toda aquella radiación que se emita para no ser detectada por el ojo supone una pérdida de eficiencia.

La propuesta se enmarca dentro del Reto Social denominado "Energía segura, eficiente y limpia" y tiene como objetivo desarrollar tecnología fotónica empleando herramientas de la nanotecnología y del campo de materiales avanzados, todas ellas identificadas como Tecnologías Facilitadoras Esenciales en el programa de H2020 e incluidas en la Estrategia Española de Ciencia y Tecnología.

The MODO project will focus on the development of optical materials to optimize the performance of optoelectronic devices such as solar cells or light emitting devices, thereby improving their energy conversion efficiency. The main objective of this proposal is to increase their performance by controlling light absorption and emission processes occurring in the materials composing these devices. This will be achieved through the design and integration of photonic nanostructures whose properties are also compatible with the manufacture and operation requirements of these systems, such as thermal, chemical and mechanical stability, durability, ease of processing and scale-up.

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Control ambiental y de procesos con dispositivos responsivos con capas nanoestructuradas fabricadas por tecnologías innovadoras de vacio y plasmas Environmental and process monitoring with responsive devices integrating nanostructured thin films grown by innovative vacuum and plasma technologies

Código/Code: MAT2013-40852-R
Periodo/Period: 01-01-2014 / 31-12-2017
Organismo Financiador/Financial source: Ministerio de Economía y Competitividad
Importe total/Total amount: 227.183,67 €
Investigador responsable/Research head: Agustín R. González-Elipe
Componentes/Research group: José Cotrino Bautista, Ricardo Molina Mansilla, Victor Rico Gavira, Francisco Yubero Valencia, Juan Pedro Espinós Manzorro, Alberto Palmero Acebedo, Angel Barranco Quero, Fernando Lahoz Zamarro

RESUMEN / ABSTRACT

Este proyecto persigue el desarrollo de una nueva generación de sistemas responsivos y sensores de baja dimensión que integren capas delgadas nanoestructuradas con propiedades ópticas y eléctricas controladas preparadas mediante técnicas innovadoras de vacío y plasma. Los principios básicos de la deposición física en fase vapor (PVD) en configuración oblicua (OAD)

se extenderán a técnicas de plasma y de pulverización catódica para producir capas con porosidad controlada que interactúen eficientemente con el medio. Se propone también la combinación de estas técnicas con otras tecnologías novedosas de plasma, como la deposición por plasma a presión atmosférica o mediante evaporación-polimerización, para conseguir un control estricto sobre la nanoestructura y las propiedades finales de sistemaas complejos. Mediante estas tecnologías se prepararán capas finas nanoestructuradas de metales y óxidos, multicapas apiladas y nanoestructuras tipo "composites" e híbridas que, a continuación, se caracterizarán mediante microcopías electrónicas avanzadas y de proximidad, entre otras técnicas. Para encontrar nuevas rutas de procesado de capas porosas con morfologías y propiedades "a medida" y posibilitar su escalado a nivel industrial, se propone estudiar los mecanismos fundamentales que gobiernan el crecimiento de las películas a fin de modificarlos convenientemente. Conjuntos ordenados y homogéneos de estas estructuras se emplearán como sensores de gases y líquidos a temperatura ambiente, dispositivos microfluídicos responsivos y etiquetas inteligentes. Para estas y otras aplicaciones, las capas finas porosas soportadas se funcionalizarán convenientemente con nanopartículas metálicas, cadenas moleculares ancladas o capas de materiales poliméricos. Asímismo, se contempla su apilamiento en forma de estructuras fotónicas verticales. Para la implementación de estas estructuras en forma de micro-dispositivos que actúen como sensores avanzados, se desarrollarán microreactores y sistemas responsivos mediante estrategias novedosas de integración, basadas en la deposición mediante evaporación de capas eliminables de NaCl. Estos sistemas incluirán también transductores fotónicos, eléctricos y/o electroquímicos que permitan el desarrollo de dispositivos finales capaces de detectar i) oxígeno y cloro en disolución, ii) glucosa y materia orgánica en el agua, iii) vapores y gases en aire, o iv) etiquetas inteligentes que cambien como respuesta al medio. Se prevén aplicaciones específicas para el control del medio ambiente en aire y aguas, emplazamientos industriales e invernaderos, procesos agroindustriales tales como la fermentación, así como para el seguimiento y la trazabilidad de diferentes tipos de mercancías y alimentos. Se espera que la combinación de nuevos descubrimientos científicos en el campo de la tecnología de capas delgadas y el de nuevos principios de integración a las escalas micro y nano abran nuevas áreas de investigación con alto impacto en diversos campos y tecnologías facilitadoras tales como la fotónica, la nanotecnología o los materiales avanzados, así como en tecnología de plasma y microfluídica.

This project aims at the development of a new generation of low dimensional responsive systems and sensors that integrate nanostructured layers with well-controlled electrical and optical properties which, prepared by innovative vacuum and plasma methods, present a tunable and high porosity and are able to actively interact with the environment. The basic principles of the oblique angle approach (OAD) during the physical vapor deposition (PVD) of evaporated thin films will be extended to the fabrication of similar layers by plasma and magnetron sputtering techniques. Combination of these techniques along with other innovative plasma technologies, including atmospheric pressure plasma deposition or plasma-evaporation polymerization will be employed to achieve a strict control over the nanostructure and properties of final films and complex systems . Supported metal and oxide nanostructured thin films, stacked multilayers and hybrid and composite suported nanostructures will be prepared and thereafter characterized by advanced electron and proximity microscopies and other techniques. Process-control strategies will be implemented in order to understand the fundamental mechanisms governing the film structurations and to propose new synthetic routes scalable to industrial production so as to achieve tailored morphologies and properties for these porous thin film materials.

Highly ordered and homogenous arrays of these nanostructures will be used as ambient temperature gas and liquid sensors, microfluidic responsive devices and intelligent labelling tags. For these applications the supported porous thin films will be suitably functionalized with metal nanoparticles, grafted molecular chains or layers of other polymeric materials. They will be also stacked in the form of vertically ordered photonic structures. Innovative device integration approaches including the water removal of evaporated sacrificial layers of NaCl and their integration in the form of microdevices will be carried out to fabricate advanced sensors, microreactors and responsive systems. Photonic, electrical and/or electrochemical principles of transduction will be implemented into the devices for detecting and/or fabricating i) oxygen and chlorine in solutions, ii) glucose and organic matter in water iii) gas and vapor sensors or iv) inteligent labels. Specific applications are foressen for the control of the outside environment (air and waters), industrial and greenhouse locations, agroindustrial processes such as fermentation and the tracking and trazability of different kinds of goods and foods.

It is expected that the combination of scientific breakthroughs in thin film technology and new film engineering principles at the micro- and nano-scales will open new areas of research with a high impact in key enabling technologies such as photonics, nanotechnology, advanced materials and in other fields like plasma technology and microfluidics.

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Nanoestructuras Fotónicas para Dispositivos Emisores de Luz más Eficientes y Versátiles Photonic Nanostructures For More Efficient And Versatile Leds

Código/Code:	EUIN2015-62411
Periodo/Period:	01-08-2015 / 31-07-2016
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	10.000€ €
Investigador responsable/Research head:	Hernán Míguez García
Componentes/Research group:	Gabriel Lozano Barbero

RESUMEN / ABSTRACT

La solicitud está dirigida a conseguir financiación para la preparación de una propuesta a la convocatoria Starting Grant-2016 impulsada por el European Research Council (ERC). El proyecto que se preparará y presentará a dicha convocatoria tiene como título: Nanoestructuras fotónicas para dispositivos emisores de luz más eficientes y versátiles (NanoLED) y persigue mejorar la respuesta óptica de emisores empleados en aplicaciones relacionadas con la iluminación. NanoLED pretende desarrollar nuevos materiales ópticos, que estructurados en la escala de la radiación visible, permitan controlar la emisión de fuentes de luz tales como puntos cuánticos, moléculas de colorante o nanofósforos entre otros, con una precisión sin precedentes. Se combinarán dichos emisores con materiales desarrollados ad hoc tales como materiales corrugados en superficie o materiales que integren elementos dispersores coloidales en volumen fabricados mediante técnicas de fabricación basadas en procesado en solución. La propuesta también persigue investigar la integración de tales materiales nanoestructurados en dispositivos en los que sea posible alcanzar un control fino sobre las propiedades de color y la distribución angular de la luz emitida. Los resultados obtenidos en el proyecto se espera que sienten las bases para el desarrollo de dispositos de iluminación de nueva generación que hagan un uso más efectivo de la luz emitida. Los objetivos perseguidos en la propuesta representan un paso adelante en la comprensión de fenómenos fundamentales relacionados con la interacción entre la luz y los materiales nanoestructurados, así como con en el desarrollo de dispositivos de iluminación de estado sólido versátiles y más eficientes.

This project aims to elaborate ERC Starting Grant 2016 proposal entitled: Photonic nanostructures to more efficient and versatile light-emitting devices (NanoLED), which will become granted. NanoLED project seeks to largely surpass the optical performance of state-of-the-art light emitters devised for illumination applications. This project aims to develop novel optical nanostructures and emitters, such as colloidal quantum dots, dye molecules and nanophosphors among others, to achieve an unprecedented control of their emission properties. Photonic nanostructures prepared using solution-processed techniques comprise surface corrugations and optical materials that include colloidal scatterers by design. NanoLED aims at integrating such nanostructured materials in light-emitting devices in which full control over the color quality and the angular emission profile is attained to yield more efficient and versatile devices. Results achieved within this proyect will contribute to pave the way for the development of the next generation of illumination devices that make an efficient use of the emitted light to overcome technical barriers and maximize performance. The project’s outcome is twofold: a substantial expansion on the preparation of optical nanostructures to control light-mater interaction, and the practical realization of nanostructured light-emitting devices with unprecedented properties.

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Desarrollo de recubrimientos y andamios bioactivos de material cerámico nanoestructurado para la regeneración ósea (BIOCEREG) Development of Nanostructured Ceramic Coatings and Scaffolds for Bone Regeneration (BIOCEREG)

Código/Code:	CTS-661 (Proyecto de Excelencia)
Periodo/Period:	06-07-2011 / 30-06-2016
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	240.497 €
Investigador responsable/Research head:	M. Aránzazu Díaz Cuenca
Componentes/Research group:	M. Lourdes Ramiro Gutiérrez, Sara Borrego González

RESUMEN / ABSTRACT

Este Proyecto pretende contribuir al avance en el desarrollo de nuevos biomateriales con bioactividad mejorada para su aplicación en la reparación y regeneración ósea. Se propone la preparación de nuevos recubrimientos y andamios cerámicos mediante técnicas de procesado láser a partir de materiales cerámicos particulados, nanoestructurados de composición bioactiva en el sistema SiO_2-CaO-P_2O_5 sintetizados en el ICMS por la Dra. Díaz (IP de esta solicitud). La hipótesis de partida es que las características nanoestructurales y texturales del material cerámico particulado en combinación con la fuente láser tiene un gran potencial para resultar en deposiciones con microestructura y nanoestructura controlada. Se plantea procesar dos tipos de pieza prototipo: i) piezas de sustrato metálico (de titanio) con recubrimiento cerámico de composición bioactiva y ii) piezas monolito (andamio) solo cerámica bioactiva con geometría controlada. Hay que distinguir por tanto la persecución de dos hitos. Un primer Hito es la obtención de materiales prototipo (recubrimientos y andamios) con unos requerimientos de micro y nanoestructura, superficie, propiedades mecánicas, homogeneidad y reproducibilidad mínimas. Y un segundo Hito consistente en la verificación de sus propiedades biológicas in vitro e in vivo. El éxito en ambos hitos es lo que finalmente denominaremos biomaterial prototipo. La obtención de los recubrimientos tendría una translación muy directa para implementarse en distintas partes de los componentes de implantes ortopédicos. En este sentido el equipo de investigación integra a biólogos de regeneración esquelética y clínicos de cirugía ortopédica y traumatología que van a estudiar la bioactividad y biocompatibilidad de estos recubrimientos sobre sustratos de titanio suministrados por Synthes, empresa líder en fabricación y comercialización de implantes para ortopedia, que participa en esta propuesta como EPO. Por otra parte y desde el punto de vista del procesado de materiales, la aplicación del procesado láser a los materiales cerámicos nanoestructurados SiO_2-CaO-P_2O_5 es totalmente novedoso y creemos que puede optimizarse para obtener recubrimientos y también andamios tridimensionales, conformados con macroporosidad interconectada pero a su vez con microestructura fina y nanoestructura, que culminen en la obtención de piezas prototipo con alta reproducibilidad y de calidad e innovación tecnológica elevada. El Proyecto se encuadra en áreas de investigación fuertemente innovadoras y pujantes como son la nanotecnología y la medicina regenerativa ambas con un carácter multidisciplinar intrínseco donde la frontera entre las distintas disciplinas científicas tradicionales aparece difuminada. El Proyecto aúna esfuerzos de investigadores con formación básica en las disciplinas de química (ICMS), física e ingeniería (Empresa Subcontratada AIMEN), biología (UMA-CIBER-BBN) y medicina clínica y traslacional (HCS). Creemos que la integración de estos tres pilares i) síntesis, procesado y caracterización de materiales, ii) biología de la regeneración e ingeniería tisular y iii) práctica clínica ofrece una propuesta con capacidad para aportar resultados de impacto trasferibles a la industria y que puede por tanto ayudar al desarrollo de productos para aplicaciones en la reparación y regeneración esquelética en Andalucía.

The aim of this Project is to advance in the development of new biomaterials with improved bioactivity for their application in bone repair and regeneration. The goal is the preparation of new coatings and scaffolds of ceramic materials using laser processing techniques from nanostructured ceramic particulates in the SiO_2-CaO-P_2O_5 system which will be synthesised at the ICMS. The hypothesis is the compositional properties and the textural parameters of the particulates in combination with the laser source have potential for processing depositions with controlled macro-nanostructure. It is programmed to prepare two types of prototype pieces: i)

Titanium metallic substrates with bioactive ceramic coatings and ii) monolith scaffolds of bioactive ceramic with controlled geometry. There are two milestones to highlight. The first one is the fabrication of prototype pieces (coatings and scaffolds) with reproducibility, homogeneity, micro-nanostructural features, and surface and mechanical properties requirements. A second milestone will be the evaluation of their in vitro an in vivo biological properties. The achievement of both mentioned milestones will lead to the final biomaterial prototype. Bone regeneration biologists and orthopaedic surgeons will study the bioactivity and biocompatibility of the coatings on titanium substrates provided by Synthes which is a leader Company in orthopaedic trauma devices for internal and external fixation and is included in the proposal as EPO. The application of the laser processing to the SiO_2-CaO-P_2O_5 nanostructured ceramic materials is completely new and we believe that it could be optimised for obtaining coatings and reticulated scaffolds while keeping their nanostructural features. The Project integrates material scientist, laser engineers, biologists and orthopaedic surgeons. We believe that this multidisciplinary approach with work in the i) synthesis, processing and characterisation of materials, ii) regeneration biology and tissue engineering and iii) medical practise could achieve results with potential to be transferred to the industry to promote the orthopaedic products to improve Andalusian bone repair and regeneration therapies.

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Dispositivos luminiscentes basados en láminas delgadas con tierras raras depositadas mediante tecnología de plasma (LUMEN) Luminescent devices based on rare earth containing thin films deposited by plasma technology (LUMEN)

Código/Code:	P11-TEP-8067 (Proyecto de Excelencia Motriz)
Periodo/Period:	16-05-2013 / 15-05-2016
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	143.750 €
Investigador responsable/Research head:	Angel Barranco
Componentes/Research group:	Agustín R. González-Elipe, Juan Pedro Espinós, Richard Lambert, Juan Carlos González-González, Francisco J. García García, Victor J. Rico Gavira, , Jorge Gil Rostra, Lola González García, F. Javier Ferrer (CNA), Fabián Frutos Rayego

RESUMEN / ABSTRACT

El presente proyecto persigue el desarrollo de una serie de dispositivos luminiscentes basados en láminas delgadas con tierras raras depositadas mediante tecnologías de plasma. Las láminas delgadas luminiscentes se fabricarán mediante un nuevo procedimiento desarrollado por el grupo investigador que combina procesos clásicos como "magnetrón sputtering" o deposición por plasma y la evaporación de compuestos metalorgánicos de baja o nula tensión de vapor a temperatura ambiente pero fácilmente sublimables en la cámara de reacción. Esta metodología ofrece la oportunidad de integrar uno o varios elementos de tierras raras dentro de la capa formada, controlando perfectamente su concentración en la misma. Dada la versatilidad del proceso, se plantea fabricar capas delgadas luminiscentes de interés para tres campos de aplicación: procesos de "up conversión", detectores de ion-luminiscencia y sistemas de señalización. Los materiales a desarrollar deben permitir superar algunas de las limitaciones existentes con los materiales actualmente existentes, proporcionando nuevas funcionalidades y mejoras sustanciales en sus prestaciones en relación con aplicaciones como detectores en procesos de fusión nuclear, cambiadores de longitud de onda en sistemas para comunicaciones ópticas y señalización en automoción. Dadas las características del nuevo método propuesto, el proceso de síntesis de las capas luminiscente es compatible con otros procesos en línea proponiéndose la integración de las capas luminiscentes en dispositivos fotónicos simples formadas por estructuras multicapa tipo reflectores de Bragg o similares. El proyecto aborda todo la cadena de valor que lleva de la síntesis de los materiales a sus aplicaciones, incluyendo su caracterización avanzada, el análisis de sus propiedades ópticas y de luminiscencia, su integración en dispositivos y el análisis de su resistencia medioambiental.

The objective of the LUMEN project is the development of luminescent devices incorporating as active layers rare earth containing thin films deposited by plasma CVD. The thin films will be deposited by novel synthetic approaches that combined classic approaches as magnetron sputtering and plasma CVD with the sublimation of functional molecules. This methodology is very effective to introduce a controlled amount of functional elements (i.e., rare earth cations of functional organic groups) in the growing film. Due to the full compatibility of the proposed methodology with optoelectronics processes the active layers will be directly incorporated in photonic structures as Bragg reflectors and photonic crystals to fabricate prototype devices. Thus, the LUMEN projects start with the development of new materials but also intend to study the functionality of devices that integrates these novel materials in real life conditions. These devices are intelligent label structures, up-converters and ion detectors.

Plasmas de Descarga de Barrera Dieléctrica para el Desarrollo de Procesos Industriales a Presión Atmosférica (DBD-Tech) Dielectric Barrier Discharge plasma for the developing of industrial process at atmospheric pressure (DBD-Tech)

Código/Code:	P12-FQM-2265 (Proyecto de Excelencia)
Periodo/Period:	30-01-2014 / 29-01-2017
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	143.750 €
Investigador responsable/Research head:	José Cotrino Bautista
Componentes/Research group:	Francisco García García, Jorge Gil Rostra, Richard M. Lambert, Manuel J. Macías Montero, Alberto Palmero Acebedo, Victor Rico Gavira

RESUMEN / ABSTRACT

La presente propuesta de proyecto de investigación persigue en primera instancia abordar una serie de aspectos básicos no resueltos relacionados con los mecanismos de la descarga barrera, las condiciones óptimas que deben cumplir los electrodos, la definición de un diseño óptimo de los mismos y el establecimiento de las mejores condiciones para la descarga.

En una segunda instancia y desde una perspectiva aplicada, se pretende la fabricación de dos tipos de reactores de descarga barrera mejorados para dos aplicaciones tecnológicas de gran impacto industrial. Primeramente para la funcionalización superficial de materiales avanzados persiguiendo, entre otros, el control lateral de la funcionalización según patrones litográficos. En segundo término, para el desarrollo de procesos de “plasma-catálisis” tendentes a aumentar la selectividad y disminuir el consumo energético de una serie de reacciones químicas de alto valor añadido e impacto industrial. Se prevé que, para ambos tipos de aplicaciones, los estudios básicos planteados permitan avanzar de manera clara en la optimización de los procesos finales con perspectivas de uso industrial.

This research project aims first the study of different unknown basic aspects of the construction of the dielectric barrier discharge, better design conditions for: barrier electrodes, the design of the metallic electrodes and dielectrics and to know the best working conditions (size and operation frequency) for the plasma. One goal is to control the lateral functionalization of advanced materials and other objective, is the discovering of new plasma catalysis processes that can increase selectivity and the reduction of energy consumption by plasma chemical reactions in controlled industrial processes of high added value and/or impact. It is expected for both applications, a clear advance in optimization of the industrial process.

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Síntesis y propiedades de nanopartículas luminiscentes para aplicaciones biomédicas Synthesis and properties of luminescent nanoparticles for biomedical applications

Código/Code: TAPOST-234
Periodo/Period: 01-10-2014 / 30-09-2016
Organismo Financiador/Financial source: Junta de Andalucía /Unión Europea
Importe total/Total amount: 155.008,57 €
Investigador responsable/Research head: Alberto Escudero Belmonte

RESUMEN / ABSTRACT

Las nanopartículas luminiscentes resultan de interés en Nanobiomedicina debido a sus diferentes aplicaciones, que incluyen sensores ópticos para la imagen de tejidos o estructuras intracelulares y para la detección y cuantificación de moléculas de interés biológico. En este proyecto se desarrollan nuevos métodos de síntesis de nanoestructuras uniformes de diferentes materiales inorgánicos (vanadatos, fluoruros, fosfatos y molibdatos dopados con cationes lantánidos) más económicos y respetuosos con el medio ambiente. También se evalúan las aplicaciones biomédicas de estos materiales, con especial atención al diseño de sensores y a su potencialidad como dispositivos para la detección e imagen de células cancerígenas. Este proyecto incluye la caracterización de los materiales obtenidos, la optimización de sus propiedades ópticas y magnéticas, el desarrollo de nuevos métodos de funcionalización y conjugación con moléculas de interés biológico, el análisis de la citotoxicidad de los materiales resultantes y el estudio de la interacción de las diferentes nanoestructuras funcionalizadas con células de distinta naturaleza, con especial atención al efecto de la morfología y composición química de las nanopartículas.

Luminescent nanoparticles are currently attracting wide research interest in Nanobiomedicine due to their applications, ranging from optical biolabels for imaging of tissues or intracellular structures to sensors to detect biological molecules, and as tracking devices. This project is focused on the design of new, cheaper, and environmentally friendly synthesis methods of uniform luminescent nanoparticles, such as rare earth doped fluorides, phosphates, molybdates, and vanadates. It also evaluates their biomedical applications, with especial attention to their sensing properties and their ability to detect tumour cells. This scientific work includes the characterization of the resulting materials, the optimization of their optical and magnetic properties, and the development of different functionalization processes. The final step of this research project deals with the study of the interaction of the functionalised nanoparticles with cells of different nature, and includes cytotoxicity studies, with special attention to the role played by the morphology and chemistry of the particles.

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Válvulas de espín orgánicas e híbridas orgánica-inorgánicas en nanofibras soportadas, producidas por técnicas avanzadas de deposición en vacío y asistidas por plasma Purely organic and hybrid organic-inorganic spin valves on supported nanowires produced by advanced vacuum and plasma-assisted deposition techniques

Periodo/Period:	01-10-2015 / 30-09-2017
Organismo Financiador/Financial source:	Junta de Andalucía /Unión Europea
Importe total/Total amount:	155.008,57 €
Investigador responsable/Research head:	Victor López Flores

RESUMEN / ABSTRACT

La transición a la electrónica orgánica requiere de nuevos elementos en la escala nanométrica compuestos por materiales orgánicos, lo que proporciona dispositivos flexibles, transparentes y baratos. Entre los dispositivos electrónicos, las válvulas de espín han destacado por su rápida transición desde la fase experimental a los productos de uso común, pero aún no se ha fabricado una válvula de espín orgánica que sea fiable. El objetivo científico de este proyecto es llenar ese vacío. Mediante el uso de métodos nanotecnológicos avanzados y escalables industrialmente, pretendemos producir una válvula de espín híbrida orgánica-inorgánica, y puramente orgánica en la forma de una nanofibra soportada de 200 nm de grosor y varias micras de longitud, con la multicapa de la válvula de espín depositada concéntricamente. Las técnicas de fabricación principales seran la deposición física en fase vapor de materiales orgánicos (O-PVD), la deposición química en fase vapor asistida por plasma (PE-CVD), y la deposición en vacío asistida por plasma remoto (RPAVD). Las medidas de magnetoresistencia se efectuarán por microscopía de fuerza atómica con sonda conductora (CP-AFM), que dará la medida definitiva de la calidad de la muestra producida.

El proyecto se desarrollará en el gripo Nanotecnología en Superficies (NanoOnSurf) del Instituto de Ciencia de Materiales de Sevilla, localizado en el centro de investigación multidisciplinar CicCartuja (Sevilla, España). Las técnicas de síntesis y cartacterización más avanzadas, desarrolladas dentro del grupo de investigación serán la clave para el éxito de este proyecto.

Este proyecto está directamente relacionado con el Programa de Trabajo Horizonte 2020, capítulo 5.i (2014-2015), acción ICT3 – 2014: “Tecnologías avanzadas en electrónica delgada, orgánica y de gran área”, y por tanto se espera un gran impacto del mismo en el futuro de la industria electrónica europea.

The transition to organic electronics requires new devices on the nanometer scale composed only by organic materials, providing small, flexible, transparent and cheap devices. Among electronic devices, the spin valves have stood out for their rapid transfer from the experimental phase to the general public products, but a reliable organic spin valve nanometric device is yet to be developed.

The scientific objective of this project is to fill that gap. By using advanced, industrially scalable nanotechnology methods, we intend to produce a hybrid organic-inorganic and a fully organic spin valve in the form of a supported nanowire of ~200 nm width and several microns length, with a concentric spin valve stack. Three main fabrication techniques will be used: organic Physical Vapor Deposition (O-PVD), plasma-enhanced Chemical Vapour Deposition (PE-CVD) and remote plasma assisted vacuum deposition (RPAVD). Magnetoresistance measurements will be performed on single nanowires by conducting-probe atomic force microscopy (CP-AFM), and will give the definite measurement of quality of the samples produced

This project will be developed within the Nanotechnology on Surfaces research group (NanoOnSurf), at the Institute of Materials Science of Seville (CSIC – University of Seville), located in the multidisciplinary CicCartuja research centre (Seville, Spain). State-of-the-art synthesis and characterisation techniques developed in the host research group will be the key for the success of this proposal.

This project is directly related with Horizon 2020 Work Programme 2014-2015, chapter 5.i, action ICT 3 – 2014: Advanced Thin, Organic and Large Area Electronics (TOLAE) technologies, and thus is expected to have a strong impact in the future European electronic industry.

Otros Proyectos / Other Projects

Red de Terapia Cellular (TERCEL)

Código/Code:	RD16/0011/0022
Periodo/Period:	01-01-2016 / 31-12-2020
Organismo Financiador/Financial source:	Instituto de Salud Carlos III
Importe total/Total amount:	182.286,50 €
Investigador responsable/Research head:	José Becerra Ratia (UMA)
Componentes/Research group:	Daniel Amat Trujillo (CIBER-BBN), Maria Aránzazu Díaz Cuenca, Leonor Santos Ruiz (CIBER-BBN)

Implantes personalizados de titanio poroso bioingenierizados para cirugía constructiva maxilofacial. Pruebas de concepto y ensayo preclinico

Código/Code:	BIO2015-66266-R
Periodo/Period:	01-01-2016 / 31-12-2018
Organismo Financiador/Financial source:	Ministerio Economia y Competitividad
Importe total/Total amount:	133.100 €
Investigador responsable/Research head:	José Becerra Ratia (UMA)
Componentes/Research group:	Maria Aránzazu Díaz Cuenca, Leonor Santos Ruiz (CIBER-BBN)

Desarrollo de nuevos pigmentos inorgánicos con aplicaciones cerámicas

Código/Code:	201460E005
Periodo/Period:	01-01-2014 / 31-12-2017
Organismo Financiador/Financial source:	CSIC (Intramural)
Importe total/Total amount:	72.693 €
Investigador responsable/Research head:	Manuel Ocaña Jurado

Nanopartículas basadas en matrices inorgánicas de tierras raras para aplicaciones biotecnolóticas

Código/Code:	201560E056
Periodo/Period:	01-06-2015 / 01-05-2019
Organismo Financiador/Financial source:	CSIC (Intramural)
Importe total/Total amount:	75.789 €
Investigador responsable/Research head:	Ana Isabel Becerro Nieto

CONVENIOS Y CONTRATOS / CONTRACTS AND AGREEMENTS

Promover el progreso científico en líneas de investigación de común interés y favorecer la transferencia de nuevo conocimiento a los sectores productivos

Periodo/Period:	02-08-2012 / 01-08-2016
Organismo Financiador/Financial source:	ABENGOA RESEARCH, S.L.
Importe total/Total amount:	290.400 €
Investigador responsable/Research head:	Agustín R. González-Elipe

SPIRE (FEDER INTERCONNECTA)

Periodo/Period:	24-11-2016 / 31-12-2018
Organismo Financiador/Financial source:	GHENOVA INGENIERIA S.L.U.
Importe total/Total amount:	302.500 €
Investigador responsable/Research head:	Hernán Míguez García
Componentes/Research group:	Gabriel S. Lozano Barbero, Mauricio Calvo Roggiani, Juan F. Galisteo López, Alberto Jiménez Solano

MULTILAYER MIRROR

Periodo/Period:	15-03-2015 / 15-02-2016
Organismo Financiador/Financial source:	ABENGOA SOLAR NEW TECHNOLOGIES, S.A.
Importe total/Total amount:	133.100€
Investigador responsable/Research head:	Hernán Míguez García
Componentes/Research group:	Mauricio Calvo Roggiani, Juan F. Galisteo López, Alberto Jiménez Solano, Miguel Anaya Martín

Caracterización de la respuesta químico-resistiva de composites plásticos conductores

Periodo/Period:	02-06-2015 / 01-12-2016
Organismo Financiador/Financial source:	ASOC. INDUSTRIA NAVARRA (AIN)
Importe total/Total amount:	18.150 €
Investigador responsable/Research head:	Agustín R. González-Elipe
Componentes/Research group:	Angel Barranco Quero, Alberto Palmero Acebedo, Ana Isabel Borrás Martos, Francisco Yubero Valencia, Juan Pedro Espinós Manzorro, Victor J. Rico Gavira, Jorge Gil Rostra

Monitorización en tiempo real de múltiples propiedades de fluidos mediante transductores fotónicos y electromagnéticos (REMO)

Periodo/Period:	01-09-2015 / 30-09-2018
Organismo Financiador/Financial source:	Universidad Carlos III, Indra Sistemas S.A., Universidad Politécnica de Madrid, Repsol S.A.
Importe total (ICMS)/Total amount (ICMS):	220.000 €

Investigador responsable (ICMS)/
Research head (ICMS): Agustín R. González-Elipe
Componentes/Research group: Francisco Yubero Valencia, Jorge Gil Rostra, Victor Rico Gavira, Juan Pedro Espinós Manzorro, Angel Barranco Quero, Ana Isabel Borrás Martos

Mechanically resistant superhydrophobic micro/nanostructures of stainless steel

Periodo/Period: 01-10-2015 / 30-10-2016
Organismo Financiador/Financial source: Proyecto I+D con BSH
Investigador responsable / Research head: Agustín R. González-Elipe
Componentes/Research group: Victor Rico Gavira, Francisco Yubero Valencia

PATENTES / PATENTS

Sensor, dispositivo y procedimiento para la determinación de la concentración de solutos en disoluciones

Inventores: Manuel Oliva Ramírez, Francisco Yubero Valencia, Agustín R. González-Elipe, Angel Barranco Quero
Tipo de Patente: Nacional
Número de Solicitud: PCT/ES16/070764
Fecha Solicitud: 27 de octubre de 2016
Entidad Titular: Universidad Politécnica de Madrid, Universidad de Zaragoza. Consejo Superior de Investigaciones Científicas

Biocompatible implants made of nanostructured titanium with antibacterial properties

Inventores: Rafael Alvarez Molina, José Miguel García Martín, Alberto Palmero Acebedo
Tipo de Patente: Nacional
Número de Solicitud: 15/306751
Fecha Solicitud: 25 octubre 2016
Entidad/es Titular/es: Universidad Complutense de Madrid. Consejo Superior de Investigaciones Científicas

Method for producing functionalised nanofibrous biopolymer matrices using nanostructured collagen

Inventores: Sara Borrego González, María Aránzazu Díaz Cuenca
Tipo de Patente: Nacional
Número de Solicitud: 201630913
Fecha Solicitud: 5 julio 2016
Entidad/es Titular/es: Consejo Superior de Investigaciones Científicas

Nanostructured perovskite

Inventores: Miguel Anaya Martín, Juan Francisco Galisteo López, Hernán Ruy Míguez García, Mauricio Calvo Roggiani
Tipo de Patente: Nacional
País de inscripción: España
Número de Solicitud: PCT/ES/16/061939
Fecha Solicitud: 26 mayo 2016
Entidad/es Titular/es: Consejo Superior de Investigaciones Científicas

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

Perspectives on oblique angle deposition of thin films: From fundamentals to devices

Barranco, A; Borras, A; Gonzalez-Elipe, AR; Palmero, A
Progress in Materials Science, **78** (2016) 59-153
Marzo, 2016 | **DOI:** 10.1016/j.pmatsci.2015.06.003

The oblique angle configuration has emerged as an invaluable tool for the deposition of nanostructured thin films. This review develops an up to date description of its principles, including the atomistic mechanisms governing film growth and nanostructuration possibilities, as well as a comprehensive description of the applications benefiting from its incorporation in actual devices. In contrast with other reviews on the subject, the electron beam assisted evaporation technique is analyzed along with other methods operating at oblique angles, including, among others, magnetron sputtering and pulsed laser or ion beam-assisted deposition techniques. To account for the existing differences between deposition in vacuum or in the presence of a plasma, mechanistic simulations are critically revised, discussing well-established paradigms such as the tangent or cosine rules, and proposing new models that explain the growth of tilted porous nanostructures. In the second part, we present an extensive description of applications wherein oblique-angle-deposited thin films are of relevance. From there, we proceed by considering the requirements of a large number of functional devices in which these films are currently being utilized (e.g., solar cells, Li batteries, electrochromic glasses, biomaterials, sensors, etc.), and subsequently describe how and why these nanostructured materials meet with these needs.

Maximized performance of dye solar cells on plastic: a combined theoretical and experimental optimization approach

Li, Yuelong; Carretero-Palacios, Sol; Yoo, Kicheon; Kim, Jong Hak; Jimenez-Solano, Alberto; Lee, Chul-Ho; Miguez, Hernan; Ko, Min Jae
Energy & Environmental Science, **9** (2016) 2061-2071
Febrero, 2016 | **DOI:** 10.1039/C6EE00424E

We demonstrate that a combined optimization approach based on the sequential alternation of theoretical analysis and experimental realization gives rise to plastic supported dye solar cells for which both light harvesting efficiency and electron collection are maximized. Rationalized configurations with optimized light trapping and charge extraction are realized to achieve photoanodes on plastic prepared at low temperature, showing a power conversion efficiency of 8.55% and a short circuit photocurrent of 16.11 mA cm−2, unprecedented for plastic based dye solar cell devices. Furthermore, the corresponding fully flexible designs present stable mechanical properties after several bending cycles, displaying 7.79% power conversion efficiency, an average broadband internal quantum efficiency above 90%, and a short circuit photocurrent of 15.94 mA cm−2, which is the largest reported value for bendable cells of this sort to date.

Unbroken Perovskite: Interplay of Morphology, Electro-optical Properties, and Ionic Movement

Correa-Baena, JP; Anaya, M; Lozano, G; Tress, W; Domanski, K; Saliba, M; Matsui, T; Jacobsson, TJ; Calvo, ME; Abate, A; Gratzel, M; Miguez, H; Hagfeldt, A
Advanced Materials, **28** (2016) 5031-5037
Julio, 2016 | **DOI:** 10.1002/adma.201600624

Hybrid organic-inorganic perovskite materials have risen up as leading components for light-harvesting applications. However, to date many questions are still open concerning the operation of perovskite solar cells (PSCs). A systematic analysis of the interplay among structural features, optoelectronic performance, and ionic movement behavior for FA(0.83)MA(0.17)Pb(I0.83Br0.17)(3) PSCs is presented, which yield high power conversion efficiencies up to 20.8%.

Metallic nanostructures for efficient LED lighting

Lozano, G; Rodriguez, SRK; Verschuuren, MA; Rivas, JG
Light: Science and Applications, **5** (2016) e16080
Junio, 2016 | **DOI:** 10.1038/lsa.2016.80

Light-emitting diodes (LEDs) are driving a shift toward energy-efficient illumination. Nonetheless, modifying the emission intensities, colors and directionalities of LEDs in specific ways remains a challenge often tackled by incorporating secondary optical components. Metallic nanostructures supporting plasmonic resonances are an interesting alternative to this approach due to their strong light-matter interaction, which facilitates control over light emission without requiring external secondary optical components. This review discusses new methods that enhance the efficiencies of LEDs using nanostructured metals. This is an emerging field that incorporates physics, materials science, device technology and industry. First, we provide a general overview of state-of-the-art LED lighting, discussing the main characteristics required of both quantum wells and color converters to efficiently generate white light. Then, we discuss the main challenges in this field as well as the potential of metallic nanostructures to circumvent them. We review several of the most relevant demonstrations of LEDs in combination with metallic nanostructures, which have resulted in light-emitting devices with improved performance. We also highlight a few recent studies in applied plasmonics that, although exploratory and eminently fundamental, may lead to new solutions in illumination.

Optofluidic Modulation of Self-Associated Nanostructural Units Forming Planar Bragg Microcavities

Oliva-Ramirez, M; Barranco, A; Loffler, M; Yubero, F; Gonzalez-Elipe, AR
ACS Nano, **10** (2016) 1256-1264

Enero, 2016 | **DOI:** 10.1021/acsnano.5b06625

Bragg microcavities (BMs) formed by the successive stacking of nanocolumnar porous SiO2 and TiO2 layers with slanted, zigzag, chiral, and vertical configurations are prepared by physical vapor deposition at oblique angles while azimuthally varying the substrate orientation during the multilayer growth. The slanted and zigzag BMs act as wavelength-selective optical retarders when they are illuminated with linearly polarized light, while no polarization dependence is observed for the chiral and vertical cavities. This distinct optical behavior is attributed to a self-nanostructuration mechanism involving a fence-bundling association of nanocolumns as observed by focused ion beam scanning electron microscopy in the slanted and zigzag microcavities. The outstanding retarder response of the optically active BMs can be effectively modulated by dynamic infiltration of nano- and mesopores with liquids of different refraction indices acting as a switch of the polarization behavior. The unprecedented polarization and tunable optofluidic properties of these nanostructured photonic systems have been successfully simulated with a simple model that assumes a certain birefringence for the individual stacked layers and accounts for the light interference phenomena developed in the BMs. The possibilities of this type of self-arranged nanostructured and optically active BMs for liquid sensing and monitoring applications are discussed.

High-Throughput Fabrication of Resonant Metamaterials with Ultrasmall Coaxial Apertures via Atomic Layer Lithography

Yoo, D; Nguyen, NC; Martin-Moreno, L; Mohr, DA; Carretero-Palacios, S; Shaver, J; Peraire, J; Ebbesen, TW; Oh, SH
Nano Letters, **16** (2016) 2040-2046
Marzo, 2016 | **DOI:** 10.1021/acs.nanolett.6b00024

We combine atomic layer lithography and glancing angle ion polishing to create wafer-scale metamaterials composed of dense arrays of ultrasmall coaxial nanocavities in gold films. This new fabrication scheme makes it possible to shrink the diameter and increase the packing density of 2 nm-gap coaxial resonators, an extreme subwavelength structure first manufactured via atomic layer lithography, both by a factor of 100 with respect to previous studies. We demonstrate that the nonpropagating zeroth-order Fabry-Perot mode, which possesses slow light-like properties at the cutoff resonance, traps infrared light inside 2 nm gaps (gap volume similar to lambda(3)/10(6)). Notably, the annular gaps cover only 3% or less of the metal surface, while open-area normalized transmission is as high as 1700% at the epsilon-near-zero (ENZ) condition. The resulting energy accumulation alongside extraordinary optical transmission can benefit applications in nonlinear optics, optical trapping, and surface-enhanced spectroscopies. Furthermore, because the resonance wavelength is independent of the cavity length and dramatically red shifts as the gap size is reduced, large-area arrays can be constructed with lambda(resonance) >> period, making this fabrication method ideal for manufacturing resonant metamaterials.

Electrocatalytic System for the Simultaneous Hydrogen Production and Storage from Methanol

Gonzalez-Cobos, J; Rico, VJ; Gonzalez-Elipe, AR; Valverde, JL; de Lucas-Consuegra, A
ACS Catalysis, **6** (2016) 1942-1951
Marzo, 2016 | **DOI:** 10.1021/acscatal.5b02844

This paper reports a groundbreaking approach for simultaneous hydrogen production and storage that entails catalysis, electrochemistry, surface science, and materials synthesis. A novel electrocatalytic system is developed based on nickel nanocolumnar films of controlled microstructure prepared on K-βAl2O3 solid electrolyte supports by oblique angle physical vapor deposition. The outstanding characteristics of this system are a hydrogen storage capacity of up to 19 g of H2 (100 g of Ni)−1, which is unparalleled in the literature and the possibility of controlling its release electrochemically, under fixed mild conditions (280 °C and normal pressure). H2 is produced in situ by methanol steam re-forming on the Ni catalyst, and it spills over onto graphene oxide aggregates formed during the catalytic process, as confirmed by SEM, FTIR, and Raman spectroscopy. The proposed storage mechanism considers a synergetic contribution of both Ni and graphene oxide, promoted by K+ ions, in enhancing the hydrogen storage capacity of the system.

Stabilization of catalyst particles against sintering on oxide supports with high oxygen ion lability exemplified by Ir-catalyzed decomposition of N_2O

Yentekakis, IV; Goula, G; Panagiotopoulou, P; Kampouri, S; Taylor, MJ; Kyriakou, G; Lambert, RM
Applied Catalysis B-Environmental, **192** (2016) 357-364
Septiembre, 2016 | **DOI:** 10.1016/j.apcatb.2016.04.011

Iridium nanoparticles deposited on a variety of surfaces exhibited thermal sintering characteristics that were very strongly correlated with the lability of lattice oxygen in the supporting oxide materials. Specifically, the higher the lability of oxygen ions in the support, the greater the resistance of the nanoparticles to sintering in an oxidative environment. Thus with gamma-Al_2O_3 as the support, rapid and extensive sintering occurred. In striking contrast, when supported on gadolinia-ceria and alumina-ceria-zirconia composite, the Ir nanoparticles underwent negligible sintering. In keeping with this trend, the behavior found with yttria-stabilized zirconia was an intermediate between the two extremes. This resistance, or lack of resistance, to sintering is considered in terms of oxygen spillover from support to nanoparticles and discussed with respect to the alternative mechanisms of Ostwald ripening versus nanoparticle diffusion. Activity towards the decomposition of N_2O, a reaction that displays pronounced sensitivity to catalyst particle size (large particles more active than small particles), was used to confirm that catalytic behavior was consistent with the independently measured sintering characteristics. It was found that the nanoparticle active phase was Ir oxide, which is metallic, possibly present as a capping layer. Moreover, observed turnover frequencies indicated that catalyst-support interactions were important in the cases of the sinter-resistant systems, an effect that may itself be linked to the phenomena that gave rise to materials with a strong resistance to nanoparticle sintering.

Three-Dimensional Optical Tomography and Correlated Elemental Analysis of Hybrid Perovskite Microstructures: An Insight into Defect-Related Lattice Distortion and Photoinduced Ion Migration

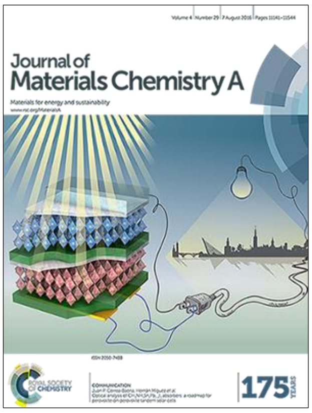
Galisteo-Lopez, JF; Li, YL; Miguez, H
Journal of Physical Chemistry Letters, **7** (2016) 5227-5234
Diciembre, 2016 | **DOI:** 10.1021/acsjpclett.6b02456

Organic lead halide perovskites have recently been proposed for applications in light-emitting devices of different sorts. More specifically, regular crystalline microstructures constitute an efficient light source and fulfill the geometrical requirements to act as resonators, giving rise to waveguiding and optical amplification. Herein we show three-dimensional laser scanning confocal tomography studies of different types of methylammonium lead bromide microstructures which have allowed us to dissect their photoemission properties with a precision of 0.036 mu m(3). This analysis shows that their spectral emission presents strong spatial variations which can be attributed to defect-related lattice distortions. It is also largely enhanced under light exposure, which triggers the migration of halide ions away from illuminated regions, eventually leading to a strongly anisotropic degradation. Our work points to the need for performing an optical quality test of individual crystallites prior to their use in optoelectronics devices and provides a means to do so.

Efficient bifacial dye-sensitized solar cells through disorder by design

Miranda-Munoz, JM; Carretero-Palacios, S; Jimenez-Solano, A; Li, YL; Lozano, G; Miguez, H
Journal of Materials Chemistry A, **4** (2016) 1953-1961
Febrero, 2016 | **DOI:** 10.1039/C5TA10091G

Herein we realize an optical design that optimizes the performance of bifacial solar cells without modifying any of the usually employed components. In order to do so, dielectric scatterers of controlled size and shape have been successfully integrated in the working electrodes of dye-sensitized solar cells (DSSCs), resulting in bifacial devices of outstanding performance. Power conversion efficiencies (PCEs) as high as 6.7% and 5.4% have been attained under front and rear illumination, respectively, which represent a 25% and a 33% PCE enhancement with respect to an 8 µm-thick standard solar cell electrode using platinum as the catalytic material. The remarkable bifacial character of our approach is demonstrated by the high rear/front efficiency ratio attained, around 80%, which is among the largest reported for this sort of device. The proposed optimized design is based on a Monte Carlo approach in which the multiple scattering of light within the cell is fully accounted for. We identified that the spherical shape of the scatterers is the key parameter controlling the angular distribution of the scattering, the most efficient devices being those in which the inclusions provide a narrow forward-oriented angular distribution of the scattered light.



Optical analysis of CH3NH3SnxPb1−xI3 absorbers: a roadmap for perovskite-on-perovskite tandem solar cells

Anaya, M.; Correa-Baena, J.P.; Lozano, G.; Saliba, M.; Anguita, P.; Roose, B.; Abate, A.; Steiner, U.; Gratzel, M.; Calvo, M.E.; Hagfeldt, A.; Míguez, H.
Journal lf Materials Chemistry A, **4** (2016) 11214-11221
Agosto, 2016 | **DOI:** 10.1039/C6TA04840D

Organic–inorganic perovskite structures in which lead is substituted by tin are exceptional candidates for broadband light absorption. Herein we present a thorough analysis of the optical properties of CH3NH3SnxPb1−xI3 films, providing the field with definitive insights about the possibilities of these materials for perovskite solar cells of superior efficiency. We report a user's guide based on the first set of optical constants obtained for a series of tin/lead perovskite films, which was only possible to measure due to the preparation of optical quality thin layers. According to the Shockley–Queisser theory, CH3NH3SnxPb1−xI3 compounds promise a substantial enhancement of both short circuit photocurrent and power conversion efficiency in single junction solar cells. Moreover, we propose a novel tandem architecture design in which both top and bottom cells are made of perovskite absorbers. Our calculations indicate that such perovskite-on-perovskite tandem devices could reach efficiencies over 35%. Our analysis serves to establish the first roadmap for this type of cells based on actual optical characterization data. We foresee that this study will encourage the research on novel near-infrared perovskite materials for photovoltaic applications, which may have implications in the rapidly emerging field of tandem devices.

Light management: porous 1-dimensional nanocolumnar structures as effective photonic crystals for perovskite solar cells

Ramos, FJ; Oliva-Ramirez, M; Nazeeruddin, MK; Graetzel, M; Gonzalez-Elipe, AR; Ahmad, S
Journal of Materials Chemistry A, **4** (2016) 4962-4970
Abril, 2016 | **DOI:** 10.1039/c5ta08743k

Hybrid organic-inorganic perovskite solar cells are a topic of increasing interest, as in a short time span they are able to lead in the third generation photovoltaics. Organohalide perovskites possess exceptional optoelectronic and physical properties, thus making their implementation possible in many diverse configurations of photovoltaic devices. In this work, we present three different configurations of porous 1-dimensional photonic crystals (1-DPCs) based on alternated nanocolumnar layers of oxides with different refractive indices (n) that were deposited by Physical Vapor Deposition at Oblique Angle Deposition (PVD-OAD). They are then implemented as the photoanode in CH3NH3PbI3 solar cells to improve the management of light into the device. These configurations improved the performance of the photovoltaic system by designing a light interference structure capable of enhancing the absorption capability of the perovskite. A device fabricated using these photonic crystal structures presented an efficiency >12% in contrast with only 10.22% for a reference device based on non-photonic crystal TiO2 layers deposited under analogous conditions.

Optical sensing by integration of analyte-sensitive fluorophore to particles

Carrillo-Carrion, C; Escudero, A; Parak, WJ
TrAC Trends in Analytical Chemistry, **84** (2016) 84-85
Noviembre, 2016 | **DOI:** 10.1016/j.trac.2016.05.001

Analyte-sensitive fluorophores are a common tool in analytical chemistry. In case they are conjugated to the surface of colloidal nanoparticles new or improved applications are possible. An overview of the potential of such fluorophore-particle conjugates is given by means of several examples. First, using pH-sensitive fluorophores attached to particles are a helpful tool for investigating particle uptake by cells, as they can indicate whether particles are in the neutral slightly alkaline extracellular medium, or in acidic intracellular vesicles after endocytosis. Second, relating to lifetime-based methodologies, the fluorescence resonance energy transfer between fluorophores attached to quantum dots leads to longer lifetimes, improving their performance and expanding the possibilities of methods, such as lifetime imaging for in vivo applications. It also can be exploited for multiplexing approaches, in which the effective lifetime of the fluorophores can be tuned, allowing thus for the detection of several analytes based on temporal discrimination. Attention is focused to these three areas of application, because they are among the most reported in recent literature, and therefore of particular interest.

Laser Treatment of Nanoparticulated Metal Thin Films for Ceramic Tile Decoration

Rico, VJ; Lahoz, R; Rey-Garcia, F; Yubero, F; Espinos, JP; de la Fuente, GF; Gonzalez-Elipe, AR
Applied Materials & Interfaces, **8** (2016) 24880-24886
Septiembre, 2016 | **DOI:** 10.1021/acsami.6b07469

This paper presents a new method for the fabrication of metal-like decorative layers on glazed ceramic tiles. It consists of the laser treatment of Cu thin films prepared by electron-beam evaporation at glancing angles. A thin film of discontinuous Cu nanoparticles was electron-beam-evaporated in an oblique angle configuration onto ceramic tiles and an ample palette of colors obtained by laser treatment both in air and in vacuum. Scanning electron microscopy along with UV–vis–near-IR spectroscopy and time-of-flight secondary ion mass spectrometry analysis were used to characterize the differently colored layers. On the basis of these analyses, color development has been accounted for by a simple model considering surface melting phenomena and different microstructural and chemical transformations of the outmost surface layers of the samples.

Growth Assisted by Glancing Angle Deposition: A New Technique to Fabricate Highly Porous Anisotropic Thin Films

Sanchez-Valencia, JR; Longtin, R; Rossell, MD; Groning, P
ACS Applied Materials & Interfaces, **8** (2016) 8686-8693
Abril, 2016 | **DOI:** 10.1021/acsami.6b00232

We report a new methodology based on glancing angle deposition (GLAD) of an organic molecule in combination with perpendicular growth of a second inorganic material. The resulting thin films retain a very well-defined tilted columnar microstructure characteristic of GLAD with the inorganic material embedded inside the columns. We refer to this new methodology as growth assisted by glancing angle deposition or GAGLAD, since the material of interest (here, the inorganic) grows in the form of tilted columns, though it is deposited under a nonglancing configuration. As a “proof of concept”, we have used silver and zinc oxide as the perpendicularly deposited material since they usually form ill-defined columnar microstructures at room temperature by GLAD. By means of our GAGLAD methodology, the typical tilted columnar microstructure can be developed for materials that otherwise do not form ordered structures under conventional GLAD. This simple methodology broadens significantly the range of materials where control of the microstructure can be achieved by tuning the geometrical deposition parameters. The two examples presented here, Ag/Alq3 and ZnO/Alq3, have been deposited by physical vapor deposition (PVD) and plasma enhanced chemical vapor deposition (PECVD), respectively: two different vacuum techniques that illustrate the generality of the proposed technique. The two type of hybrid samples present very interesting properties that demonstrate the potentiality of GAGLAD. On one hand, the Ag/Alq3 samples present highly optical anisotropic properties when they are analyzed with linearly polarized light. To our knowledge, these Ag/Alq3 samples present the highest angular selectivity reported in the visible range. On the other hand, ZnO/Alq3 samples are used to develop highly porous ZnO thin films by using Alq3 as sacrificial material. In this way, antireflective ZnO samples with very low refractive index and extinction coefficient have been obtained.

Synthesis and functionalization of monodisperse near-ultraviolet and visible excitable multifunctional Eu^{3+}, Bi^{3+}: $REVO_4$ nanophosphors for bioimaging and biosensing applications

Escudero, A.; Carrillo-Carrion, C.; Zyuzin, Mikhail V.; Ashraf, S.; Hartmann, R.; Nunez, N.O.; Ocaña, M.; Parak, W.J.

Nanoscale, ***8*** (2016) 12221-12236

Junio, 2016 | **DOI:** 10.1039/c6nr03369e

Near-ultraviolet and visible excitable Eu-and Bi-doped NPs based on rare earth vanadates ($REVO_4$, RE = Y, Gd) have been synthesized by a facile route from appropriate RE precursors, europium and bismuth nitrate, and sodium orthovanadate, by homogeneous precipitation in an ethylene glycol/water mixture at 120 degrees C. The NPs can be functionalized either by a one-pot synthesis with polyacrylic acid (PAA) or by a Layer-by-Layer approach with poly(allylamine hydrochloride) (PAH) and PAA. In the first case, the particle size can also be tuned by adjusting the amount of PAA. The Eu-Bi-doped $REVO_4$ based nanophosphors show the typical red luminescence of Eu(III), which can be excited through an energy transfer process from the vanadate anions, resulting in a much higher luminescence intensity in comparison to the direct excitation of the europium cations. The incorporation of Bi into the $REVO_4$ structure shifts the original absorption band of the vanadate anions towards longer wavelengths, giving rise to nanophosphors with an excitation maximum at 342 nm, which can also be excited in the visible range. The suitability of such nanophosphors for bioimaging and biosensing applications, as well as their colloidal stability in different buffer media of biological interest, their cytotoxicity, their degradability at low pH, and their uptake by HeLa cells have been evaluated. Their suitability for bioimaging and biosensing applications is also demonstrated.

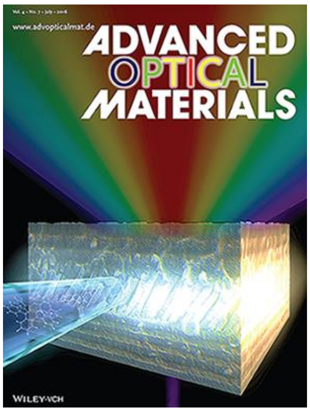
Integration of Photonic Crystals into Flexible Dye Solar Cells: A Route toward Bendable and Adaptable Optoelectronic Devices Displaying Structural Color and Enhanced Efficiency

Li, YL; Calvo, ME; Miguez, H

Advanced Optical Materials, ***4*** (2016) 464-471

Marzo, 2016 | **DOI:** 10.1002/adom.201500547

Herein is presented what is believed to be the first example of integration of photonic structures in a flexible optoelectronic device. The resulting devices may be designed to display any color in the visible range and, simultaneously, present enhanced power conversion efficiency as a consequence of the increased light harvesting caused by the colored back reflection. The achievement results from the incorporation of nanoparticle-based multilayers with photonic crystal properties that are modified to be compatible with the chemical and physical processing of flexible nanocrystalline titania electrodes of dye solar cells. The photovoltaic performance of these colored flexible cells remains unaltered after one hundred bending cycles, thus showing the high-mechanical stability of the ensemble. These devices reunite most characteristics required for building integration or for the construction of solar window panes, such as light weight, stability upon bending, adaptability, and color. This work may trigger promising applications of these highly adaptable and versatile photonic crystals in other flexible devices.



A Full Vacuum Approach for the Fabrication of Hybrid White-Light-Emitting Thin Films and Wide-Range In Situ Tunable Luminescent Microcavities

Y. Oulad-Zian, J.R. Sánchez-Valencia, M. Oliva, J. Parra-Barranco, M. Alcaire, F.J. Aparicio, A. Mora-Boza, J.P. Espinós, F. Yubero, A.R. González-Elipe, A. Barranco, A. Borras
Advanced Optical Materials, **4** (2016) 1134
Julio, 2016 | **DOI:** 10.1002/adom.201670041

A wide-range in situ tunable 1D Bragg microcavity including a hybrid layer as white light emitter defect is shown by J. R. Sanchez-Valencia, A. Borras, and co-workers on page 1124. White emission is obtained by Förster resonance energy transfer between blue (1,3,5-triphenyl-2-pyrazoline) and orange (rubrene) dyes homogeneously infiltrated within the host nanocolumnar SiO_2film, which is formed by glancing angle deposition. Sequential physical vapor deposition at low temperatures provides the organic dyes localization within the porous nanostructure of the defect layer.

Isotope labelling to study molecular fragmentation during the dielectric barrier discharge wet reforming of methane

Montoro-Damas, AM; Gomez-Ramirez, A; Gonzalez-Elipe, AR; Cotrino, J
Journal of Power Sources, **325** (2016) 501-505
Septiembre, 2016 | **DOI:** 10.1016/j.jpowsour.2016.06.028

Isotope labelling is used to study the wet plasma reforming of methane in a dielectric barrier discharge reactor using D_2O and CH_4 as reactants. Besides the formation of CO and hydrogen as main products, different partitions of H and D atoms are found in the hydrogen (i.e., Hz, HD, D-2), methane (i.e., CH_4, CH_3D and CH_2D_2) and water (D_2O, DHO) molecules detected by mass spectrometry as outlet gases of the plasma process. The effect of operating parameters such as applied current, residence time and the addition of oxygen to the reaction mixture is correlated with the H/D distribution in these molecules, the overall reaction yield and the energetic efficiency of the process. The results prove the plasma formation of intermediate excited species that rendering water and methane instead of CO and hydrogen greatly contribute to decrease the overall energy efficiency of the reforming process.

Synthesis, characterization and performance of robust poison resistant ultrathin film yttria stabilized zirconia nickel anodes for application in solid electrolyte fuel cells

Garcia-Garcia, FJ; Yubero, F; Espinos, JP; Gonzalez-Elipe, AR; Lambert, RM
Journal of Power Sources, **324** (2016) 679-686
Julio, 2016 | **DOI:** 10.1016/j.jpowsour.2016.05.124

We report on the synthesis of undoped ~5 µm YSZ-Ni porous thin films prepared by reactive pulsed DC magnetron sputtering at an oblique angle of incidence. Pre-calcination of the amorphous unmodified precursor layers followed by reduction produces a film consisting of uniformly distributed tilted columnar aggregates having extensive three-phase boundaries and favorable gas diffusion characteristics. Similarly prepared films doped with 1.2 at.% Au are also porous and contain highly dispersed gold present as Ni-Au alloy particles whose surfaces are strongly enriched with Au. With hydrogen as fuel, the performance of the undoped thin film anodes is comparable to that of 10–20 times thicker typical commercial anodes. With a 1:1 steam/carbon feed, the un-doped anode cell current rapidly falls to zero after 60 h. In striking contrast, the initial performance of the Au-doped anode is much higher and remains unaffected after 170 h. Under deliberately harsh conditions the performance of the Au-doped anodes decreases progressively, almost certainly due to carbon deposition. Even so, the cell maintains some activity after 3 days operation in dramatic contrast with the un-doped anode, which stops working after only three hours of use. The implications and possible practical application of these findings are discussed.

A panchromatic modification of the light absorption spectra of metal-organic frameworks

Otal, E. H.; Kim, M. L.; Calvo, M. E.; Karvonen, L.; Fabregas, I. O.; Sierra, C. A.; Hinestroza, J. P.
Chemical Communications, **52** (2016) 6665-6668
Mayo, 2016 | **DOI:** 10.1039/c6cc02319c

The optical absorption of UiO-66–NH2 MOF was red-shifted using a diazo-coupling reaction. The modifications performed with naphthols and aniline yielded reddish samples, and the modifications with diphenylaniline yielded dark violet ones. The photocatalytic activity of these modified MOFs was assessed for methylene blue degradation, showing a good performance relative to traditional TiO2. The degradation performance was found to correlate with the red shift of the absorption edge. These findings suggest potential applications of these materials in photocatalysis and in dye sensitized solar cells.

Effect of TiO_2-Pd and TiO_2-Ag on the photocatalytic oxidation of diclofenac, isoproturon and phenol

Espino-Estevez, MR; Fernandez-Rodriguez, C; Gonzalez-Diaz, OM; Arana, J; Espinos, JP; Ortega-Mendez, JA; Dona-Rodriguez, JM
Chemical Engineering Journal, **298** (2016) 82-95
Agosto, 2016 | **DOI:** 10.1016/j.cej.2016.04.016

The effects of silver and palladium metals on the photocatalytic degradation of diclofenac sodium salt (DCF), isoproturon (IP) and phenol (PHL) in water over lab-made TiO2 synthesized following a sol-gel method were investigated. Silver and palladium catalysts were prepared by photodeposition at 1 wt.% of loading metal. The resulting materials were characterized through BET, XRD, TEM, SEM, XPS and DRS-UV-Vis. The photodeposition test conditions of both metals determined their final oxidation state, with reduced particles of palladium and silver as well as silver oxides found on the catalysts. The results showed that the type of metal had different effects on the photodegradation mechanism depending on the nature of the pollutants. Accordingly, the highest degradation rate for IP and DCF was obtained when using the catalyst photodeposited with palladium and for PHL the catalyst photodeposited with silver. The photodegradation intermediates of PHL, DCF and IP were also identified.

Nickel-copper bilayer nanoporous electrode prepared by physical vapor deposition at oblique angles for the non-enzymatic determination of glucose

Salazar, P; Rico, V; Gonzalez-Elipe, AR

Sensors and Actuators B: Chemical, **226** (2016) 436-443
Abril, 2016 | **DOI:** 10.1016/j.snb.2015.12.003

This work presents a novel bilayer Ni/Cu porous nanostructured film electrode prepared by physical vapor deposition (PVD) in an oblique angle configuration. Scanning electron microscopy (SEM) data revealed that the film, with an approximate thickness of 200 nm, is formed by tilted nanocolumns of around 50 nm of diameter and an inclination of 30° with respect to the surface normal. X ray photoelectron spectroscopy (XPS) data confirmed a bilayer configuration with Cu and Ni located at the top and bottom parts of the film, respectively. A porosity of ca. 45–35% as determined by Rutherford back scattering (RBS) offered a large exposed area and excellent diffusion properties that, combined with a very good catalytic activity, rendered these films excellent electrodes for the quantitative determination of glucose. Under optimized working conditions of detection these electrodes presented a high sensitivity of 2.53 A M−1 cm−2 (R2: 0.999), a limit of detection of 0.23 μM and a time response of ca. 2 s. The sensors did not show any loss of response during a period of 4 months. The selectivity of the sensor was checked against various interferences, including physiological compounds, different sugars and ethanol, in all cases with excellent results. The feasibility of using of this sensor for practical applications was confirmed by successfully determining the glucose content in different commercial beverages.

Dye-based photonic sensing systems

Aparicio, FJ; Alcaire, M; Gonzalez-Elipe, AR; Barranco, A; Holgado, M; Casquel, R; Sanza, FJ; Griol, A; Bernier, D; Dortu, F; Caceres, S; Antelius, M; Lapisa, M; Sohlstrom, H; Niklaus, F
Sensors and Actuators B: Chemical, **228** (2016) 649-657
Junio, 2016 | **DOI:** 10.1016/j.snb.2016.01.092

We report on dye-based photonic sensing systems which are fabricated and packaged at wafer scale. For the first time luminescent organic nanocomposite thin-films deposited by plasma technology are integrated in photonic sensing systems as active sensing elements. The realized dye-based photonic sensors include an environmental NO2 sensor and a sunlight ultraviolet light (UV) A+B sensor. The luminescent signal from the nanocomposite thin-films responds to changes in the environment and is selectively filtered by a photonic structure consisting of a Fabry-Perot cavity. The sensors are fabricated and packaged at wafer-scale, which makes the technology viable for volume manufacturing. Prototype photonic sensor systems have been tested in real-world scenarios.

Glutamate microbiosensors based on Prussian Blue modified carbon fiber electrodes for neuroscience applications: In-vitro characterization

Salazar, P; Martin, M; O'Neill, RD; Gonzalez-Mora, JL
Sensors and Actuators B: Chemical, **235** (2016) 117-125
Noviembre, 2016 | **DOI:** 10.1016/j.snb.2016.05.057

Herein we report a Prussian Blue modified carbon fiber electrode (CFE/PB) to be used in microbiosensors for glutamate monitoring in physiological applications as an alternative to the classical Pt and Pt-Ir transducers. Their low dimensions (~250 μm CFE length and ~10 μm diameter) are advantageous for measuring in living tissues. In addition, PB-modified microelectrodes allow the detection of enzyme-generated hydrogen peroxide at a low applied potential (~0.0 V against SCE), contrasting the high potential used in many previous designs (~0.7 V), decreasing the endogenous interference contributions. Moreover, the electrosynthesized polymer, poly-o-phenylenediamine (PoPD), was used to improve biosensor stability and selectivity. CFE/PB was conveniently characterized using impedance, Raman and XPS spectroscopies. Optimization of the fabrication procedure and analytical conditions is described, including activation of CFE/PB, enzyme enrichment, cross-linking, stabilization and anti-interference. A range of analytical parameters were also characterized such as sensitivity, limit of detection, linear range, and enzymatic loading. Finally, an optimized biosensor displaying a linear sensitivity of 135 ± 2 nA μM−1 cm−2 (n = 3), LOD of <2 μM, linear range up to 150 μM and effectively free of interference, is proposed as a suitable candidate for in-vivo glutamate monitoring in the central nervous system.

Reduced graphene oxide-carboxymethylcellulose layered with platinum nanoparticles/PAMAM dendrimer/magnetic nanoparticles hybrids. Application to the preparation of enzyme electrochemical biosensors

Borisova, B; Sanchez, A; Jimenez-Falcao, S; Martin, M; Salazar, P; Parrado, C; Pingarron, JM; Villalonga, R

Sensors and Actuators B: Chemical, **232** (2016) 84-90
Septiembre, 2016 | **DOI:** 10.1016/j.snb.2016.02.106

The assembly of a novel layer-by-layer biosensor architecture using hybrid nanomaterials is explored for the construction of an amperometric enzyme biosensors. The nanostructured sensing interface was prepared with poly(dopamine)-modified magnetic nanoparticles which were covalently coated with four-generation ethylenediamine core polyamidoamine G-4 dendrimers and further decorated with platinum nanoparticles. This nanohybrid was fully characterized and further layered on glassy carbon electrodes coated with a graphene oxide-carboxymethylcellulose hybrid nanomaterial through electrostatic interactions. The nanostructured surface was then employed as scaffold for the covalent immobilization of the enzyme xanthine oxidase through a glutaraldehyde-mediated cross-linking. The enzyme electrode allowed the amperometric detection of xanthine in the 50 nM-12 mu M range, with a high sensitivity of 140 mA/M cm(2) and low detection limit of 13 nM. The biosensor exhibited high reproducibility and repeatability, and was successfully tested for the quantification of xanthine in fish samples.

Solution processed high refractive index contrast distributed Bragg reflectors

Anaya, M; Rubino, A; Calvo, ME; Miguez, H

Journal of Physical Chemistry C, **4** (2016) 4532-4537
Mayo, 2016 | **DOI:** 10.1039/C6TC00663A

We have developed a method to alternate porous and dense dielectric films in order to build high refractive index contrast distributed Bragg reflectors (DBRs) capable of reflecting very efficiently in a targeted spectral range employing a small number of layers in the stack. Porous layers made of SiO_2 nanoparticles and compact sol–gel processed TiO_2 layers are sequentially deposited. The key to the preservation of porosity of every other layer during the deposition process is the use of a sacrificial layer of polystyrene that prevents the infiltration of the interstitial voids between nanoparticles with the homogeneous solution of TiO_2 precursors. Our approach allows preparing a series of DBRs operating along the whole visible spectral range.

Reflectance values as high as 90% are achieved from only seven layers. The particular distribution of porosity along one direction gives rise to an interesting interplay between the optical properties of the system and the vapor pressure in the surrounding atmosphere, which we foresee could be put into practice in gas sensing devices.

Transparent polycrystalline SrREGa3O7 melilite ceramics: potential phosphors for tuneable solid state lighting

Boyer, M; Carrion, AJF; Ory, S; Becerro, AI; Villette, S; Eliseeva, SV; Petoud, S; Aballea, P; Matzen, G; Allix, M

Journal of Materials Chemistry C, **15** (2016) 3238-3247

Abril, 2016 | **DOI:** 10.1039/C6TC00633G

Full and congruent crystallization from glass is applied to the SrREGa3O7 melilite family (RE = Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb and Y). This innovative process enables the synthesis of polycrystalline ceramics exhibiting high transparency both in the visible and near infrared regions, despite tetragonal crystal structures and micrometer scale grain sizes. Moreover, glass crystallization provides an original route to synthesize new crystalline phases which are not accessible via a classic solid state reaction, as demonstrated for SrYbGa3O7 and SrTmGa3O7. To illustrate the potential optical applications of such materials, SrGdGa3O7 transparent polycrystalline ceramics are doped with Dy3+ or Tb3+/Eu3+ in order to generate white light emission under UV excitation. It is foreseen that such transparent melilite ceramic phosphors, prepared via a cost-effective process, can be successfully used in solid state lighting devices of considerable technological interest.

Quantitative uptake of colloidal particles by cell cultures

Feliu, N; Huhn, J; Zyuzin, MV; Ashraf, S; Valdeperez, D; Masood, A; Said, AH; Escudero, A; Pelaz, B; Gonzalez, E; Duarte, MAC; Roy, S; Chakraborty, I; Lim, ML; Sjoqvist, S; Jungebluth, P; Parak, WJ

Science of the Total Environment, **568** (2016) 819-828

Octubre, 2016 | **DOI:** 10.1016/j.scitotenv.2016.05.213

The use of nanotechnologies involving nano-and microparticles has increased tremendously in the recent past. There are various beneficial characteristics that make particles attractive for a wide range of technologies. However, colloidal particles on the other hand can potentially be harmful for humans and environment. Today, complete understanding of the interaction of colloidal particles with biological systems still remains a challenge. Indeed, their uptake, effects, and final cell cycle including their life span fate and degradation in biological systems are not fully understood. This is mainly due to the complexity of multiple parameters which need to be taken in consideration to perform the nanosafety research. Therefore, we will provide an overview of the common denominators and ideas to achieve universal metrics to assess their safety. The review discusses aspects including how biological media could change the physicochemical properties of colloids, how colloids are endocytosed by cells, how to distinguish between internalized versus membrane-attached colloids, possible correlation of cellular uptake of colloids with their physicochemical properties, and how the colloidal stability of colloids may vary upon cell internalization. In conclusion three main statements are given. First, in typically exposure scenarios only part of the colloids associated with cells are internalized while a significant part remain outside cells attached to their membrane. For quantitative uptake studies false positive counts in the form of only adherent but not internalized colloids have to be avoided. pH sensitive fluorophores attached to the colloids, which can discriminate between acidic endosomal/lysosomal and neutral extracellular environment around colloids offer a possible solution. Second, the metrics selected for uptake studies is of utmost importance. Counting the internalized colloids by number or by volume may lead to significantly different results. Third, colloids may change their physicochemical properties along their life cycle, and appropriate characterization is required during the different stages.

Characterization and application of a new pH sensor based on magnetron sputtered porous WO3 thin films deposited at oblique angles

Salazar, P; Garcia-Garcia, FJ; Yubero, F; Gil-Rostra, J; Gonzalez-Elipe, AR
Electrochimica Acta, **193** (2016) 24-31
Marzo, 2016 | **DOI:** 10.1016/j.electacta.2016.02.040

In this communication we report about an outstanding solid-state pH sensor based on amorphous nanocolumnar porous thin film electrodes. Transparent WO3 thin films were deposited by reactive magnetron sputtering in an oblique angle configuration to enhance their porosity onto indium tin oxide (ITO) and screen printed electrodes (SPE). The potentiometric pH response of the nanoporous WO3-modified ITO electrode revealed a quasi-Nernstian behaviour, i.e. a linear working range from pH 1 to 12 with a slope of about -57.7 mV/pH. pH detection with this electrode was quite reproducible, displayed excellent anti-interference properties and a high stable response that remained unaltered over at least 3 months. Finally, a pH sensor was developed using nanoporous WO3-modified screen printed electrode (SPE) using a polypyrrole-modified Ag/AgCl electrode as internal reference electrode. This full solid state pH sensor presented a Nernstian behaviour with a slope of about -59 mV/pH and offered important analytical and operation advantages for decentralized pH measurements in different applications.

Non-enzymatic Glucose electrochemical sensor made of porous NiO thin films prepared by reactive magnetron sputtering at oblique angles

Garcia-Garcia, FJ; Salazar, P; Yubero, F; Gonzalez-Elipe, AR
Electrochimica Acta, **201** (2016) 38-44
Mayo, 2016 | **DOI:** 10.1016/j.electacta.2016.03.193

Porous nanostructured NiO thin films have been prepared in one step by magnetron sputtering in an oblique angle configuration (MS-OAD) and used as electrodes for the non-enzymatic detection of glucose. The films have been thoroughly characterized by different complementary techniques and their performance for the analysis of glucose in basic solutions determined by electrochemical methods. These electrodes presented four times higher sensitivity that equivalent compact thin films prepared by MS in a normal configuration and were superior in terms of sensitivity than majority of nickel based electrodes prepared by other methods. Finally, a high sensitivity towards detection of glucose in blood, insensitivity to common interferences, a long term stability and high reproducibility confirmed the good performance and reliability of these electrodes for practical analytical purposes.

Photophysical Analysis of the Formation of Organic–Inorganic Trihalide Perovskite Films: Identification and Characterization of Crystal Nucleation and Growth

Anaya, M; Galisteo-Lopez, JF; Calvo, ME; Lopez, C; Miguez, H
Journal of Physical Chemistry C, **120** (2016) 3071-3076
Febrero, 2016 | **DOI:** 10.1021/acs.jpcc.6b00398

In this work we demonstrate that the different processes occurring during hybrid organic–inorganic lead iodide perovskite film formation can be identified and analyzed by a combined in situ analysis of their photophysical and structural properties. Our observations indicate that this approach permits unambiguously identifying the crystal nucleation and growth regimes that lead to the final material having a cubic crystallographic phase, which stabilizes to the well-known tetragonal phase upon cooling to room temperature. Strong correlation between the dynamic and static photoemission results and the temperature-dependent X-ray diffraction data allows us to provide a description and to establish an approximate time scale for each one of the stages and their evolution. The combined characterization approach herein explored yields key information about the kinetics of the process, such as the link between the evolution of the defect density during film formation, revealed by a fluctuating photoluminescence quantum yield, and the gradual changes observed in the PbI2-related precursor structure.

Microstructural, spectroscopic, and antibacterial properties of silver-based hybrid nanostructures biosynthesized using extracts of coriander leaves and seeds

Luna, C; Barriga-Castro, ED; Gomez-Trevino, A; Nunez, NO; Mendoza-Resendez, R
International Journal of Nanomedicine, **11** (2016) 4787-4798
Septiembre, 2016 | **DOI:** 10.2147/IJN.S105166

Coriander leaves and seeds have been highly appreciated since ancient times, not only due to their pleasant flavors but also due to their inhibitory activity on food degradation and their beneficial properties for health, both ascribed to their strong antioxidant activity. Recently, it has been shown that coriander leaf extracts can mediate the synthesis of metallic nanoparticles through oxidation/reduction reactions. In the present study, extracts of coriander leaves and seeds have been used as reaction media for the wet chemical synthesis of ultrafine silver nanoparticles and nanoparticle clusters, with urchin-and tree-like shapes, coated by biomolecules (mainly, proteins and polyphenols). In this greener route of nanostructure preparation, the active biocompounds of coriander simultaneously play the roles of reducing and stabilizing agents. The morphological and microstructural studies of the resulting biosynthesized silver nanostructures revealed that the nanostructures prepared with a small concentration of the precursor Ag salt (AgNO3 =5 mM) exhibit an ultrafine size and a narrow size distribution, whereas particles synthesized with high concentrations of the precursor Ag salt (AgNO3 =0.5 M) are polydisperse and formation of supramolecular structures occurs. Fourier transform infrared and Raman spectroscopy studies indicated that the bioreduction of the Ag-ions takes place through their interactions with free amines, carboxylate ions, and hydroxyl groups. As a consequence of such interactions, residues of proteins and polyphenols cap the biosynthesized Ag nanoparticles providing them a hybrid core/shell structure. In addition, these biosynthesized Ag nanomaterials exhibited size-dependent plasmon extinction bands and enhanced bactericidal activities against both Gram-positive and Gram-negative bacteria, displaying minimal inhibitory Ag concentrations lower than typical values reported in the literature for Ag nanoparticles, probably due to the synergy of the bactericidal activities of the Ag nanoparticle cores and their capping ligands..

Gold-Based Nanomaterials for Applications in Nanomedicine

Ashraf, S; Pelaz, B; del Pino, P; Carril, M; Escudero, A; Parak, WJ; Soliman, MG; Zhang, Q; Carrillo-Carrion, C
Light-Responsive Nanostructured Systems for Applications in Nanomedicine, **370** (2016) 169-202
Enero, 2016 | **DOI:** http://link.springer.com/chapter/10.1007%2F978-3-319-22942-3_6

In this review, an overview of the current state-of-the-art of gold-based nanomaterials (Au NPs) in medical applications is given. The unique properties of Au NPs, such as their tunable size, shape, and surface characteristics, optical properties, biocompatibility, low cytotoxicity, high stability, and multifunctionality potential, among others, make them highly attractive in many aspects of medicine. First, the preparation methods for various Au NPs including functionalization strategies for selective targeting are summarized. Second, recent progresses on their applications, ranging from the diagnostics to therapeutics are highlighted. Finally, the rapidly growing and promising field of gold-based theranostic nano-platforms is discussed. Considering the great body of existing information and the high speed of its renewal, we chose in this review to generalize the data that have been accumulated during the past few years for the most promising directions in the use of Au NPs in current medical research.

Vacuum template synthesis of multifunctional nanotubes with tailored nanostructured walls

Filippin, AN; Macias-Montero, M; Saghi, Z; Idigoras, J; Burdet, P; Barranco, A; Midgley, P; Anta, JA; Borras, A
Scientific Reports, **5** (2016) 20637
Febrero, 2016 | **DOI:** 10.1038/srep20637

A three-step vacuum procedure for the fabrication of vertical TiO_2 and ZnO nanotubes with three dimensional walls is presented. The method combines physical vapor deposition of small-molecules, plasma enhanced chemical vapor deposition of inorganic functional thin films and layers and a post-annealing process in vacuum in order to remove the organic template. As a result, an ample variety of inorganic nanotubes are made with tunable length, hole dimensions and shapes and tailored wall composition, microstructure, porosity and structure. The fabrication of multishell nanotubes combining different semiconducting oxides and metal nanoparticles is as well explored. This method provides a feasible and reproducible route for the fabrication of high density arrays of vertically alligned nanotubes on processable substrates. The emptying mechanism and microstructure of the nanotubes have been elucidated through SEM, STEM, HAADF-STEM tomography and energy dispersive X-ray spectroscopy. In this article, as a proof of concept, it is presented the straightforward integration of ZnO nanotubes as photoanode in a photovoltaic cell and as a photonic oxygen gas sensor.

Application of Prussian Blue electrodes for amperometric detection of free chlorine in water samples using Flow Injection Analysis

Salazar, P; Martin, M; Gonzalez-Mora, JL; Gonzalez-Elipe, AR
Talanta, **146** (2016) 410-416
Enero, 2016 | **DOI:** 10.1016/j.talanta.2015.08.072

The performance for free chlorine detection of surfactant-modified Prussian Blue screen printed carbon electrodes (SPCEs/PB-BZT) have been assessed by cyclic voltammetry and constant potential amperometry. The characterization of SPCEs/PB-BZT by X-ray photoemission, Raman and infrared spectroscopies confirmed the correct electrodeposition of the surfactant-modified PB film. These electrodes were incorporated in a Flow Injection device and the optimal working conditions determined as a function of experimental variables such as detection potential, electrolyte concentration or flow-rate. The sensor presented a linear response in the range 0–3 ppm free chlorine, with a sensitivity of 16.2 µA ppm−1 cm−2. The limit of detection (LOD) (S/N=3.3) and the limit of quantification (S/N=10) amounted to 8.25 and 24.6 ppb, respectively, adequate for controlling tap and drinking waters. To demonstrate the feasibility of using this free chlorine sensor for real applications possible interferences such as nitrate, nitrite and sulfate ions were successfully tested and discarded. Real free chlorine analysis was carried out in spiked tap water samples and commercial bleaches.

The interaction between hybrid organic-inorganic halide perovskite and selective contacts in perovskite solar cells: an infrared spectroscopy study

Idigoras, J; Todinova, A; Sanchez-Valencia, JR; Barranco, A; Borras, A; Anta, JA
Physical Chemistry Chemical Physics, **18** (2016) 13583-13590
Mayo, 2016 | **DOI:** 10.1039/c6cp01265e

The interaction of hybrid organic-inorganic halide perovskite and selective contacts is crucial to get efficient, stable and hysteresis-free perovskite-based solar cells. In this report, we analyze the vibrational properties of methylammonium lead halide perovskites deposited on different substrates by infrared absorption (IR) measurements (4000-500 cm(-1)). The materials employed as substrates are not only characterized by different chemical natures (TiO2, ZnO and Al2O3), but also by different morphologies. For all of them, we have investigated the influence of these substrate properties on perovskite formation and its degradation by humidity. The effect of selective-hole contact (Spiro-OmeTad and P3HT) layers on the degradation rate by moisture has also been studied. Our IR results reveal the existence of a strong interaction between perovskite and all ZnO materials considered, evidenced by a shift of the peaks related to the N-H vibrational modes. The interaction even induces a morphological change in ZnO nanoparticles after perovskite deposition, pointing to an acid-base reaction that takes place through the NH3+ groups of the methylammonium cation. Our IR and X-ray diffraction results also indicate that this specific interaction favors perovskite decomposition and PbI2 formation for ZnO/perovskite films subjected to humid conditions. Although no interaction is observed for TiO2, Al2O3, and the hole selective contact, the morphology and chemical nature of both contacts appear to play an important role in the rate of degradation upon exposure to moisture.

Luminescent Rare-earth-based Nanoparticles: A Summarized Overview of their Synthesis, Functionalization, and Applications

Escudero, A; Carrillo-Carrion, C; Zyuzin, MV; Parak, WJ
Topics in current chemistry, **374** (2016) Article number 48
Agosto, 2016 | **DOI:** 10.1007/s41061-016-0049-8

Rare-earth-based nanoparticles are currently attracting wide research interest in material science, physics, chemistry, medicine, and biology due to their optical properties, their stability, and novel applications. We present in this review a summarized overview of the general and recent developments in their synthesis and functionalization. Their luminescent properties are also discussed, including the latest advances in the enhancement of their emission luminescence. Some of their more relevant and novel biomedical, analytical, and optoelectronic applications are also commented on.

Multifunctional Eu-doped NaGd(MoO4)(2) nanoparticles functionalized with poly(L-lysine) for optical and MRI imaging

M. Laguna; N.O. Nuñez; V. Rodríguez; E. Cantelar, G. Stepien, M.L. García, J.M. de la Fuente; M. Ocaña
Dalton Transactions, **45** (2016) 16354-16365
Noviembre, 2016 | **DOI:** 10.1039/c6dt02663j

A method for the synthesis of non-aggregated and highly uniform Eu3+ doped NaGd(MoO4)(2) nanoparticles is reported for the first time. The obtained particles present tetragonal structure, ellipsoidal shape and their size can be varied by adjusting the experimental synthesis parameters. These nanoparticles, which were coated with citrate anions and functionalised with PLL, have also been developed in order to improve their colloidal stability in physiological medium (2-(N-morpholino) ethanesulfonic acid, MES). A study of the luminescent dynamics of the samples as a function of the Eu doping level has been conducted in order to find the optimum nanophosphors, whose magnetic relaxivity and cell viability have also been evaluated for the first time for this system, in order to assess their suitability as multifunctional probes for optical (in vitro) and magnetic bioimaging applications.

Modified emission of extended light emitting layers by selective coupling to collective lattice resonances

Ramezani, Mohammad; Lozano, Gabriel; Verschuuren, Marc A.; Gomez-Rivas, Jaime
Physical Review B, **94** (2016) 12
Septiembre, 2016 | **DOI:** 10.1103/PhysRevB.94.125406

We demonstrate that the coupling between light emitters in extended polymer layers and modes supported by arrays of plasmonic particles can be selectively enhanced by accurate positioning of the emitters in regions where the electric field intensity of a given mode is maximized. The enhancement, which we measure to reach up to 70%, is due to the improved spatial overlap and coupling between the optical mode and emitters. This improvement of the coupling leads to a modification of the emission spectrum and the luminous efficacy of the sample.

Ligand-Free Synthesis of Tunable Size Ln:BaGdF5 (Ln = Eu3+ and Nd3+) Nanoparticles: Luminescence, Magnetic Properties, and Biocompatibility

Becerro, AI; Gonzalez-Mancebo, D; Cantelar, E; Cusso, F; Stepien, G; de la Fuente, JM; Ocaña, M
Langmuir, **32** (2016) 411-420
Enero, 2016 | **DOI:** 10.1021/acs.langmuir.5b03837

Bifunctional and highly uniform Ln:BaGdF5 (Ln = Eu3+ and Nd3+) nanoparticles have been successfully synthesized using a solvothermal method consisting of the aging at 120 degrees C of a glycerol solution containing the corresponding Lanthanide acetylacetonates and butylmethylimidazolium tetrafluoroborate. The absence of any surfactant in the synthesis process rendered hydrophilic nanospheres (with tunable diameter from 45 nm 85 nm, depending on the cations concentration of the starting solution) which are suitable for bioapplications. The particles are bifunctional because they showed both optical and magnetic properties due to the presence of the optically active lanthanides (Eu3+ in the visible and Nd3+ in the NIR regions of the electromagnetic spectrum) and the paramagnetic gadolinium ion, respectively. The luminescence decay curves of the nanospheres doped with different amounts of Eu3+ and Nd3+ have been recorded in order to determine the optimum dopant concentration in each case, which turned out to be 5% Eu3+ and 0.5% Nd3+. Likewise, proton relaxation times were measured at 1.5 T in water suspensions of the optimum particles found in the luminescence study. The values obtained suggested that both kinds of particles could be used as positive contrast agents for MRI. Finally, it was demonstrated that both the 5% Eu3+ and 0.5% Nd3+-doped BaGdF5 nanospheres showed negligible cytotoxicity for VERO cells for concentrations up to 0.25 mg mL(-1).

Cellular Viscosity in Prokaryotes and Thermal Stability of Low Molecular Weight Biomolecules

Cuecas, A; Cruces, J; Galisteo-Lopez, JF; Peng, XJ; Gonzalez, JM
Biophysical Journal, **111** (2016) 875–882
Agosto, 2016 | **DOI:** 10.1016/j.bpj.2016.07.024

Some low molecular weight biomolecules, i.e., NAD(P)H, are unstable at high temperatures. The use of these biomolecules by thermophilic microorganisms has been scarcely analyzed. Herein, NADH stability has been studied at different temperatures and viscosities. NADH decay increased at increasing temperatures. At increasing viscosities, NADH decay rates decreased. Thus, maintaining relatively high cellular viscosity in cells could result in increased stability of low molecular weight biomolecules (i.e., NADH) at high temperatures, unlike what was previously deduced from studies in diluted water solutions. Cellular viscosity was determined using a fluorescent molecular rotor in various prokaryotes covering the range from 10 to 100°C. Some mesophiles showed the capability of changing cellular viscosity depending on growth temperature. Thermophiles and extreme thermophiles presented a relatively high cellular viscosity, suggesting this strategy as a reasonable mechanism to thrive under these high temperatures. Results substantiate the capability of thermophiles and extreme thermophiles (growth range 50–80°C) to stabilize and use generally considered unstable, universal low molecular weight biomolecules. In addition, this study represents a first report, to our knowledge, on cellular viscosity measurements in prokaryotes and it shows the dependency of prokaryotic cellular viscosity on species and growth temperature.

In vitro stimulation of MC3T3-E1cells and sustained drug delivery by a hierarchical nanostructured SiO_2-CaO-$P2O_5$ scaffold

Ramiro-Gutierrez, ML; Santos-Ruiz, L; Borrego-Gonzalez, S; Becerra, J; Diaz-Cuenca, A
Microporous and Mesoporous Materials, **229** (2016) 31–43
Julio, 2016 | **DOI:** 10.1016/j.micromeso.2016.04.018

A hierarchical scaffold, SP1_h_HA, consisting of a biomimetic nano-hydroxyapatite surface coating growth onto a reticulated structure having a nano-organized porous texture was fabricated and functionally studied in vitro using osteoprogenitor cells. Three scaffold materials (designated as SP0_l, SP0_h and SP1_h) were also prepared through modifications of the processing variables as control materials. The scaffolds were characterized showing well-interconnected micron-sized voids and a nano (4–6 nm)-organized porosity. In order to evaluate potential local risks and performance over mammalian cells the scaffolds were studied in comparison with a commercial clinical grade scaffold material, ProOsteon® 500R. MC3T3-E1 pre-osteoblast viability was evaluated using the resazurin assay and field emission gun scanning electron microscopy (FEG-SEM), showing in all cases good proliferative response. Alkaline phosphatase (ALP) production and analysis of the differentiation marker osteocalcin (OC), both in non-osteoinductive and osteoinductive media, were assessed using colorimetric and RT-PCR methods. The implementation of the new scaffold processing variables enhanced ALP activity with respect to the SP0_l control material. The cell proliferation, ALP activity, and mRNA OC expression response to SP1_h_HA scaffold were higher than those observed after the use of ProOsteon® 500R. In addition, SP1_h_HA scaffold showed a two stage sustained release of gentamicin sulfate (GS) instead of the quick release shown by ProOsteon® 500R. These results suggest that our synthesized scaffold could be effective for antibiotic delivery and bone regeneration and a better option than ProOsteon® 500R.

Pre-prosthetic use of poly(lactic-co-glycolic acid) membranes treated with oxygen plasma and TiO2 nanocomposite particles for guided bone regeneration processes

Castillo-Dali, G; Castillo-Oyague, R; Terriza, A; Saffar, JL; Batista-Cruzado, A; Lynch, CD; Sloan, AJ; Gutierrez-Perez, JL; Torres-Lagares, D

Journal of Dentistry, **47** (2016) 71-79

Abril, 2016 | **DOI:** 10.1016/j.jdent.2016.01.015

Objectives: Guided bone regeneration (GBR) processes are frequently necessary to achieve appropriate substrates before the restoration of edentulous areas. This study aimed to evaluate the bone regeneration reliability of a new poly-lactic-co-glycolic acid (PLGA) membrane after treatment with oxygen plasma (PO2) and titanium dioxide (TiO2) composite nanoparticles.
Methods: Circumferential bone defects (diameter: 10 mm; depth: 3 mm) were created on the parietal bones of eight experimentation rabbits and were randomly covered with control membranes (Group 1: PLGA) or experimental membranes (Group 2: PLGA/PO2/TiO2). The animals were euthanized two months afterwards, and a morphologic study was then performed under microscope using ROI (region of interest) colour analysis. Percentage of new bone formation, length of mineralised bone formed in the grown defects, concentration of osteoclasts, and intensity of osteosynthetic activity were assessed. Comparisons among the groups and with the original bone tissue were made using the Kruskal-Wallis test. The level of significance was set in advance at a = 0.05.
Results: The experimental group recorded higher values for new bone formation, mineralised bone length, and osteoclast concentration; this group also registered the highest osteosynthetic activity. Bone layers in advanced formation stages and low proportions of immature tissue were observed in the study group.

Nanocolumnar association and domain formation in porous thin films grown by evaporation at oblique angles

Lopez-Santos, C; Alvarez, R; Garcia-Valenzuela, A; Rico, V; Loeffler, M; Gonzalez-Elipe, AR; Palmero, A

Nanotechnology, **27** (2016) 395702
Septiembre, 2016 | **DOI:** 10.1088/0957-4484/27/39/395702

Porous thin films grown at oblique angles by evaporation techniques are formed by tilted nanocolumnar structures which, depending on the material type and growth conditions, associate along certain preferential directions, giving rise to large domains. This arrangement, commonly denoted as bundling association, is investigated in the present work by performing fundamental experiments and growth simulations. It is proved that trapping processes of vapor species at the film surface, together with the shadowing mechanism, mediate the anisotropic widening of the nanocolumns and promote their preferential coalescence along certain directions, giving rise to domains with different shape and size. The role of these two processes is thoroughly studied in connection with the formation of these domains in materials as different as SiO_2 and TiO_2.

Metallization of ceramic substrates by laser induced decomposition of coordination complexes

Rico, V; Lopez-Gascon, C; Espinos, JP; Lahoz, R; Laguna, M; Gonzalez-Elipe, AR; de la Fuente, GF
Journal of the European Ceramic Society, **36** (2016) 2831-2836
Septiembre, 2016 | **DOI:** 10.1016/j.jeurceramsoc.2016.04.016

This work describes an in-situ Nd:YAG laser-assisted coating method to modify industrial glazed ceramic surfaces. The method makes use of a Cu polymer coordination complex, transformed via 1064 nm continuos wave (cw) laser irradiation, into a lustre-type glassy coating covering the ceramic substrate. The obtained coatings, with typical thicknesses ranging between 4 and 14 µm, become integrated onto the ceramic glaze via a sharp interface, as found by SEM observation. Diffuse Reflectance UV-vis spectroscopy shows that the lustre effect arises from surface plasmon resonant effects associated to the formation of nanometric size Cu particles dispersed throughout the glaze coating. This was confirmed by XPS analysis and other techniques showing that the laser decomposition treatment induces the redox transformation of the Cu (II) complexes, present in the original precursor, into reduced Cu (0) nanoparticles.

Portable IR dye laser optofluidic microresonator as a temperature and chemical sensor

ahoz, F; Martin, IR; Gil-Rostra, J; Oliva-Ramirez, M; Yubero, F; Gonzalez-Elipe, AR
Optics Express, **24** (2016) 14383-14392
Junio, 2016 | **DOI:** 10.1364/OE.24.014383

A compact and portable optofluidic microresonator has been fabricated and characterized. It is based on a Fabry-Perot microcavity consisting essentially of two tailored dichroic Bragg mirrors prepared by reactive magnetron sputtering deposition. The microresonator has been filled with an ethanol solution of Nile-Blue dye. Infrared laser emission has been measured with a pump threshold as low as 0.12 MW/cm2 and an external energy conversion efficiency of 41%. The application of the device as a temperature and a chemical sensor is demonstrated. Small temperature variations as well as small amount of water concentrations in the liquid laser medium are detected as a shift of the resonant laser modes.

Non-Enzymatic Glucose Sensors Based on Nickel Nanoporous Thin Films Prepared by Physical Vapor Deposition at Oblique Angles for Beverage Industry Applications

Salazar, P; Rico, V; Gonzalez-Elipe, AR
Journal of the Electrochemical Society, **163** (2016) B704-B709
Diciembre, 2016 | **DOI:** 10.1149/2.1241614jes

Nickel nanoporous thin films deposited on Indium tin oxide conductive plates have been prepared by physical vapor deposition in an oblique angle configuration. The scanning electron microscopy characterization of these films revealed a microstructure formed by tilted nanocolumns of ca. 40-60 nm of diameter inclined by ca. 26 degrees with respect to the normal. These highly porous films had ca. 30% of void space and provided a large exposed area and outstanding diffusion properties for sensor applications. X-ray diffraction analysis confirmed the deposition of metallic nickel, while Raman and X-ray photoelectron spectroscopies demonstrated that electrochemically treated films presented an oxi/hydroxide outer layer that is the active phase for glucose sensing. The activated electrodes had a high sensitivity (2.05 A M-1 cm(-2)), an excellent coefficient of determination (R-2: 0.999), an outstanding reproducibility (3.2%) and a detection limit of 0.34 mu M. Their glucose selectivity was excellent with regard to common electroactive interferences and other sugars found in agro-alimentary products. Tests carried out with commercial beverages proved the reliability of these electrodes for glucose analysis in real conditions.

Effect of Nickel and Magnesium on the Electrochemical Behavior of AA 1050 Alloys in Nitric Acid Solution

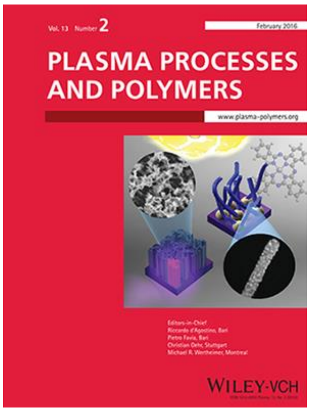
Garcia-Garcia, FJ; Skeldon, P; Thompson, GE
Journal of the Electrochemical Society, **163** (2016) C593-C601
Septiembre, 2016 | **DOI:** 10.1149/2.1181609jes

The study investigates the influence of nickel and magnesium additions to AA 1050 aluminum alloy on the electrochemical behavior of the alloy in nitric acid solution under conditions relevant to the lithographic and electronic industries. Magnesium and nickel additions are of interest, since they can improve the alloy properties for the printing process by improving reverse bending fatigue strength and thermal softening resistance, while nickel may provide uniform pitting during electrograining. Scanning electron microscopy was used to characterize the resulting surface morphologies. The addition of nickel led to an increase in the pitting and corrosion potentials; additionally, it reduced the rate of dissolution of intermetallic particles during anodic polarization and increased the rate of aluminum dissolution during cathodic polarization. In contrast, the addition of magnesium had negligible influence on the open circuit and pitting behaviors, since the magnesium is retained in solid solution and has negligible influence on the cathodic behavior of intermetallic particles, which dominate the corrosion behavior.

Ripening and recrystallization of NaCl nanocrystals in humid conditions

Oliva-Ramirez, M; Macias-Montero, M; Borras, A; Gonzalez-Elipe, AR
RSC Advances, **6** (2016) 3778-3782
Marzo, 2016 | **DOI:** 10.1039/C5RA22425J

This study shows that Ostwald ripening, a universal mechanism responsible for the increase of crystal size during precipitation from solutions, can be meditated by ion diffusion through condensed monolayers of water that connect separated nanocrystals. In an environmental electron microscope we have observed "in situ" the time evolution of the number, shape, size and crystallographic texture of NaCl nanoparticles deposited by electron beam evaporation at oblique angles. Analysis of NaCl nanoparticles before and after water vapor condensation has evidenced that the size of nanocrystals is not the unique driving force inducing nanoparticle ripening and recrystallization, but the faceting of their crystalline habits and the amorphisation degree of the initially deposited nuclei also play important roles. These findings have implications for other crystallization and nucleation processes and can be of relevance for rock weathering and related phenomena.



Highly Porous ZnO Thin Films and 1D Nanostructures by Remote Plasma Processing of Zn-Phthalocyanine

Alcaire, M; Filippin, AN; Macias-Montero, M; Sanchez-Valencia, JR; Rojas, TC; Mora-Boza, A; Lopez-Santos, C; Espinos, JP; Barranco, A; Borras, A

Plasma Processes and Polymers, **13** (2016) 287-297

Febrero, 2016 | **DOI:** 10.1002/ppap.201500133

In this paper the fabrication of highly porous 1D nanostructures by a vacuum and plasma etching combined protocol is presented. Zn-phthalocyanine (ZnPc) is utilized as a solid precursor to form the ZnO. First the ZnPc is sublimated in low argon pressure. Depending on the substrate temperature and microstructure, polycrystalline films or single crystal ZnPc nanowires are grown. These starting materials are then subjected to a remote plasma oxidizing treatment. Experimental parameters such as substrate position, plasma power, treatment duration, and substrate temperature determine the microstructure and properties of the final ZnO nanostructures. The article gathers an in depth study of the obtained porous nanostructured films following scanning and transmission electron microscopy (SEM and TEM), X-ray photoelectron spectroscopy (XPS), X-ray Diffraction (XRD), UV-Vis transmittance, and fluorescence spectroscopies.

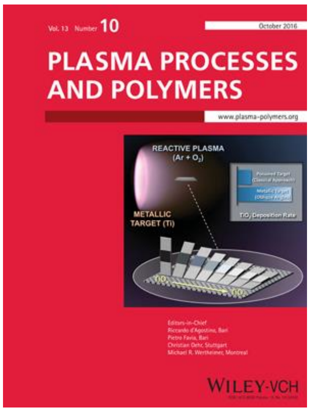
Stoichiometric Control of SiOx Thin Films Grown by Reactive Magnetron Sputtering at Oblique Angles

Garcia-Valenzuela, A; Alvarez, R; Lopez-Santos, C; Ferrer, FJ; Rico, V; Guillen, E; Alcon-Camas, M; Escobar-Galindo, R; Gonzalez-Elipe, AR; Palmero, A

Plasma Processes and Polymers, **13** (2016) 1242-1248

Diciembre, 2016 | **DOI:** 10.1002/ppap.201600077

The deposition of SiOx ($x <= 2$) compound thin films by the reactive magnetron sputtering technique at oblique angles is studied from both theoretical and experimental points of view. A simple mathematical formula that links the film stoichiometry and the deposition conditions is deduced. Numerous experiments have been carried out to test this formula at different deposition pressures and oblique angle geometries obtaining a fairly good agreement in all studied conditions. It is found that, at low deposition pressures, the proportion of oxygen with respect to silicon in the film increases a factor of similar to 5 when solely tilting the film substrate with respect to the target, whereas at high pressures the film stoichiometry depends very weakly on the tilt angle. This behavior is explained by considering the fundamental processes mediating the growth of the film by this technique.



High-Rate Deposition of Stoichiometric Compounds by Reactive Magnetron Sputtering at Oblique Angles

Rafael Alvarez, Aurelio Garcia-Valenzuela, Carmen Lopez-Santos, Francisco J. Ferrer, Victor Rico, Elena Guillen, Mercedes Alcon-Camas, Ramon Escobar-Galindo, Agustin R. Gonzalez-Elipe, Alberto Palmero

Plasma Processes and Polymers, **13** (2016) 571-576
Octubre, 2016 | **DOI:** 10.1002/ppap.201600019

Target poisoning in reactive magnetron sputtering deposition of thin films is an undesired phenomenon, well known for causing a drastic fall of the process efficiency. We demonstrate that when this technique is operated at oblique angles, films with composition raging from pure metallic to stoichiometric compound can be grown in non-poisoned conditions, thus avoiding most of the associated drawbacks. We have employed amorphous TiOx, although the presented results can be easily extrapolated to other materials and conditions. It is found that the proposed method improves 400% the growth rate of TiO2 thin films.

A novel 3D absorption correction method for quantitative EDX-STEM tomography

Burdet, P; Saghi, Z; Filippin, AN; Borras, A; Midgley, PA

Ultramicroscopy, **160** (2016) 118-129
Enero, 2016 | **DOI:** 10.1016/j.ultramic.2015.09.012

This paper presents a novel 3D method to correct for absorption in energy dispersive X-ray (EDX) microanalysis of heterogeneous samples of unknown structure and composition. By using STEM-based tomography coupled with EDX, an initial 3D reconstruction is used to extract the location of generated X-rays as well as the X-ray path through the sample to the surface. The absorption correction needed to retrieve the generated X-ray intensity is then calculated voxel-by-voxel estimating the different compositions encountered by the X-ray. The method is applied to a core/shell nanowire containing carbon and oxygen, two elements generating highly absorbed low energy X-rays. Absorption is shown to cause major reconstruction artefacts, in the form of an incomplete recovery of the oxide and an erroneous presence of carbon in the shell. By applying the correction method, these artefacts are greatly reduced. The accuracy of the method is assessed using reference X-ray lines with low absorption.

Cathode and ion-luminescence of Eu:ZnO thin films prepared by reactive magnetron sputtering and plasma decomposition of non-volatile precursors

Gil-Rostra, J; Ferrer, FJ; Martin, IR; Gonzalez-Elipe, AR; Yubero, F

Journal of Luminescence, **178** (2016) 139-146
Octubre, 2016 | **DOI:** 10.1016/j.jlumin.2016.01.034

This paper reports the luminescent behavior of Eu:ZnO thin films prepared by an one-step procedure that combines reactive magnetron sputtering deposition of ZnO with the plasma activated decomposition of a non-volatile acetylacetonate precursor of Eu sublimated in an effusion cell. Chemical composition and microstructure of the Eu:ZnO thin films have been characterized by several methods and their photo-, cathode- and ion-luminescent properties studied as a function of Eu concentration. The high transparency and well controlled optical properties of the films have demonstrated to be ideal for the development of cathode- and ion-luminescence sensors.

Nanoindentation and scratch resistance of multilayered TiO_2-SiO_2 coatings with different nanocolumnar structures deposited by PV-OAD

Roa, JJ; Rico, V; Oliva-Ramirez, M; Gonzalez-Elipe, AR; Jimenez-Pique, E
Journal of Physics D-Applied Physics, **49** (2016) 13
Abril, 2016 | **DOI:** 10.1088/0022-3727/49/13/135104

This paper presents a study of the mechanical properties and an evaluation of damage mechanisms of nanocolumnar TiO_2-SiO_2 multilayer coatings prepared by physical vapour oblique angle deposition at different configurations (slanted, zigzag or chiral) and two zenithal evaporation angles (70 degrees or 85 degrees). The characterization at micro-and nanometric length scales of the mechanical properties of the multilayers has been carried out by nanoindentation and nanoscratch tests, while the morphological evaluation of the surface and sub-surface damages produced with a sharp indenter and the adhesive and/or cohesive failures between coating and substrate have been investigated by field emission scanning electron microscopy and focused ion beam, respectively. The obtained results have shown that the main processing parameters controlling the mechanical response of the different multilayers is the zenithal angle of deposition and the number of layers in the multilayer stack, while the coating architecture had only a minor effect on the mechanical response. This analysis also revealed a higher resistance to scratch testing and a brittle failure behaviour for the low zenithal angle coatings as compared with the high angle ones.

Nanostructured Ti thin films by magnetron sputtering at oblique angles

Alvarez, R; Garcia-Martin, JM; Garcia-Valenzuela, A; Macias-Montero, M; Ferrer, FJ; Santiso, J; Rico, V; Cotrino, J; Gonzalez-Elipe, AR; Palmero, A
Journal of Physics D-Applied Physics, **49** (2016) 045303
Febrero, 2016 | **DOI:** 10.1088/0022-3727/49/4/045303

The growth of Ti thin films by the magnetron sputtering technique at oblique angles and at room temperature is analysed from both experimental and theoretical points of view. Unlike other materials deposited in similar conditions, the nanostructure development of the Ti layers exhibits an anomalous behaviour when varying both the angle of incidence of the deposition flux and the deposition pressure. At low pressures, a sharp transition from compact to isolated, vertically aligned, nanocolumns is obtained when the angle of incidence surpasses a critical threshold. Remarkably, this transition also occurs when solely increasing the deposition pressure under certain conditions. By the characterization of the Ti layers, the realization of fundamental experiments and the use of a simple growth model, we demonstrate that surface mobilization processes associated to a highly directed momentum distribution and the relatively high kinetic energy of sputtered atoms are responsible for this behaviour.

Confinement and surface effects on the physical properties of rhombohedral-shape hematite (alpha-Fe2O3) nanocrystals

Luna, C; Cuan-Guerra, AD; Barriga-Castro, ED; Nunez, NO; Mendoza-Resendez, R
Materials Research Bulletin, **80** (2016) 44-52
Agosto, 2016 | **DOI:** 10.1016/j.materresbull.2016.03.029

Morphological, microstructural and vibrational properties of hematite (alpha-Fe2O3) nanocrystals with a rhombohedral shape and rounded edges, obtained by forced hydrolysis of iron(III) solutions under a fast nucleation, have been investigated in detail as a function of aging time. These studies allowed us to propose a detailed formation mechanism and revealed that these nanocrystals are composed of four {104} side facets, two {110} faces at the edges of the long diagonal of the nanocrystals and two {-441} facets as the top and bottom faces. Also, the presence of nanoscopic pores and fissures was evidenced. The vibrational bands of such nanocrystals were shifted to lower frequencies in comparison with bulk hematite ones as the nanocrystal size was reduced due to phonon confinement effects. Also, the indirect and direct transition band gaps displayed interesting dependences on the aging time arising from quantum confinementand surface effects.

Effect of temperature variations on equilibrium distances in levitating parallel dielectric plates interacting through Casimir forces

Esteso, V; Carretero-Palacios, S; Miguez, H
Journal of Applied Physics, **119** (2016) 144301
Abril, 2016 | **DOI:** 10.1063/1.4945428

We study at thermal equilibrium the effect of temperature deviations around room temperature on the equilibrium distance (d(eq)) at which thin films made of Teflon, silica, or polystyrene immersed in glycerol levitate over a silicon substrate due to the balance of Casimir, gravity, and buoyancy forces. We find that the equilibrium nature (stable or unstable) of d(eq) is preserved under temperature changes, and provide simple rules to predict whether the new equilibrium position will occur closer to or further from the substrate at the new temperature. These rules depend on the static permittivities of all materials comprised in the system (epsilon((m))(0)) and the equilibrium nature of d(eq). Our designed dielectric configuration is excellent for experimental observation of thermal effects on the Casimir force indirectly detected through the tunable equilibrium distances (with slab thickness and material properties) in levitation mode.

Quantitative analysis of Ni 2p photoemission in NiO and Ni diluted in a SiO2 matrix

Pauly, N; Yubero, F; Garcia-Garcia, FJ; Tougaard, S
Surface Science, **644** (2016) 46-52
Febrero, 2016 | **DOI:** 10.1016/j.susc.2015.09.012

In X-ray excited photoelectron emission (XPS), besides the initial excitation process, the shape and intensity of photoelectron peaks are strongly affected by extrinsic excitations due to electron transport out of the surface (including bulk and surface effects) and to intrinsic excitations due to the sudden creation of the static core hole. To make an accurate quantitative interpretation of features observed in XPS, these effects must be included in the theoretical description of the emitted photoelectron spectra. It was previously shown [N. Pauly, S.

Tougaard, F. Yubero, Surf. Sci. 620 (2014) 17] that these three effects can be calculated by means of the QUEELS-XPS software (Quantitative analysis of Electron Energy Losses at Surfaces for XPS) in terms of effective energy-differential inelastic electron scattering cross-sections. The only input needed to calculate these cross-sections is the energy loss function of the media which is determined from analysis of Reflection Electron Energy Loss Spectra (REELS). The full XPS spectrum is then modeled by convoluting this energy loss cross-section with the primary excitation spectrum that accounts for all effects which are part of the initial photo-excitation process, i.e. lifetime broadening, spin-orbit coupling, and multiplet splitting. In this paper we apply the previously presented procedure to the study of Ni 2p photoemission in NiO and Ni diluted in a SiO2 matrix (Ni:SiO2), samples being prepared by reactive magnetron sputtering at room temperature. We observe a significant difference between the corresponding Ni 2p primary excitation spectra. The procedure allows quantifying the relative intensity of the c3d(9)L, c3d(10)L(2), and c3d(8) final states contributing to the Ni 2p photoemission spectra of the Ni2+ species in the oxide matrices. Especially, the intensity ratio in NiO between the non-local and local contributions to the 3d(9)L configuration is determined to be 2.5. Moreover the relative intensity ratio of the c3d(9)L/c3d(10)L(2)/c3d(8) configurations is found to be 1.0/0.83/0.11 for both the NiO and Ni:SiO2 samples.

Deposition of silica protected luminescent layers of Eu:GdVO4 nanoparticles assisted by atmospheric pressure plasma jet

Moretti, E; Pizzol, G; Fantin, M; Enrichi, F; Scopece, P; Nunez, NO; Ocaña, M; Benedetti, A; Polizzi, S
Thin Solid Films, **598** (2016) 88-94
Enero, 2016 | **DOI:** 10.1016/j.tsf.2015.11.061

Eu:GdVO4 nanophosphors with an average size of 60 nm, synthesized by a facile solvothermal method, were deposited on monocrystalline silicon wafers by a spray-coating technique with artworks anti-counterfeiting applications in mind. Atmospheric pressure plasma jet (APPJ) was used to deposit a silica-based layer on top of the nanometric luminescent layer, in order to improve its adhesion to the substrate and to protect it from the environment. The nanophosphors were characterized by X-ray diffraction (XRD) and transmission electron microscopy (TEM). Coating composition was investigated by Fourier transform infrared spectroscopy (FT-IR) and its morphology was characterized by scanning electron microscopy (FEG-SEM). The film thickness was evaluated by means of ellipsometry and adhesion was estimated by a peeling test. Luminescent properties of the nanophosphors deposited and fixed on silicon wafers were also measured. The whole layer resulted well-adhered to the silicon substrate, transparent and undetectable in the presence of visible light, but easily activated by UV light source.

Plasmonic Nanoparticles as Light-Harvesting Enhancers in Perovskite Solar Cells: A User's Guide

Carretero-Palacios, S.; Jiménez-Solano, A.; Míguez, H.
ACS Energy Letters, **1** (2016) 323-331
Junio, 2016 | **DOI:** 10.1021/acsenergylett.6b00138

In this Perspective we discuss the implications of employing metal particles of different shape, size, and composition as absorption enhancers in methylammonium lead iodide perovskite solar cells, with the aim of establishing some guidelines for the future development of plasmonic resonance-based photovoltaic devices. Hybrid perovskites present an extraordinarily high absorption coefficient which, as we show here, makes it difficult to extrapolate concepts and designs that are applied to other solution-processed photovoltaic materials. In addition, the variability of the optical constants attained from perovskite films of seemingly similar composition further complicates the analysis. We demonstrate that, by means of rigorous design, it is possible to provide a realistic prediction of the magnitude of the absorption enhancement that can be reached for perovskite films embedding metal particles. On the basis of this, we foresee that localized surface plasmon effects will provide a means to attain highly efficient perovskite solar cells using films that are thinner than those usually employed, hence facilitating collection of photocarriers and significantly reducing the amount of potentially toxic lead present in the device.

ARTICULOS PUBLICADOS EN REVISTAS (NO SCI) / PAPERS IN NON-SCI JOURNALS

Consiguen Fabricar Celdas Solares de Colores
Miguel Anaya
Revista Muy Interesante
Enero 2016

Perovskita, un material protagonista en el último lustro
Miguel Anaya
La cuadratura del Círuclo.
Blog del CSIC Andalucía
Septiembre 2016

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

Grupo Especializado del Estado Sólido de la Real Sociedad Española de Física. IX GEFES 2016 Meeting

13 - 15 enero [Cuenca, España]

Photonic structures for colorful perovskite solar cells
M. Anaya, G. Lozano, M.E. Calvo, H. Míguez
Comunicación oral

Efficient bifacial dye-sensitized solar cells through disorder by design
J.M.Miranda-Muñoz, S. Carretero-Palacios, A. Jiménez-Solano, Y. Li, G. Lozano, H. Míguez
Poster

Fine tuning of the emission properties of nano-emitters in multilayered structures by deterministic control of their local photonic environment
A. Jiménez Solano, J.F. Galisteo López, H. Míguez
Poster

Levitation of self-standing thin films due to the Casimir force
V. Esteso, S. Carretero-Palacios, H. Míguez
Poster

SPIE Photonics West 2016

13 - 18 febrero [San Francisco, Estados Unidos de América]

Optical Design in Perovskite Solar Cells
M. Anaya, G. Lozano, S. Carretero-Palacios, M.E. Calvo, H. Míguez
Conferencia invitada

Efficient bifacial dye-sensitized solar cells through disorder by design
J.M. Miranda-Muñoz, S. Carretero-Palacios, A. Jiménez-Solano, Y. Li, G. Lozano, H. Míguez
Comunicación oral

nanoPortugal International Conference 2016

16 - 19 febrero [Braga, Portugal]

Nanostructured biocompatible coatings to prevent implant infections
J.M. Garcia-Martin, I. Izquierdo-Barba, D. Arcos, R. Alvarez, A. Palmero, J. Esteban, C. Perez-Jorge, M. Vallet-Regi
Comunicación oral

One Step Synthesis and Polyacrylic Acid Functionalization of Multifunctional Eu-doped NaGdF4 Nanoparticles with Selected Size for Optical and MRI Imaging
N.O. Nuñez, M. García, J. García-Sevillano, S. Rivera-Fernández, J.M. de la Fuente, M. Ocaña
Poster

International Conference on Perovskite Thin Film Photovoltaics ABXPV 2016

3 - 4 marzo [Barcelona, España]

Environmental Effectson the Photophysics of CH3NH3PbI3
J.F. Galisteo-López, M. Anaya, M.E. Calvo, H. Míguez
Comunicación oral

Optical description of perovskite solar cells
G. Lozano, M. Anaya, M.E Calvo, W. Zhang, M.B. Johnston, H.J. Snaith, H. Míguez
Comunicación oral

Nanolight 2016
6 - 11 marzo [Benasque, España]

Solution Processed Optical Materials for Optoelectronic Devices
H. Míguez
Conferencia invitada

Full solution process approach for deterministic control of light emission at the nanoscale
A. Jiménez Solano, J.F. Galisteo López, H, Míguez
Poster

NanoSpain Conference 2016
15 - 18 marzo [Logroño, España]

Tunable size Ln:BaGdF5 (Ln= Eu3+ and Nd3+) nanoparticles. Luminescence, magnetic properties, and biocompatibility
D. González-Mancebo, E. Cantelar, F. Cussó, G. Stepien, J.M. de la Fuente, M. Ocaña, A.I. Becerro
Poster

SPIE Photonics Europe 2016
4 - 7 abril [Bruselas, Bélgica]

Full solution process approach for deterministic control of light emission at the nanoscale
A. Jiménez Solano, J. Galisteo López, H. Míguez
Comunicación oral

Casimir and van der Waals Physics: Progress and Prospects
25 - 28 abril [Hong Kong]

Temperature dependence of the equilibrium distance of levitating thin films due to the Casimir force
V. Esteso, S. Carretero-Palacios, H. Míguez
Poster

E-MRS Spring Meeting 2016
2 - 6 mayo [Lille, Francia]

Full solution-process approach for a deterministic control of the photonic environment of nanoemitters
J.F. Galisteo-López, A. Jiménez-Solano, H. Míguez
Comunicación oral

Optofluidic Modulation and Liquid Monitoring with Self-Associated Nanostructural Arrays Forming Planar Bragg Microcavities
M. Oliva-Ramírez, A. Barranco, M. Löffler, F.Yubero, A.R.González-Elipe
Comunicación oral

In Operando Monitoring of Structural Changes in WO3 and WxSiyOz Electrochromic Thin Films
F.J. García-García, J. Gil-Rostra, F. Yubero, J.P. Espinós, A.R. González-Elipe
Comunicación oral

Photophysics of CH3NH3PbI3 as a characterization tool: from environmental effects to material formation
J.F. Galisteo-López, M. Anaya, M.E. Calvo, H. Míguez
Comunicación oral

Optical management in perovskite solar cells
M. Anaya, G. Lozano, M. Calvo, H. Míguez
Comunicación oral

Adsorption of nitrile species on Ag/Al2O3 nanostructures exposed to the atmosphere and produced by laser techniques
R.J. Peláez, J.P. Espinós
Poster

Glass and Optical Materials Division Annual Meeting 2016
22 - 26 mayo [Wisconsin, Estados Unidos de América]

Preparation of Lithium Disilicate Glass-Ceramics by the Sol-Gel Method
H.A. Celik, L.B. Romero-Sánchez, A. Díaz Cuenca, E. Dolekcekic
Comunicación oral

The International Conference on Hybrid and Organic Photovoltaics HOPV16
29 junio - 1 julio [Swansea, Reino Unido]

Spatial and temporal fluctuations of the photophysics of perovskite films
M. Anaya, J. Galisteo, M.E. Calvo, H. Míguez
Comunicación oral

6th International Conference on NANOstructures and nanomaterials SElf-Assembly NanoSEA 2016
3 - 8 julio [Giardini Naxos, Italia]

Fabrication of black gold coatings by glancing angle deposition with sputtering
A. Vitrey, R. Alvarez, A. Palmero, M.U. González, J.M. García-Martín
Comunicación oral

Workshop on bio-inspired nanosystems for energy conversion

11 - 15 julio [Berlin, Alemania]

Optical materials and designs for perovskite optoelectronic devices
H. Míguez García
Conferencia invitada

7th International Conference on Metamaterials, Photonic Crystals and Plasmonics META 16

25 - 28 julio [Málaga, España]

Full-solution process approach for tailoring light-matter interaction at the nanoscale
J.F. Galisteo-López, A. Jiménez-Solano, H. Miguez
Conferencia invitada

6th EuCheMS Chemistry Congress

11 - 15 septiembre [Sevilla, España]

Physical vapor deposited thin films as template for chemical processing of nanostructured materials and advanced chemical devices
A.R. González-Elipe
Conferencia invitada

Water-dispersible rare earth molybdates and wolframates nanoparticles for (magnetic?) imaging and sensing applications
A. Escudero, M. Laguna, N.O. Núñez, M. Ocaña
Poster

15th International Conference on Plasma Surface Engineering

12 – 16 septiembre [Garmisch-Partenkrchen, Alemania]

Stoichiometric Control of Compound Thin Films Grown by Reactive Magnetron Sputtering at Oblique Angles
A.Palmenro, A. García-Valenzuela, R. Alvarez, C. López Santos, F.J. Ferrer, V. Rico, M. Alcon-Camas, E. Guillen, R. Escobar, A.R. González-Elipe
Comunicación oral

Organic Nanomaterials and Nanostructures iPlasmaNano-VII 2016

16 - 20 octubre [Vravrona, Grecia]

Multifunctional Organic Thin Films by Remote Plasma Assisted Vacuum Deposition
A. Barranco
Conferencia invitada

From one to three dimensional nanostructures by extending the core@shell concept
A.N. Filippin, M. Alcaire, J.R. Sanchez-Valencia, F.J. Aparicio, M. Macias-Montero, A. Barranco, A. Borras
Conferencia invitada

Global Congress & Expo on Materials Science & Nanoscience

24 - 26 octubre [Dubai, Emiratos Arabes Unidos]

Optical characterization of Eu3+:NaGdF4 luminescent nano-phosphors
E. Cantelar, J. García-Sevillano, F. Cussó, N.O. Nuñez, M. Ocaña, J.E. Muñoz-Santiuste
Comunicación oral

Plasma Surfaces in Healthcare and Industry

30 noviembre - 1 diciembre [Braunschweig, Alemania]

Multifunctional organic thin films by remote plasma assisted vacuum deposition
A. Barranco
Conferencia invitada

Applied Nanotechnology and Nanoscience International Conference ANNIC 2016

9 - 11 noviembre [Barcelona, España]

Poly(L-lysine) functionalized Eu-doped NaGd(MoO4)2 Nanophosphors for Optical and MRI Imaging
M. Ocaña, N. Nuñez, M. Laguna, J.M. De La Fuente, E. Cantelar, M. Garcia
Poster

Thin Film Emerging Photovoltaic and Optoelectronic Technologies (EMTECH) 2016

17 - 18 noviembre [Valencia, España]

Relevance of Optical Design for Perovskite Solar Cell Devices: from Structural Color to Absorption Enhancement
H. Míguez
Conferencia invitada

OSA Congress: Light, Energy and Environment 2016

14 - 17 noviembre [Leipzig, Alemania]

Nanophotonics for color conversion in solid-state lighting
G. Lozano, D. Geng, J. Miranda Muñoz, H. Míguez
Comunicación oral

Color Tuning of GdVO4:Dy3+ Nanophosphor via photonic multilayers
D. Geng, G. Lozano, N. Núñez, A. Becerro, M. Ocaña, H. Míguez
Poster

E-MRS Fall Meeting 2016

27 noviembre - 2 diciembre [Boston, Estados Unidos de América]

Optical design as a tool to attain perovskite based single and double junction solar cells of superior performance
M. Anaya, G. Lozano, M. Calvo, H. Míguez
Comunicación oral

CONGRESOS Y REUNIONES NACIONALES / NATIONAL CONGRESS AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

XIV Congreso Nacional de Materiales

8 - 10 junio [Gijón, España]

Capas nanoestructuradas preparadas por evaporación a ángulo rasante para la detección optofluídica y electroquímica de azúcares
M. Oliva-Ramírez, P. Salazar-Carballo, F. Yubero, A.R. González-Elipe
Comunicación oral

58th Polish Crystallography Meeting

22 junio [Breslavia, Polonia]

Is it possible to change the well-known diagram of phase transitions?
P. Tomaszewski, A.I. Becerro, A. Fernández Carrion
Comunicación oral

Conferencia Española de Nanofotónica CEN 2016

20 - 22 junio [Valencia, España]

Theoretical prediction of levitation due to Casimir force of plane-parallel systems made of realistic materials
V. Esteso, S. Carretero-Palacios, H. Míguez
Conferencia invitada

Nanosources shine on demand thanks to a deterministic control of the local density of states
A. Jiménez Solano, J.F. Galisteo López, H. Míguez
Conferencia invitada

Nanosources shine on demand thanks to a deterministic control of the local density of states
A. Jiménez Solano, J.F. Galisteo López, H. Míguez
Poster

Tailoring disorder for absorption enhancement in bifacial dye-sensitized solar cells
J.M. Miranda-Muñoz, S. Carretero-Palacios, A. Jiménez-Solano, Y. Li, G. Lozano, H. Míguez
Poster

Tailoring disorder for absorption enhancement in bifacial dye-sensitized solar cells
J.M. Miranda-Muñoz, S. Carretero-Palacios, A. Jiménez-Solano, Y. Li, G. Lozano, H. Míguez
Poster

LV Congreso de la Sociedad Española de Cerámica y Vidrio (SECV) Y I Congreso Bienal de la SECV

5 - 7 octubre [Sevilla, España]

Fósforos uniformes basados en CaMO4 con diferentes morfologías: síntesis y dopado con iones lantánidos (Dy3+, Eu3+) para emisión de luz blanca
N.O. Núñez, M. Ocaña, M. Laguna
Comunicación oral

XXXVII Congreso Nacional de Endodoncia AEDE

28 - 30 octubre [San Sebastián, España]

Vidrios Bioactivos Nanoporosos Ordenados de Zinc para Aplicaciones en Endodoncia
M.C. Jiménez Sánchez, L. Beatriz Romero Sánchez, J. Martín González, J.J. Segura Egea, A. Díaz Cuenca
Poster

FORMACION / TRAINING

TESIS DOCTORALES/ DOCTOR DEGREE THESIS

Título: **Optofluidic thin-film sensors prepared by oblique angle deposition**
Autor: Manuel Oliva Ramírez
Directores: Francisco Yubero Valencia, Agustín R. González-Elipe
Calificación: Apto "Cum Laude" por Unanimidad
Centro: Universidad de Sevilla
Fecha: 22 de abril de 2016

Título: **Nanostructured biomaterials for application in bone healing: processing, characterisation and properties**
Autor: Sara Borrego González
Directores: María Aránzazu Díaz Cuenca
Calificación: Apto "Cum Laude" por Unanimidad
Centro: Universidad Pablo de Olavide
Fecha: 8 de julio de 2016

FORMACIÓN DE GRADUADOS / MASTER DEGREE THESIS

Título: **Vidrios Bioactivos Nanoporosos Ordenados de Zinc para Aplicaciones en Odontología**
Autor: María del Carmen Jiménez Sánchez
Directores: María Aránzazu Díaz Cuenca
Grado: Trabajo Fin de Máster
Año Académico: 2015-2016 (5 julio 2016)

Título: **Descripción óptica de láminas delgadas de perovskita CH3NH3SnxPb1-xI3**
Autor: Pablo Anguita Rodríguez
Directores: Gabriel Lozano Barbero, Hernán Míguez García
Grado: Trabajo Fin de Máster
Año Académico: 2015-2016 (23 septiembre 2016)

Título: **3D nanomembranes for liquid separation**
Autor: José María Román
Directores: Ana Isabel Borrás Martos, Carmen López Santos
Grado: Trabajo Fin de Máster
Año Académico: 2015-2016 (9 septiembre 2016)

Título: **Síntesis y funcionalización de partículas uniformes luminiscentes basadas en CaWO4**
Autor: Francisco Javier García Moscoso
Directores: Nuria Ofelia Nuñez Alvarez
Grado: Trabajo Fin de Máster
Año Académico: 2015-2016 (23 noviembre 2016)

Título: **Síntesis y caracterización de nanopigmentos multifuncionales basados en NaCe(MoO4)2**
Autor: Marcial Fernández Castro
Directores: Manuel Ocaña Jurado, Nuria Ofelia Nuñez Alvarez
Grado: Trabajo Fin de Máster
Año Académico: 2015-2016 (9 diciembre 2016)

Título: **Caracterización y propiedades de recubrimientos biocompatibles nanoestructurados de óxido de titanio sobre aleaciones de Ti de implantes comerciales**
Autor: Juan León Ramos
Directores: Ana Isabel Borrás Martos, Angel Barranco Quero
Grado: Trabajo Fin de Máster
Año Académico: 2015-2016 (9 diciembre 2016)

DOCENCIA / TEACHING

Máster "Láser, Plasma y Tecnología de Superficies"

Tecnología de lámina delgada
Ana Isabel Borrás Martos, Agustín R. González-Elipe
Funcionalización de superficies para aplicaciones mecánicas, protectoras y de bioactividad controladas
María Carmen López Santos, Francisco Yubero Valencia
Nuevos materiales para dispositivos
Victor López Flores, Ana Isabel Borrás Martos
Materiales e Ingeniería de Superficies
Ana Isabel Becerro Nieto
Lugar: Universidad de Córdoba, Universidad Politécnica de Madrid, Instituto de Ciencia de Materiales de Madrid

5th Intensive School on Conservation Science: Ceramic Materials

Layers' microsctructure: influence and utility control for specific applications
Agustín R. González-Elipe
Lugar: Toledo, España

Máster Oficial y Doctorado en Biotecnología Avanzada

Nanotecnología

María Aránzazu Díaz Cuenca
Lugar: Universidad Internacional de Andalucía. Universidad de Málaga

Máster en Física Avanzada
Modelos de sistemas físicos
Gabriel Lozano Barbero
Lugar: Universidad de Córdoba

PREMIOS Y RECONOCIMIENTOS / PRIZES AND ACKNOWLEDGEMENTS

En el año 2016 Miguel Anaya obtuvo:

1) E-MRS Student Award 2016. Mayo
2) SPIE scholarship. Junio 2016.
3) Premio Artículo de Especial Relevancia Universidad de Sevilla 2016 en el área de Ciencias Naturales.
4) Premio Joven a la Cultura Científica del Ayuntamiento de Sevilla 2016.
5) Ganador del Premio cicCartuja-Ebro Foods 2016.

ESTANCIAS Y VISITAS DE PERSONAL DEL ICMS EN OTROS CENTROS
PERSONNEL OF THE ICMS IN OTHER LABORATORIES

Photovoltaic and Optoelectronic Devices Group dirigido por Henry Snaith. Universidad de Oxford

Oxford, Reino Unido	**Miguel Anaya Martín**	3 meses

EQUIPAMIENTO CIENTÍFICO AVAILABLE EQUIPMENT

- Sistema de Magnetron Sputttering para la preparación de láminas delgadas
- Potenciostato y sistema electroquímico
- Sistema de medida de ángulos de contacto (advancing and receding)
- Equipo de crecimiento en vacío de nanofibras y otros materiales nanoestructurados
- Microscopio de efecto túnel (STM)
- Sistema de desorción térmica programada
- Espectrómetro de Fotoemisión (XPS)
- Analizador de potencial Z, tamaño de partícula y pesos moleculares (Malvern, ZS90)
- Bomba híbrida de vacío resistente a químicos VACUUBRAND RC-6

- Software y sondas para monitorización para equipo de liofilización CHRIST EPSILON 2-4 LSC
- Liofilizador de altas prestaciones Epsilon 2-4 (CHRIST)
- Estufa de desecación de 90 litros (RAYPA)
- Cámara Incubadora Opaq + Orbital Maxi (OVAN)
- pH & Ion-metro GLP 22+ (CRISON A)
- Malvern, Modelo Zetasizer Nano: para determinar mediante “Dynamic light scattering” la distribución de tamaños de partícula de sistemas coloidales en suspensión (disolvente acuoso u orgánico) en los rangos 1-3000 nanómetros.
  Este equipo también es adecuado para la evaluación del potencial “Z” de sistemas coloidales en suspensión (disolvente acuoso u orgánico) mediante medidas de Movilidad Electroforética.
- Colorímetro Dr. Lange: para la medida de parámetros de color (x, y, Y/L*a*b*, etc.) de superficies y polvos
- Ellipsómetro espectroscópico Woolan VB-400 con rango de frecuencias entre 300 y 1700 nm. Medida de índices de refracción y coeficiente de extención de capas delgadas y superficies.
- Espectrómetro visible-UV CARY-100. Medidas de coeficiente de absorción con luz normal y polarizada.
- Fluorímetro espectroscópico (HORYBA Jobin Yvon Fluorolog) con accesorio para la determinación de tiempos de vida. Microscopio de fluorescencia (HORYBA Jobin Yvon sigle photon controller: FluoroHub).
- Medidor de ángulos de contacto líquidos. Medidas ángulos de avance y retroceso, así como de energías de adhesión de líquidos sobre superficies (Dataphysics Contact Angle System SCA 20).
- Medida de cuatro puntos de conductividad eléctrica en superficies y láminas delgadas (Fuente de corriente Keithley 617 y voltímetro Keithley 2400).
- Medidas eléctricas en capas delgadas en función de la temperatura y la atmósfera
- Microscopio de Fuerzas atómicas (AFM) para la caracterización de superficies (Cervantes de Nanotec).
- Microscopio de efecto túnel (STM) con posibilidad de trabajar desde nitrógeno líquido hasta 600 ºC (VT-STM de Omicrom).
- Técnicas de caracterización de plasmas: sonda de Langmuir (Plasma Consult single and double sound), espectroscopía de emisión óptica (Avantes 200-900 nm resolución 1 nm) y espectrometría de masas (Hyden=
- Espectrómetro FT-IR con celda DRIFT (Pelkin elmer Spectrum One)
- Sistema de medida de porosidades en capas delgadas.
- Sistema de desorción térmica programada dotado con espectroscopía Auger (VG-8047).
- Espectrómetro de XPS (espectrómetro VSW) con sistema REELS de alta resolución (Kimball Physics EGPS-1022B) y fuente de átomos incorporada (Oxford Scientific Osprey plasma Source).

DISEÑO DE NANOMATERIALES Y MICROESTRUCTURAS
TAILORED NANOMATERIALS AND MICROSTRUCTURE

Personal / Personnel

Profesores de Investigación

Dra. Asunción Fernández Camacho

Catedráticos

Dr. Luis M. Esquivias Fedriani
Dr. Diego Gómez García

Investigadores Científicos

Dr. Juan Carlos Sánchez López

Científicos Titulares

Dra. Rosalía Poyato Galán
Dra. T. Cristina Rojas Ruiz

Investigadores Contratados

Dra. Gisela M. Arzac Di Tomaso
Dra. Vanda C. Fortio Godinho
Dra. Ana María Beltrán Custodio
Dr. Victor Manuel Morales Florez

Becarios Predoctorales

Lda. Mariana Paladini San Martin

Personal Contratado

Ing. Tec. M. Rocío García Gil
Lda. Jennifer López Viejobueno
Ldo. Dirk Hufschmidt
D. Miguel Nieto Redondo

Proyectos de Investigación / Research Projects

Cerámicas nanoestructuradas a base de carburo de boro y nitruro de titanio para aplicaciones estructurales Boron carbide and titanium nitride-based nanostructured ceramics for structural applications

Código/Code: MAT2015-71411-R
Periodo/Period: 01-01-2016 / 31-12-2019
Organismo Financiador/Financial source: Ministerio de Economía y Competitividad
Importe total/Total amount: 157.300 €
Investigador responsable/Research head: Diego Gómez García / Arturo Domínguez Rodríguez
Componentes/Research group: Francisco L. Cumbreras Hernández, Felipe Gutíerrez Mora, Ana Morales Rodríguez

RESUMEN / ABSTRACT

El proyecto tiene como misión la fabricación de forma controlada científicamente de nanocerámicos de carburo de boro y de nitruro de titanio mediante la técnica de chispa de plasma. Se estudiarán las propiedades mecánicas de ambos cerámicos a temperatura ambiente (dureza y tenacidad), así como su plasticidad a alta temperatura (resistencia a la fluencia, deformación a velocidad constante).

Se pretende estudiar la influencia de la microestructura en la respuesta mecánica, así como dilucidar los mecanismos que controlan la plasticidad (particularmente la interacción de dislocaciones con maclas). Los resultados se modelarán analíticamente o mediante simulación a escala mesoscópica (vía modelos de campos de fases).

Boron carbide and titanium nitride are among the most promising ceramic materials nowadays. In the first case, this is due to the outstandng mechanical properties (it is the third hardest material in nature) and its high resistance to chemical attack. In the case of Titanium nitride, its remarkable optical properties and electrical conductivity makes this a potential material for electronic devices. In both cases, sintering is a challenging issue due to the low diffusitivty. In this project, sintering of these materials by spark plasma sintering will be studied and the conditions for nanostructuration will be determined. Preliminary results show that average grain sizes as low as 100 nm can be achieved. In a second stage, plasticity will be studied. A previous model developed by the authors show that twinning is a key ingredient as a driving force of plasticity of boron carbide. The case of titanium nitride is mostly exciting because the stacking fault energy is the lowest ever known and it can make twinnin very favoured. The comparison between these two systems can be a clue about the basic mechanism for hardening in these ceramic materials.

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Desarrollo de catalizadores soportados sobre estructuras porosas para aplicaciones de generación y combustión catalítica de hidrógeno en el contexto de energías renovables Development of supported catalysts on porous structrures for hydrogen generation and catalytic combustión applications in the framework of renewable energies

Código/Code:	CTQ2015-65918-R
Periodo/Period:	01-01-2016 / 31-12-2018
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	177.870 €
Investigador responsable/Research head:	Asunción Fernández Camacho
Componentes/Research group:	Mª Carmen Jiménez de Haro, Vanda Godinho, Gisela Arzac, Dirk Hufschmidt, Rocio García

RESUMEN / ABSTRACT

El agotamiento de combustibles fósiles a corto y medio plazo y los cambios climáticos producidos por el efecto invernadero son algunas de las principales consecuencias del uso extendido de estos combustibles. En este escenario el hidrógeno como vector de transporte y almacenamiento de energía es un candidato muy atractivo en el contexto de un mayor uso de las energías renovables y limpias. En consecuencia se plantean actualmente retos importantes para el desarrollo de tecnologías adecuadas, tanto en la producción de hidrógeno libre de CO2, como en su transporte y almacenamiento seguro, y en su combustión eficiente para producir calor ó electricidad en una pila de combustible. Sobre la base de los proyectos previos del grupo en el estudio de hidruros complejos para almacenamiento de hidrógeno y en el desarrollo de catalizadores y procesos integrados de generación y uso del hidrógeno en aplicaciones portátiles; se abordarán en este proyecto nuevas investigaciones para desarrollar catalizadores novedosos soportados sobre estructuras porosas: membranas y espumas de materiales poliméricos, metálicos y cerámicos de alto interés actual. Los catalizadores se desarrollarán y estudiarán en reacciones seleccionadas de generación y combustión de acuerdo a las siguientes líneas de actuación:

1) Desarrollo de materiales novedosos con alto valor añadido del conjunto soporte-catalizador. Por un lado los soportes porosos basados en membranas de PTFE, espumas metálicas de Ni y espumas cerámicas de SiC. El objetivo es desarrollar los nuevos catalizadores sobre soportes de interés como membranas separadoras, electrolitos, electrodos ó combustores de hidrógeno. Los nuevos catalizadores persiguen la reducción del uso de metales nobles (i.e. bimetálicos Pt-Cu, Ni-Fe) y el desarrollo de nuevos materiales metal-metaloide (carburos, boruros, etc.). Se usarán métodos químicos de impregnación, y muy especialmente la tecnología de deposición de películas delgadas, pulverizacón catódica, que hemos aplicado recientemente con éxito a la fabricación de catalizadores de Co. La metodología abre un campo de investigación de

gran interés al permitirnos el control de la microestructura y/o la composición (i.e. Co, Co-B, Co-C) de los catalizadores a demanda.

2) La caracterización microestructural y química de los nuevos materiales y catalizadores desarrollados en el proyecto. Se trata típicamente de materiales con una microestructura y nanoestructura controlada en donde las modernas técnicas nanoscopicas van a jugar un papel fundamental en la fabricación a medida de estos.

3) Estudio de actividad en tres ensayos catalíticos: i) la generación hidrolítica de hidrógeno, ii) la descomposición fotocatalítica del agua y iii) la combustión catalítica del hidrógeno. Todas ellas reacciones de alto interés en el contexto del uso del hidrógeno como vector de transporte y almacenamiento de energías renovables.

--Sobre la base de los resultados obtenidos en estas líneas de actuación, el proyecto se ha diseñado para alcanzar un conocimiento fundamental y un diseño racional en la nanoescala de catalizadores soportados en sustratos porosos. Las relaciones composición-estructura-propiedades se investigarán usando los ensayos catalíticos y fotocatalíticos acoplados a la microscopía electrónica de alta resolución analítica y otras técnicas espectroscópicas.

The depletion of fossil fuels (in a short and long term) and the global warming derived from greenhouse effect are consequences of the extensive use of these fuels. In this context, hydrogen appears as an attractive, clean and abundant energy carrier in the context of a wider use of clean and removable energies. For the implementation of the “hydrogen economy” many technological challenges regarding hydrogen production (free from CO2), transport, storage (in a safe manner) and combustion (to produce heat or electricity) should be met first. New research will be conducted in this project on the basis of our previous results regarding the study of complex hydrides for hydrogen storage and the development of catalysts and processes for hydrogen generation and use in portable applications. In particular, new catalysts will be developed on porous structures such as polymeric, metallic and ceramic membranes and/or foams with high actual interest. Catalysts will we developed and studied for hydrogen generation and combustion reactions according to the following research lines:

1) Development of new materials (catalysts and supports) with a high added value of the complete system catalyst + support. Porous Ni and SiC foams together with PTFE membranes will be selected as supports for the studies. The main objective is to design new catalysts on technologically interesting supports such as separating membranes, electrolytes, electrodes and/or hydrogen combustors. These new catalysts will be developed following the objective of reducing the amount of noble metals by combining or replacing with another non-noble metals (e.g. Pt-Cu and Ni-Fe) and/or with metalloids (e.g carbides, borides, etc). Wet impregnation methods will be used and special emphasis will be put on the use of the PVD methodology (magnetron sputtering) recently employed in our laboratory for the fabrication of Co thin films with very good results. The latter methodology opens a highly interesting research field because permits to tune microstructure and composition (i.e. Co, Co-B, Co-C) on demand.

2) Characterization of the prepared materials from a microstructural and chemical point of view. Modern nanoscopies will play a key role in the characterization, comprehension and further improvement of these highly nanostructured catalysts.

3) Catalytic studies on the prepared materials will be carried out in three catalytic tests: i) the hydrogen generation through hydrolysis reactions, ii) the photocatalytic water splitting, and iii) the catalytic hydrogen combustion. These reactions are of high interest in the context of the hydrogen economy.

--The interaction of these three research lines as proposed in this project will permit to achieve basic knowledge on the rational design of nanocatalysts supported on porous materials. Structure-composition-activity relationships will be established through catalytic and photo-catalytic studies in combination with characterization techniques based on high resolution analytical TEM and additional spectroscopic techniques.

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Procesado y caracterización microestructural, mecánica y eléctrica de compuestos cerámica-grafeno Processing and microstructural, mechanical and electrical characterization of ceramic-graphene composites

Código/Code:	MAT2015-67889-P
Periodo/Period:	01-01-2016 / 31-12-2018
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	89.177 €
Investigador responsable/Research head:	Angela Gallardo López (UEI). Rosalía Poyato Galán
Componentes/Research group:	Antonio Muñoz Bernabé, Felipe Gutiérrez Mora, Ana Morales Rodríguez

RESUMEN / ABSTRACT

En la actualidad se plantean interesantes expectativas sobre los compuestos cerámica/grafeno, propuestos para aplicaciones en catálisis, almacenamiento y conversión de energía, protección del medio ambiente y biotecnología. Pero aún se requieren importantes esfuerzos para dar respuesta a cuestiones abiertas. Hay que incidir en aspectos como la resistencia a la cizalla de las intercaras cerámica-grafeno -esencial para asegurar la transferencia de carga efectiva sobre las láminas de grafeno-, la distribución homogénea del grafeno en la matriz cerámica y la relación de tamaños entre matriz y refuerzo, para maximizar el aumento de tenacidad y de conductividad eléctrica, así como también explorar sus propiedades mecánicas a alta temperatura.

En este proyecto se plantea un estudio sistemático de compuestos de matrices cerámicas con grafeno, desde la fabricación hasta la caracterización microestructural, mecánica y eléctrica, con el objetivo de mejorar la comprensión de los mecanismos que controlan estas propiedades al incorporar nanoestructuras de grafeno a una matriz cerámica. Se procesarán compuestos de dos matrices cerámicas diferentes, de alúmina y de circona tetragonal dopada con óxido

de itrio (3YTZP), con grafeno mediante técnicas coloidales, prestando especial atención a la dispersión del grafeno en la matriz cerámica, aspecto no exento de dificultades y que es clave para conseguir la mejora de las propiedades. La sinterización se realizará en un horno de descarga de plasma (SPS, spark plasma sintering) de última generación, optimizando las condiciones para conseguir compuestos densos y de tamaño de grano nanométrico. Para el análisis microestructural se utilizarán técnicas como la difracción de rayos X, la espectroscopía Raman, y la microscopía electrónica de barrido y transmisión. Con ellas se evaluarán las fases cristalográficas presentes, el tamaño de grano, la distribución de las nanoestructuras de grafeno, etc.

Desde el punto de vista del diseño de materiales avanzados, es fundamental investigar la relación entre microestructura y propiedades mecánicas y eléctricas. Las propiedades mecánicas a temperatura ambiente (dureza, tenacidad a la fractura y resistencia a flexión) se abordarán mediante indentación y ensayos de flexión, a escalas macro y microscópica. A alta temperatura, se estudiará la deformación plástica de los compuestos cerámica-grafeno mediante ensayos de fluencia en atmósferas controladas. También se estudiará el comportamiento tribológico de los compuestos y se evaluará su conductividad eléctrica, una de las propiedades más interesantes ya que se modifica de forma notoria como resultado de la incorporación del grafeno a estos sistemas cerámicos. La respuesta eléctrica se analizará en un amplio rango de temperaturas, bien mediante espectroscopía de impedancia compleja, bien mediante medidas de conductividad en corriente continua en el caso de los compuestos menos resistivos.

Nowadays, interesting prospects are proposed for ceramic-graphene composites, in application fields such as catalysis, energy storage and conversion, environment protection and biotechnology. A great effort is still required to answer open questions. Issues such as shear resistance of the ceramic-graphene interface essential to obtain an effective load transfer to the graphene sheets-, distribution of graphene in the ceramic matrix -to maximize the reinforcement mechanisms and electrical conductivity- and the high temperature mechanical properties in these composites need special attention.

A systematic study of ceramic matrix graphene composites, including processing and microstructural, mechanical and electrical characterization is proposed in this project, with the aim of improving the comprehension of mechanisms controlling these properties when adding graphene nanostructures to a ceramic matrix.

Both alumina and yttria tetragonal zirconia (3YTZP) graphene composites will be processed by means of colloidal techniques. Special attention will be devoted to the dispersion of graphene in the ceramic matrix which is not a straightforward aspect, but is key to improve mechanical and functional properties. Sintering will be carried out by spark plasma sintering, SPS. Conditions will be optimized in order to obtain fully dense composites with nanometric grain size. Microstructural analysis will be performed by X ray diffraction, Raman spectroscopy, scanning and transmission electronmicroscopy (SEM and TEM). The present crystallographic phases, grain size and distribution of graphene nanostructures will be evaluated.

In order to design advanced materials, it is necessary to study the relationship between microstructure and mechanical or electrical properties. Room temperature mechanical properties (hardness, fracture toughness and flexural resistance) will be characterized by indentation and bending tests at macro and microscopic scales. At high temperature, the plastic behavior of these ceramic-graphene composites will be assessed by creep tests under controlled atmosphere. Tribological behavior of the composites will also be studied to evaluate their resistance to wear. The electrical response will be assessed in a wide range of temperatures by means of complex impedance spectroscopy or by direct current conductivity measurements in the composites with lower resistivity. This is a most interesting property since it can be strongly increased when incorporating graphene to these ceramic systems.

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Recubrimientos para aplicaciones en energía y alta temperatura High temperatura energy application coatings

Código/Code:	MAT2015-65539-P
Periodo/Period:	01-01-2016 / 31-12-2018
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	83.006 €
Investigador responsable/Research head:	Juan Carlos Sánchez López
Componentes/Research group:	Iñigo Braceras Izaguirre (INASMET), Teresa Cristina Rojas Ruiz, Maria Belinda Sigüenza Carballo

RESUMEN / ABSTRACT

La protección de las superficies frente a la temperatura, los fenómenos de oxidación o el desgaste ha logrado un progreso substancial mediante el desarrollo de nuevos materiales y recubrimientos con propiedades mejoradas tales como dureza extrema, baja fricción y tasas de desgaste, elevada resistencia ante la temperatura y la oxidación. Estas mejoras suponen un enorme ahorro de energía y reducción de costes debido a la vida media de los componentes mecánicos sin necesidad de sustitución, así como, a una reducción del impacto medioambiental. Este campo de investigación tiene una profunda repercusión en una gran variedad de sectores industriales (energía, herramientas de mecanizado, automoción, aeronáutico, metalurgia, etc.). El reto para la mayoría de estos procesos de funcionalización superficial residen en un control estricto de la micro y nanoestructura de la superficie y de las intercaras que hagan posible la aparición de nuevas propiedades y aplicaciones que la nanotecnología ofrece.

En este proyecto, se prepararán recubrimientos nanoestructurados para la protección de componentes sometidos a altas temperaturas y ambientes agresivos buscando un comportamiento mejorado. Este objetivo será abordado para tres diferentes aplicaciones que contribuirían a procesos energéticos más eficientes, energías renovables y soluciones para disminuir el impacto medioambiental. Basándonos en el sistema Cr-Al-N, se depositarán diferentes recubrimientos mediante la técnica de pulverización catódica reactiva cambiando la composición química (contenido en metal, incorporación de dopantes tales como Y o Si); microestructura; distribución de fases; arquitectura (multicapa/nanocomposite) o estructuras más complejas (tándem, multicapa en gradiente) sobre los sustratos apropiados dependiendo de la aplicación prevista: a) resistencia a la oxidación a alta temperatura (hasta 1000ºC) para herramientas; b) absorbedores solares selectivos estables térmicamente a medias (300-500ºC) y alta temperatura (>600ºC); resistencia a la corrosión para componentes en turbinas de vapor supercríticos (650ºC/100% vapor).

La investigación sobre los mecanismos de oxidación, transformaciones de fases, modificaciones estructurales, etc. serán objeto de un estudio detallado sobre los sustratos definidos para lograr un conocimiento fundamental sobre los procesos de degradación y los efectos protectores. El establecimiento de correlaciones entre las propiedades iniciales y el comportamiento funcional permitirá una mejor comprensión de los mecanismos de protección y por ende, una optimización de tales sistemas en forma de recubrimientos nanoestructurados para las aplicaciones previstas.

The protection of surfaces from thermal, wear and oxidation phenomena has reached a substantial progress by developing new materials and coatings with improved properties as extreme hardness, low friction and wear rates, increased thermal and oxidation resistance. These improvements suppose a huge energy-saving and cost reduction due to the increased life-time of mechanical components without needs of replacement as well as a reduction in the environmental impact. This field of research has a deep impact in a large variety of industrial sectors (energy, machining tools, automotive, aeronautic, metallurgy, etc.). The challenge for most of these surface functionalization procedures is to get a strict control of the micro and nanostructure of the surface and interfaces that make possible the advent of new properties and applications that nanotechnology concept offers.

In this project, tailored nanostructured coatings for protection of components submitted to high temperature and aggressive environments are prepared seeking for an improved performance. This goal will be explored for three different applications that would contribute to an energy efficiency, renewable energies and solutions to decrease environmental impact. Based on the Cr-Al-N system, different coatings will be prepared by reactive magnetron sputtering technology changing chemical composition (metal content, incorporation of dopants like Y or Si); microstructure; phase distribution; architecture (multilayer/ nanocomposite) or more complex structures (tandem, multilayer gradient) on appropriated substrates depending on the foreseen application: a) oxidation resistance at high temperature (up to 1000ºC) for tool components; b) thermal stable solar selective absorber coating for mid (300-500ºC) and high temperature (>600ºC); c) corrosion resistant coating for supercritical turbine components (650ºC and 100% steam atmosphere).

The investigation of the oxidation mechanisms, phase transformations, structural modifications, etc. will be object of a careful study directly over the defined substrates for these applications to get fundamental knowledge on the degradation phenomena and protective effects. The establishment of the relationships between the initial properties and observed functional performance will enable the better understanding of the protection mechanisms and the optimization of such nanostructured coating systems for the selected application.

c) Studies of coupling a hydrogen generator system with a low cost fuel cell. Typically a continuous reactor for the hydrolysis of NaBH4 with Co-B catalyst for providing H2 at constant flow rate conditions to directly feed a PEM fuel cell of 60 W.

d) Fundamental studies for the development of catalysts and supports for the controlled combustion of hydrogen. It's a new line in the research group based on wet chemical preparation of noble metal nanoparticle catalysts on commercial porous ceramic supports (i.e. SiC). The line also includes the design of a reactor for laboratory-scale study of heat production by controlled combustion of hydrogen.

e) Development of sputtering technology ("magnetron sputtering") for the preparation of catalysts and nano-structures on various substrates for use in the processes developed in the previous sections. The group has extensive experience in this technology to be applied in novel ways in this project leading to a great versatility regarding nanostructure, composition and addition of additives to improve catalytic activity, durability and selectivity of catalysts.

f) Microstructural and chemical characterization of new materials and catalysts developed in the project. We are dealing typically with materials of controlled nanostructure where modern nanoscopic techniques will play a key role in the custom manufacturing of these materials.

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Desarrollo de procesos de combustión catalítica de hidrógeno y estudio de su integración en dispositivos para aplicaciones portátiles Development of processes for the catalytic combustion of hydrogen and study of the integration in devices for portable applications

Código/Code:	P12-TEP-862 (Proyecto de Excelencia)
Periodo/Period:	16-05-2014 / 15-05-2016
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	153.025 €
Investigador responsable/Research head:	Asunción Fernández Camacho
Componentes/Research group:	Julián Martínez, Gisela Arzac, Dirk Hufschmidt, Joaquín Ramírez, M.Carmen Vera, Vanda Godinho, Lionel Cervera, T.Cristina Rojas, Olga Montes, Mariana Paladini, Jaime Caballero-Hernández

RESUMEN / ABSTRACT

El hidrógeno como vector de transporte y almacenamiento de energía es un candidato muy atractivo en el contexto de un mayor uso de las energías renovables y limpias. La producción y el uso de la energía basada en la tecnología del hidrógeno es de especial relevancia en pequeña escala para aplicaciones portátiles (y potencialmente escalable para aplicaciones estacionarias). En el presente proyecto se abordará el estudio del proceso de combustión catalítica o controlada de hidrógeno en los distintos aspectos que puedan conducir a una configuración final integrada con un sistema de generación de H2 en aplicaciones portátiles. Para ello se aprovecharán las sinergias integrando investigadores de dos grupos del PAI: i) Del grupo TEP217, especialistas en almacenamiento y generación de hidrógeno en sistemas basados en hidruros metálicos, hidruros complejos y composites de hidruros reactivos; así como en el uso de catalizadores y aditivos para controlar y mejorar las cinéticas de estos procesos. ii) Del grupo FQM342, especialistas en la obtención de cerámicos porosos de alto interés como soportes de catalizadores en entornos agresivos de combustión. Además la colaboración se completa con la participación de la empresa Abengoa Hidrógeno S.A. que participa en calidad de subcontratada como especialistas en sistemas de producción y almacenamiento de hidrógeno.

En particular se trabajará en este proyecto en las siguientes líneas de actuación:

1.- Desarrollo de catalizadores y soportes para la combustión controlada. Típicamente cerámicas porosas biomórficas de carburo de silicio y catalizadores clásicos tipo metal noble y nuevos catalizadores de bajo coste a desarrollar en el proyecto.

2.- Desarrollo de los reactores necesarios para el estudio de la combustión controlada. Típicamente para flujos de hidrógeno de unos pocos ml/min y para la escala de un generador de H2 ya disponible de 0.5 a 1.5 L/min.

3.- Acoplamiento al sistema de combustión controlada de los sistemas portátiles de generación de hidrógeno que hemos desarrollado en proyectos anteriores.

4.- Aplicación de la tecnología de pulverización catódica de una manera exploratoria en este proyecto para depositar los catalizadores de combustión catalítica en sustratos porosos.

5.- Caracterización microestructural y química de los soportes y catalizadores en la nanoescala para seguir los procedimientos de síntesis y evolución en operación.

Hydrogen is an attractive candidate as a vector for storage and transport of energy in the context of an increased use of renewable and clean energies. The production and use of energy based on hydrogen technology is particularly important for small-scale portable (and potentially scalable for stationary) applications. In this project the process of catalytic (controlled) combustion of hydrogen will be investigated in the various aspects that could lead to a final integrated configuration with a H2 generation system for portable applications. For that the project will take advantage of the synergy of integrating two researcher groups from the PAI: i) The TEP217 group, specialists in storage and generation of hydrogen based on metal hydrides, complex hydrides and hydride composites reactive systems; and in the use of catalysts and additives to control and improve the kinetics of these processes. ii) The FQM342, specialist group for the fabrication of porous ceramics of high interest as catalyst supports for harsh combustion environments. Further collaboration is completed with the participation of the company Abengoa Hidrógeno SA that will be involved as sub-contractor as specialist in systems for the production and storage of hydrogen.

In particular we will work on this project in the following lines:

1.- Development of catalysts and supports for catalytic combustion. Typically porous biomorphic silicon carbide ceramics and classic noble metal catalysts, as well as new low cost catalysts to be developed in the project.

2.- Development of reactors needed for the study of the catalytic combustion. Typically hydrogen flows from a few ml/min to the scale of a H2 generator already available in the range 0.5 to 1.5 L/min.

3.- Coupling the catalytic combustion system with a portable hydrogen generation systems that we have developed in previous projects.

4.- Application of the sputtering technology in an exploratory manner in this project to deposit the catalyst materials for the H2 catalytic combustion on porous substrates.

5.- Microstructural and chemical characterization of the supports and catalysts in the nanoscale to follow the procedures of synthesis and evolution in operation.

Aplicación de técnicas avanzadas de microscopía electrónica para la caracterización de recubrimientos nano-estructurados para aplicaciones en energías limpias Application of advanced electron microscopy techniques to the characterization of nanostructured coatings for clean energy applications

Código/Code:	TAHUB-050. Programa Talent HUB
Periodo/Period:	01-03-2015 / 28-02-2017
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	153.025 €
Investigador responsable/Research head:	Ana María Beltrán Custodio

RESUMEN / ABSTRACT

Este proyecto se centra en la generación y almacenamiento de hidrógeno con el objetivo de producir hidrógeno para energías limpias. Esto sucede durante una reacción exotérmica en la que es necesaria la presencia de un catalizador para que se lleve a cabo en condiciones de seguridad. Los catalizados basados en metales nobles son buenos candidatos para este objetivo (cobalto, cobre…). Aquí, los sistemas completos catalizador-soporte son estudiados. Estos sistemas son crecidos mediante técnicas de pulverización catódica (“magnetron sputtering”). La estructura y la composición son estudiadas a escala nanométrica mediante técnicas avanzadas de microscopía electrónica de transmisión-barrido (STEM), como la microscopía electrónica de alta resolución (HRTEM), imágenes adquiridas en modo campo oscuro con detector de alto ángulo (HAADF), energía dispersiva de rayos X, espectroscopia de pérdida de energía de electrones (EELS), para análisis químico. Además, el uso de la técnica de caracterización tridimensional, tomografía electrónica, aporta un completo conocimiento del sistema analizado. La combinación de técnicas de análisis estructural y de composición, en modo TEM y STEM, nos permite obtener una completa nano-caracterización del sistema. Estos análisis STEM son una herramienta esencial para determinar la relación entre la microestructura, las condiciones de crecimiento y el comportamiento final y las propiedades del sistema, que nos ayudará a mejorarlos y, por tanto, contribuir a la producción de energía limpia.

Este proyecto tiene cuatro objetivos estratégicos.

1. Nano-materiales para aplicaciones en energía limpia. Materiales para la producción, uso y almacenamiento de hidrógeno.
2. Desarrollo de la técnica de magnetron sputtering para la fabricación de nano-estructuras (capas delgadas, recubrimientos y micro-estructuras multicapas).
3. Potenciación de las facilidades LANE (Laboratorio de microscopía del centro ICMSE-CSIC).
4. Uso de técnicas avanzadas de caracterización estructural y de análisis para el estudio a nanoescala de nuevos nano-materiales.

This project is focus on the hydrogen generation and storage with the aim of producing hydrogen for clean and sustainable energies. It happens due to an exothermic reaction where a catalyst is required to do so safety. Catalysts based on noble metals are good candidates for this purpose such as, cobalt, cupper… Here, the complete catalysts systems and different supports are studied. They have been grown by magnetron sputtering technology. The structure and composition are studied, up to nano-scale, by advanced scanning-transmission electron microscopy techniques, (S)TEM, such as high-resolution (HRTEM), high-angle annular dark field (HAADF), energy dispersive X-Ray (EDX), electron energy loss spectroscopy (EELS), for chemical analysis. Furthermore, the use of the three-dimensional characterization technique electron-tomography provides a full understanding of the analysed material. The combination of structural and compositional analytical microscope techniques, in both STEM and TEM mode, allows a full nano-characterization of the systems. The (S)TEM analyses are the essential tool to determine the relationship among the microstructure, the growth conditions and the final behaviour and properties of the systems which will help to improve them and, therefore, to contribute to the production of clean energy.

This project has four main strategic objectives:

1. Nano-materials for sustainable energy applications. Materials for the production, use and storage of Hydrogen.
2. Development of sputtering technology for the fabrication of nanostructures (thin films, coatings and controlled microstructure multilayers).
3. Development of the potential capabilities of the Laboratory for Nanoscopies and Spectroscopies (LANE).
4. Use of advanced structural and analytical techniques for the nano-analysis of new nanomaterials.

Otros Proyectos / Other Projects

Tribological study of functional coatings working under extreme conditions

Código/Code:	201560E013
Periodo/Period:	01-01-2015 / 31-12-2016
Organismo Financiador/Financial source:	CSIC (Intramural)
Importe total/Total amount:	17.780 €
Investigador responsable/Research head:	Juan Carlos Sánchez López
Componentes/Research group:	T.Cristina Rojas, Santiago Domínguez Meister

Sostenibilidad del Laboratorio Avanzado de Nanoscopías y Espectroscopías

Código/Code:	201460E018
Periodo/Period:	01-01-2014 / 31-12-2016
Organismo Financiador/Financial source:	CSIC (Intramural)
Importe total/Total amount:	257.630,26 €
Investigador responsable/Research head:	Asunción Fernández Camacho

Adquisición e instalación de un microscopio electrónico de transmisión

Código/Code: CSIC15-CE-3298
Periodo/Period: 01-01-2016 / 31-12-2017
Organismo Financiador/Financial source: Ministerio de Economía y Competitividad
Importe total/Total amount: 420.180 €
Investigador responsable/Research head: Asunción Fernández Camacho

CONVENIOS Y CONTRATOS / CONTRACTS AND AGREEMENTS

Research on tribological systems. Functional coatings and their characterization

Periodo/Period: 23-10-2014 / 31-12-2018
Organismo Financiador/Financial source: Robert Bosch Gmbh
Importe total/Total amount: 24.490 €
Investigador responsable/Research head: Juan Carlos Sánchez López
Componentes/Research group: Santiago Domínguez Meister

Caracterización microstructural y química de materiales por microscopía electrónica

Periodo/Period: 14-02-2014 / 13-02-2017
Organismo Financiador/Financial source: ABENGOA RESEARCH S.L.
Importe total/Total amount: 15.125 €
Investigador responsable/Research head: Asunción Fernández Camacho
Componentes/Research group: Vanda Fortio Godinho, M. Carmen Jiménez de Haro, Inmaculada Rosa Cejudo

Caracterización microstructural y química de materiales en células CIGSe

Periodo/Period: 13-05-2014 / 12-05-2017
Organismo Financiador/Financial source: ABENGOA Solar New Technologies, S.A.
Importe total/Total amount: 55.660 €
Investigador responsable/Research head: Asunción Fernández Camacho
Componentes/Research group: Vanda Fortio Godinho, M. Carmen Jiménez de Haro, Inmaculada Rosa Cejudo, Olga Montes Amorín

PATENTES / PATENTS

Blanco sólido de gases nobles para reacciones nucleares

Inventores: Jaime Caballero Hernández, Begoña Fernández Martínez, Asunción Fernández Camacho, Francsico Javier Ferrer Fernández, Vanda Cristina Fortio Godinho, Joaquín José Gómez Camacho
Tipo de Patente: Nacional
Solicitud número: 201630711
Fecha Solicitud: 31 mayo 2016
Entidad Titular: Consejo Superior de Investigaciones Científicas y Universidad de Sevilla

COOPERACIÓN INTERNACIONAL Y OTROS INTERNATIONAL COOPERATION AND OTHERS

Nano-Phenomena and Functionality of Modern Carbon-Based Tribo-Coatings

Periodo/Period:	1-06-2015 / 30-06-2018
Código/Code:	CARBITRIB
Entidad Financiadora/Financial source:	Leverhulme Trust (UK)
Investigador responsable/Research head:	Feodor Borodich
Participantes/Participants:	Juan Carlos Sánchez López

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

Investigation of a Pt containing washcoat on SiC foam for hydrogen combustion applications

Fernandez, A; Arzac, GM; Vogt, UF; Hosoglu, F; Borgschulte, A; de Haro, MCJ; Montes, O; Zuttel, A
Applied Catalysis B: Environmental, **180** (2016) 336-343
Enero, 2016 | **DOI:** 10.1016/j.apcatb.2015.06.040

A commercial Pt based washcoat, used for catalytic methane combustion, was studied supported on a commercial SiC foam as catalytic material (Pt/SiC) for catalytic hydrogen combustion (CHC). Structural and chemical characterization was performed using Electron Microscopy, X-Ray Diffraction (XRD) and X-Ray Photoelectron Spectroscopy (XPS). The reaction was monitored following water concentration by Fourier Transform Infrared spectra (FTIR). The FTIR method was compared with H2 detection by Gas Cromatography (GC) and has shown to be adequate to study the kinetics of the CHC reaction in steady state under our experimental conditions (very lean 1% (v/v) H2/air mixtures). The catalyst is composed of 5–20 nm disperse Pt nanoparticles decorating a mixture of high surface area Al2O3 and small amounts of ceria supported on the SiC foam which also contains alumina as binder. The Pt/SiC catalytic material has demonstrated to be active enough to start up the reaction in a few seconds at room temperature. The material has been able to convert at least 18.5 Lhydrogen min−1 gPt−1 at room temperature in conditions of excess of catalyst. The Pt/SiC material was studied after use using XPS and no significant changes on Pt oxidation states were found. The material was characterized from a kinetic point of view. From the conversion-temperature plot a T50(temperature for 50% conversion) of 34 °C was obtained. Activation energy measured in our conditions was 35 ± 1 kJ mol−1.

Colloidal crystals and water: Perspectives on liquid-solid nanoscale phenomena in wet particulate media

Gallego-Gomez, F; Morales-Florez, V; Morales, M; Blanco, A; Lopez, C

Advances in Colloid and Interface Science, **234** (2016) 142-160

Agosto, 2016 | **DOI:** 10.1016/j.cis.2016.05.004

Solid colloidal ensembles inherently contain water adsorbed from the ambient moisture. This water, confined in the porous network formed by the building submicron spheres, greatly affects the ensemble properties. Inversely, one can benefit from such influence on collective features to explore the water behavior in such nanoconfinements. Recently, novel approaches have been developed to investigate in-depth where and how water is placed in the nanometric pores of self-assembled colloidal crystals. Here, we summarize these advances, along with new ones, that are linked to general interfacial water phenomena like adsorption, capillary forces, and flow. Water-dependent structural properties of the colloidal crystal give clues to the interplay between nanoconfined water and solid fine particles that determines the behavior of ensembles. We elaborate on how the knowledge gained on water in colloidal crystals provides new opportunities for multidisciplinary study of interfacial and nanoconfined liquids and their essential role in the physics of utmost important systems such as particulate media.

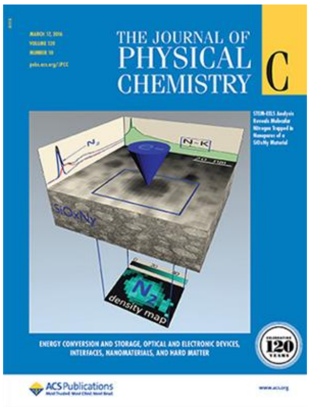
High temperature internal friction in a Ti-46Al-1Mo-0.2Si intermetallic, comparison with creep behavior

Castillo-Rodriguez, M; No, ML; Jimenez, JA; Ruano, OA; Juan, JS

Acta Materialia, **103** (2016) 46-56

Enero, 2016 | **DOI:** 10.1016/j.cej.2014.12.062

Advanced gamma-TiAl based intermetallics Mo-bearing have been developed to obtain the fine-grained microstructure required for superplastic deformation to be used during further processing. In the present work we have studied an alloy of Ti-46.8Al-1Mo-0.2Si (at%) with two different microstructures, ascast material with a coarse grain size above 300 mu m, and the hot extruded material exhibiting a grain size smaller than 20 mu m. We have used a mechanical spectrometer especially developed for high temperature internal friction measurements to study the defect mobility processes taking place at high temperature. The internal friction spectra at different frequencies has been studied and analyzed up to 1360 K in order to characterize the relaxation processes appearing inthis temperature range. A relaxation peak, with a maximum in between 900 K and 1080 K, depending on the oscillating frequency, has been attributed to Ti-atoms diffusion by the stress-induced reorientation of Al-V-Ti-Al elastic dipoles. The high temperature background in both microstructural states, as-cast and extruded, has been analyzed, measuring the apparent activation parameters, in particular the apparent energies of E-cast(IF) = 4.4 +/- 0.05 eV and E-ext(IF) = 4.75 +/- 0.05 eV respectively. These results have been compared to those obtained on the same materials by creep deformation. We may conclude that the activation parameters obtained by internal friction analysis, are consistent with the ones measured by creep. Furthermore, the analysis of the high temperaturebackground allows establish the difference on creep resistance for both microstructural states.



Nitrogen Nanobubbles in a-SiOxNy Coatings: Evaluation of Its Physical Properties and Chemical Bonding State by Spatially Resolved Electron Energy-Loss Spectroscopy

Lacroix, B.; Godinho, V.; Fernández, A.
Journal of Physical Chemistry C, **120** (2016) 5651-5658
Marzo, 2016 | **DOI:** 10.1021/acs.jpcc.5b09036

Nanoporous silicon-based materials with closed porosity filled with the sputtering gas have been recently developed by magnetron sputtering. In this work the physical properties (density and pressure) of molecular nitrogen inside closed pores in a SiOxNy coating are investigated for the first time using spatially resolved electron energy-loss spectroscopy (EELS) in a scanning transmission electron microscope. The paper offers a detailed methodology to record and process multiple EELS spectrum images (SIs) acquired at different energy ranges and with different dwell times. An adequate extraction and quantification of the N–K edge contribution due to the molecular nitrogen inside nanopores is demonstrated. Core-loss intensity and N chemical bond state were evaluated to retrieve 2D maps revealing the stable high density of molecular nitrogen (from 40 to 70 at./nm3) in nanopores of different size (20–11 nm). This work provides new insights into the quantification of molecular N2 trapped in porous nitride matrices that could also be applied to other systems.

Determination of the Anisotropic Elastic Properties of Rocksalt Ge2Sb2Te5 by XRD, Residual Stress, and DFT

Cecchini, R; Kohary, K; Fernandez, A; Cabibbo, M; Marmier, A
Journal of Physical Chemistry C, **120** (2016) 5624-5629
Marzo, 2016 | **DOI:** 10.1021/acs.jpcc.5b09867

The chalcogenide material Ge2Sb2Te5 is the prototype phase-change material, with widespread applications for optical media and random access memory. However, the full set of its independent elastic properties has not yet been published. In this study, we determine the elastic constants of the rocksalt Ge2Sb2Te5, experimentally by X-ray diffraction (XRD) and residual stress and computationally by density functional theory (DFT). The stiffnesses (XRD-stress/DFT) in GPa are C-11 = 41/58, C-12 = 7/8, and C-44 = 8/12, and the Zener ratio is 0.46/0.48. These values are important to understand the effect of elastic distortions and nonmelting processes on the performances of increasingly small phase change data bits.

Enhanced carbon nanotube dispersion in 3YTZP/SWNTs composites and its effect on room temperature mechanical and electrical properties

Gallardo-Lopez, A; Morales-Rodriguez, A; Vega-Padillo, J; Poyato, R; Munoz, A; Dominguez-Rodriguez, A
Journal of Alloys and Compounds, **682** (2016) 70-79
Octubre, 2016 | **DOI:** 10.1016/j.jallcom.2016.04.262

In this work, several modifications of the colloidal processing technique and spark plasma sintering (SPS) to prepare yttria tetragonal zirconia composites (YTZP) with single walled carbon nanotubes (SWNT) have been tested with the aim of eliminating SWNT agglomerates. These modifications include high versus low energy ultrasonic agitation during colloidal processing, lyophilization of the 3YTZP/SWNT slurry and electrical insulation during sintering of the composites. Semi-quantitative microstructural characterization of the carbon nanotube distribution in the sintered composites showed that high energy ultrasonic agitation reduces drastically agglomerate size. Lyophilization of the mixed suspensions avoids SWNT bundle size growth. Combination of both produces an enhanced carbon nanotube network distribution along the grain boundaries (GB) due to the absence of carbon nanotube agglomerates and to a limited SWNT bundle size. This results in an increase of the real SWNT content in the GBs up to nominal SWNT content and therefore an enhanced SWNT efficiency in the composites. The agglomerate-free highly-dispersed composites exhibit a decrease in density together with grain size refinement, a decrease in room temperature hardness, an increase in flexural strength and a most significant increase in room temperature electrical conductivity. Improved SWNT distribution also lowers electrical percolation threshold to a very low level in SWNT ceramic composites, <1 vol% SWNT.

Monolithic supports based on biomorphic SiC for the catalytic combustion of hydrogen

Arzac, G. M.; Ramirez-Rico, J.; Gutierrez-Pardo, A.; Jimenez de Haro, M. C.; Hufschmidt, D.; Martinez-Fernandez, J.; Fernandez, A.
RSC Advances, **6** (2016) 66373-66384
Septiembre, 2016 | **DOI:** 10.1039/c6ra09127j

Catalytic hydrogen combustion was studied with H-2/air mixtures in conditions that simulate the H-2 concentration of the exhaust gases from fuel cells (3-4% v/v H-2 in air). Pt-impregnated monoliths based on porous biomorphic SiC (bio-SiC) substrates were employed for the first time for this reaction. Capillary forces were exploited for the incipient impregnation of supports with H2PtCl6 solutions. Freeze drying permitted us to obtain a homogeneous distribution of the active phase reducing accumulation at the monolith's outer shell. The supports and catalysts were characterized from a structural and thermal point of view. Catalytic tests were performed in a homemade reactor fed with up to 1000 ml min(-1) H-2/air mixtures and a diffusional regime (non-isothermal) was achieved in the selected conditions. Catalyst loading was tested in the range of 0.25-1.5 wt% Pt and 100% conversion was achieved in all cases. Temperatures were recorded at different points of the monoliths during the reaction showing anisotropic thermal behavior for selected bio-SiC substrates. These effects are of interest for heat management applications and were explained in correlation with thermal conductivity measurements performed on the supports. Pt-impregnated monoliths were also tested in less than 100% conversion conditions (1% v/v H-2 in air) and in powder form in kinetic conditions for comparative purposes.

Timing of calcium nitrate addition affects morphology, dispersity and composition of bioactive glass nanoparticles

Zheng, K; Taccardi, N; Beltran, AM; Sui, BY; Zhou, T; Marthala, VRR; Hartmann, M; Boccaccini, AR
RSC Advances, **6** (2016) 95101-95111
Septiembre, 2016 | **DOI:** 10.1039/C6RA05548F

Bioactive glass nanoparticles (BGN) are promising materials for a number of biomedical applications. Many parameters related to the synthesis of BGN using sol–gel methods can affect their characteristics. In this study, the influence of timing of calcium nitrate (calcium precursor) addition during processing on BGN characteristics was investigated. The results showed that the addition timing could affect the morphology, dispersity and composition of BGN. With delayed addition of calcium nitrate, larger, more regular and better dispersed BGN could be synthesized while the gap between nominal and actual compositions of BGN was widened. However, the addition timing had no significant influence on structural characteristics, as BGN with different addition-timing of calcium nitrate exhibited similar infrared spectra and amorphous nature. The results also suggested that monodispersed BGN could be synthesized by carefully controlling the addition of calcium nitrate. The synthesized monodispersed BGN could release Si and Ca ions continuously for up to at least 14 days. They also showed in vitro bioactivity and non-cytotoxicity towards rat bone marrow-derived mesenchymal stem cells (rBMSCs). In conclusion, the timing of calcium precursor addition is an essential parameter to be considered when producing BGN which should exhibit monodisperse characteristics for biomedical applications.

Tailor-made preparation of Co-C, Co-B, and Co catalytic thin films using magnetron sputtering: insights into structure-composition and activation effects for catalyzed NaBH4 hydrolysis

Paladini, M; Godinho, V; Arzac, GM; de Haro, MCJ; Beltran, AM; Fernandez, A
RSC Advances, **6** (2016) 108611-108620
Noviembre, 2016 | **DOI:** 10.1039/c6ra23171c

The magnetron sputtering (MS) methodology is a powerful tool for tailor-made fabrication of Co-based thin film catalysts with controlled microstructures and compositions for sodium borohydride (SBH) hydrolysis. In particular, Co-C catalysts were tested in this reaction and compared to Co-B and Co catalyst coatings. The microstructural and chemical analyses by X-ray diffraction (XRD), scanning and transmission electron microscopy (SEM and TEM), Rutherford back scattering (RBS) and X-ray photoelectron spectroscopy (XPS) were used to characterize a complete library of thin film catalysts. Pure Co materials were characterized by their nanocrystalline microstructure, and grain refinement was achieved via an increase in the deposition pressure. The incorporation of boron or carbon via co-deposition results in amorphization and dispersion of the active metallic Co phase. The composition can be tuned while keeping a controlled microstructure, and a comparison of activity at 25 degrees C was performed on catalysts deposited on Ni foam substrates. A comparison of the initial activities showed that the Co-B samples were more active than the Co-C samples because of electronic effects. However, a strong activation was found for the Co-C catalysts after the first use. This effect was dependent upon the incorporation of cobalt boride (CoxB) species on the catalysts' surface, as shown by XPS. After the first several uses, the activity of the Co-C samples (values up to 2495 mL min(-1) g(catalyst)(-1)) were as high as that of fresh Co-B, and the surface composition of both the catalysts was similar. This activation was not observed for the pure Co and was very weak for the Co-B catalysts. The use of polymeric (PTFE) substrates (flexible membranes) illustrated the versatility of the methodology to obtain catalytic membranes and allowed for a TEM microstructural analysis at the nanoscale. Catalytic activities at 60 degrees C were as high as 16.7 and 20 L min(-1) g(Co)(-1) for the Co-C and Co-B membranes, respectively.

We determined the optimized conditions to increase the catalytic activity of Co-based coatings prepared via magnetron sputtering.

Electrical properties of reduced 3YTZP ceramics consolidated by spark plasma sintering

Poyato, R; Macias-Delgadoa, J; Garcia-Valenzuela, A; Gonzalez-Romero, RL; Munoz, A; Dominguez-Rodriguez, A

Ceramics International, **42** (2016) 6713-6719

Mayo, 2016 | **DOI:** 10.1016/j.ceramint.2016.01.040

3 mol% Yttria doped zirconia ceramics were consolidated by spark plasma sintering (SPS) at two sintering temperatures with the aim of achieving two different reduction levels. Microstructural characterization of the ceramics was performed by scanning electron microscopy (SEM). Electrical properties were investigated by means of impedance spectroscopy from room temperature up to 500 degrees C. The two ceramics presented a remarkably different electrical behavior. The effect of the extra electrons introduced by reduction during SPS on both the bulk and the grain boundary conductivity was analyzed and discussed.

Flue gas adsorption by single-wall carbon nanotubes: A Monte Carlo study

Romero-Hermida, MI; Romero-Enrique, JM; Morales-Florez, V; Esquivias, L

Journal of Chemical Physics, **145** (2016) 074701

Agosto, 2016 | **DOI:** 10.1063/1.4961023

Adsorption of flue gases by single-wall carbon nanotubes (SWCNT) has been studied by means ofMonte Carlo simulations. The flue gas is modeled as a ternary mixture of N2, CO2, and O2, emulating realistic compositions of the emissions from power plants. The adsorbed flue gas is in equilibrium with a bulk gas characterized by temperature T, pressure p, and mixture composition. We have considered different SWCNTs with different chiralities and diameters in a range between 7 and 20 Å. Our results show that the CO2 adsorption properties depend mainly on the bulk flue gas thermodynamic conditions and the SWCNT diameter. Narrow SWCNTs with diameter around 7 Å show high CO2 adsorptioncapacity and selectivity, but they decrease abruptly as the SWCNT diameter is increased. For wideSWCNT, CO2 adsorption capacity and selectivity, much smaller in value than for the narrow case, decrease mildly with the SWCNT diameter. In the intermediate range of SWCNT diameters, the CO2adsorption properties may show a peculiar behavior, which depend strongly on the bulk flue gas conditions. Thus, for high bulk CO2 concentrations and low temperatures, the CO2 adsorption capacity remains high in a wide range of SWCNT diameters, although the corresponding selectivity is moderate. We correlate these findings with the microscopic structure of the adsorbed gas inside the SWCNTs.

High-Temperature Deformation Mechanisms in Monolithic 3YTZP and 3YTZP Containing Single-Walled Carbon Nanotubes

Castillo-Rodriguez, M; Munoz, A; Dominguez-Rodriguez, A

Journal of The American Ceramic Society, **99** (2016) 286-292

Enero, 2016 | **DOI:** 10.1111/jace.13974

Monolithic 3YTZP and 3YTZP containing 2.5 vol% of single-walled carbon nanotubes (SWCNT) were fabricated by Spark Plasma Sintering (SPS) at 1250 degrees C. Microstructural characterization of the as-fabricated 3YTZP/SWCNTs composite shows a homogeneous CNTs dispersion throughout the ceramic matrix. The specimens have been crept at temperatures between 1100 degrees C and 1200 degrees C in order to investigate the influence of the SWCNTs addition on high-temperature deformation mechanisms in zirconia. Slightly higher stress exponent values are found for 3YTZP/SWCNTs nanocomposites (n similar to 2.5) compared to monolithic 3YTZP (n similar to 2.0). However, the activation energy in 3YTZP (Q=715 +/- 60kJ/mol) experiences a reduction of about 25% by the addition of 2.5vol% of SWCNTs (Q=540 +/- 40kJ/mol). Scanning electron microscopy studies indicate that there is no microstructural evolution in crept specimens, and Raman spectroscopy measurements show that SWCNTs preserved their integrity during the creep tests. All these results seem to indicate that the high-temperature deformation mechanism is grain-boundary sliding (GBS) accommodated by grain-boundary diffusion, which is influenced by yttrium segregation and the presence of SWCNTs at the grain boundary.

Structure, electrochemical properties and functionalization of amorphous CN films deposited by femtosecond pulsed laser ablation

Maddi, C; Bourquard, F; Tite, T; Loir, AS; Donnet, C; Garrelie, F; Barnier, V; Wolski, K; Fortgang, P; Zehani, N; Braiek, M; Lagarde, F; Chaix, C; Jaffrezic-Renault, N; Rojas, TC; Sanchez-Lopez, JC
Diamond and Related Materials, **65** (2016) 17-25
Mayo, 2016 | **DOI:** 10.1016/j.diamond.2016.01.001

Amorphous carbon nitride (a-C:N) material has attracted much attention in research and development Recently, it has become a more promising electrode material than conventional carbon based electrodes in electrochemical and biosensor applications. Nitrogen containing amorphous carbon (a-C:N) thin films have been synthesized by femtosecond pulsed laser deposition (fs-PLD) coupled with plasma assistance through Direct Current (DC) bias power supply. During the deposition process, various nitrogen pressures (0 to 10 Pa) and DC bias (0 to -350 V) were used in order to explore a wide range of nitrogen content into the films. The structure and chemical composition of the films have been studied by using Raman spectroscopy, electron energy-loss spectroscopy (EELS) and high-resolution transmission electron microscopy (HRTEM). Increasing the nitrogen pressure or adding a DC bias induced an increase of the N content, up to 21 at%. Nitrogen content increase induces a higher sp(2) character of the film. However DC bias has been found to increase the film structural disorder, which was detrimental to the electrochemical properties. Indeed the electrochemical measurements, investigated by cyclic voltammetry (CV), demonstrated that a-C:N film with moderate nitrogen content (10 at.%) exhibited the best behavior, in terms of reversibility and electron transfer kinetics. Electrochemical grafting from diazonium salts was successfully achieved on this film, with a surface coverage of covalently bonded molecules close to the dense packed monolayer of ferrocene molecules. Such a film may be a promising electrode material in electrochemical detection of electroactive pollutants on bare film, and of biopathogen molecules after surface grafting of the specific affinity receptor.

Disorder-order phase transformation in a fluorite-related oxide thin film: In-situ X-ray diffraction and modelling of the residual stress effects

Gaboriaud, RJ; Paumier, F; Lacroix, B
Thin Solid Films, **601** (2016) 84-88
Febrero, 2016 | **DOI:** 10.1016/j.tsf.2015.08.030

This work is focused on the transformation of the disordered fluorite cubic-F phase to the ordered cubic-C bixbyite phase, induced by isothermal annealing as a function of the residual stresses resulting from different concentrations of microstructural defects in the yttrium oxide, Y2O3.

This transformation was studied using in-situ X-ray diffraction and was modelled using Kolmogorov-Johnson-Mehl-Avrami (KJMA) analysis. The degree of the disorder of the oxygen network was associated with the residual stress, which was a key parameter for the stability and the kinetics of the transition of the different phases that were present in the thin oxide film. When the degree of disorder/residual stress level is high, this transition, which occurs at a rather low temperature (300 degrees C), is interpreted as a transformation of phases that occurs by a complete recrystallization via the nucleation and growth of a new cubic-C structure. Using the KJMA model, we determined the activation energy of the transformation process, which indicates that this transition occurs via a one-dimensional diffusion process. Thus, we present the analysis and modelling of the stress state. When the disorder/residual stress level was low, a transition to the quasi-perfect ordered cubic-C structure of the yttrium oxide appeared at a rather high temperature (800 degrees C), which is interpreted as a classic recovery mechanism of the cubic-C structure.

Effects of electronic and nuclear stopping power on disorder induced in GaN under swift heavy ion irradiation

Moisy, F; Sall, M; Grygiel, C; Balanzat, E; Boisserie, M; Lacroix, B; Simon, P; Monnet, I

Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms, **381** (2016) 39-44

Agosto, 2016 | **DOI:** 10.1016/j.nimb.2016.05.024

Wurtzite GaN epilayers, grown on the c-plane of sapphire substrate, have been irradiated with swift heavy ions at different energies and fluences, and thereafter studied by Raman scattering spectroscopy, UV–visible spectroscopy and transmission electron microscopy. Raman spectra show strong structural modifications in the GaN layer. Indeed, in addition to the broadening of the allowed modes, a large continuum and three new modes at approximately 200 cm−1, 300 cm−1 and 670 cm−1 appear after irradiation attributed to disorder-activated Raman scattering. In this case, spectra are driven by the phonon density of states of the material due to the loss of translation symmetry of the lattice induced by defects. It was shown qualitatively that both electronic excitations and elastic collisions play an important role in the disorder induced by irradiation. UV–visible spectra reveal an absorption band at 2.8 eV which is linked to the new mode at 300 cm−1observed in irradiated Raman spectra and comes from Ga-vacancies. These color centers are produced by elastic collisions (without any visible effect of electronic excitations).

Obtention of Li3xLa2/3−xTiO3 ceramics from amorphous nanopowders by spark plasma sintering

Leyet, Y.; Guerrero, F.; Anglada-Rivera, J.; Martinez, I.; Amorin, H.; Romaguera-Barcelay, Y.; Poyato, R.; Gallardo-Lopez, A.

Ferroelectrics, **498** (2016) 62-66

Mayo, 2016 | **DOI:** 10.1080/00150193.2016.1167538

In this work, Li3xLa2/3-xTiO3 powder with nominal lithium content (x = 0.08) was synthesized by mechano synthesis method. Spark plasma sintering (SPS) was employed to prepare lithium lanthanum titanium oxide solid-state ceramic. The techniques of X-ray diffraction, high resolution scanning electron microscopy, and Raman spectroscopy were used to characterize the composition and microstructure of samples. The results showed that fine-grained ceramics with relative density of 95.5% were obtained by sintering the oxide powders at 1100 degrees C for only 5 min.

Characterization and Validation of a-Si Magnetron-Sputtered Thin Films as Solid He Targets with High Stability for Nuclear Reactions

Godinho, V; Ferrer, FJ; Fernandez, B; Caballero-Hernandez, J; Gomez-Camacho, J; Fernandez, A
ACS Omega, **1** (2016) 1229-1238
Diciembre, 2016 | **DOI:** 10.1021/acsomega.6b00270

In this work, we present our magnetron sputtering based methodology to produce amorphous silicon coatings with closed porosity, as a strategy to fabricate solid helium targets, in the form of supported or self-supported thin films, for nuclear reactions. We show how by changing the He working pressure it is possible to obtain highly porous homogeneous structures incorporating different He amounts. These porous coatings (a-Si: He) are very reproducible from run to run, and the high He amount incorporated makes them excellent candidates for solid He targets. The possibility of producing self-supported films is illustrated here, and its potential use in inverse kinematics experiments with radioactive beams is shown through the dispersion in forward geometry of a stable Li-6 beam. Also the elastic scattering cross-sections for proton from helium were determined using an a-Si: He coating. The results agree well with the ones reported in the literature. These two examples validate our coatings as good candidates to be used as solid He targets in nuclear reactions. The stability of He inside the coatings, fundamental for its use as solid He targets, was investigated, both over time and after irradiation. The coatings proved to be very stable, and the amount of He inside the pores remains unaltered at least 2 years after deposition and after high irradiation fluence (5 x 10(17) particles/cm(2); with a dose rate of 5 x 10(12) particles/(cm(2) s)).

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

10th International Symposium "Hydrogen & Energy"

21 – 26 febrero [Sendai, Japón]

Pt-Cu Catalytic Coatings for Hydrogen combustion: Nanostructure and dealloying effects
V. Godinho, G.M. Arzac, A.M. Beltran, M.C. Jimenez de Haro, F. Giarratano, A. Fernández
Comunicación oral

EMN Meeting on Mesoporous Materials 2016. Energy Materials Nanotechnology

14 – 18 mayo [San Sebastian, España]

Closed porous coatings fabricated by magnetron sputtering with Helium as process gas
V. Godinho, A. Fernandez
Conferencia invitada

The 16th European Microscopy Congress 2016

28 agosto – 2 septiembre [Lyon, Francia]

Nanoscale characterization of Co and Co-B catalytyc coatings before and after catalytic tests for the sodium borohydride hydrolysis
A.M Beltran, M. Paladini, V. Godinho, G.M. Arzac, M. C. Jimenez de Haro, A. Fernandez
Poster

The first CARBTRIB Workshop on "Nano-phenomena and Functionality of modern Carbon-Based Tribo-Coatings"

6 – 9 septiembre [Cardiff, Wales, Reino Unido]

Carbon-based coatings: their specific tribological properties and multifunctionality
J.C. Sanchez-Lopez
Conferencia invitada

Comparative tribological performance of TiC/a-C(:H) nanocomposite coatings prepared by DC and HiPIMS
J.C. Sanchez-Lopez
Comunicación oral

11th International Conference on Surface Coatings and Nanostructured Materials NANOSMAT 2016

6 – 9 septiembre [Aveiro, Portugal]

Determination of the thickness of the embedding phase in crystalline/amorphous nanocomposites
J.C. Sanchez-Lopez, D. Martinez-Martinez
Comunicación oral

15th European Inter-Regional Conference on Ceramics CIEC15

5 – 7 septiembre [Villeurbanne, Francia]

Effect of processing on microstructure and room temperature mechanical and electrical properties of 3YTZPl2.5 volo/o MWNT ceramic nanocomposites

R. Poyato, A. Gallardo-López, A. Morares-Rodríguez, F. Gutiérrez-Mora, A. Muñoz, A. Domínguez-Rodríguez
Poster

Processing and characterization of zirconia (3Y-TZP) composites with graphene nanoplatelets
A. Gallardo-López, l. Márquez-Abril, A. Morales-Rodríguez, A. Muñoz, R. Poyato
Poster

Producing conductive ceramics for electro discharge machining
R. Poyato, J. Guzmán-Mínguez, A. Morales-Rodríguez, A. Muñoz, A. Gallardo-López
Poster

International Workshop on Graphene/Ceramic Composites (WGCC)

28 – 30 septiembre [Cuenca, España]

Electrical percolation in 3YTZP/graphene nanoplatelets composites
A. Gallardo-López, I. Márquez-Abril, A. Morales-Rodríguez, A. Muñoz, R. Poyato
Poster

Graphene nanosheets/alumina composites: processing and characterization
J. Guzmán-Mínguez, R. Poyato, A. Morales-Rodríguez, A. Muñoz, A. Gallardo-López
Poster

Hardness and fracture toughness of reinforced alumina composites sintered by SPS
R. Cano-Crespo, B. Malmal-Moshtaghioun, A. Rincón, R. Moreno, D. Gómez-García, A. Domínguez-Rodríguez
Poster

Tribological behavior of graphene nanoplatelet reinforced 3YTZP
F. Gutiérrez-Mora, I. Márquez-Abril, A. Morales-Rodríguez, A. Muñoz, R. Poyato, A. Gallardo-López
Poster

European Conference on Nanofilms ECNF 2016

19 – 21 octubre [Bilbao, España]

Magnetron sputtered Co, Co-C, Co-B thin films as catalysts for hydrogen generation applications. Durability and deactivation mechanism
M. Paladini, G.M. Arzac, V. Godinho, A. Fernandez, M.C. Jimenez de Haro, D. Hufschmidt, A.M. Beltrán
Comunicación oral

Magnetron sputtering as a versatile tool to fabricate catalytic coatings on porous substrates for renewable hydrogen technology

A. Fernandez, M. Paladini, V. Godinho, G.M. Arzac, D. Hufschmidt, M.C. Jimenez de Haro, A. Beltran, O. Montes
Comunicación oral

CONGRESOS Y REUNIONES NACIONALES / NATIONAL CONGRESSES AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

LV Congreso de la Sociedad Española de Cerámica y Vidrio (SECV) Y I Congreso Bienal de la SECV

13 – 15 julio [Barcelona, España]

Ánodos para pilas cerámicas de combustible: Sr0.9La0.1Ti1-xFexO3 obtenido por vía mecanoquímica
M.J. Sayagués, F.J. García, R. Poyato, F.J. Gotor
Poster

Comparación del comportamiento tribológico en compuestos de circona con distintas nanoestructuras de carbono
F. Gutiérrez Mora, R. Poyato, A. Gallardo-López, A. Morales-Rodríguez, A. Muñoz, A. Domínguez-Rodríguez
Poster

Microstructural features of 3YTZP ceramic composites with different carbon nanostructures
A. Gallardo-López, A. Morales Rodríguez, A. Muñoz Bernabé, R. Poyato
Poster

Monolitos basados en SiC biomórfico como soportes para la reacción de combustión catalítica del hidrógeno
G. M. Arzac, J. Ramírez-Rico, A. Gutiérrez Pardo, M.C. Jiménez de Haro, D. Hufschmidt, J. Martínez-Fernández, A. Fernández
Comunicación oral

Formacion / Training

Tesis Doctorales/ Doctor Degree Thesis

Título: **Síntesis, estructura y caracterización en entornos biológicos de nanopartículas de Au y heterodímeros Pt-Au**
Autor: Carlos Andrés García Negrete
Directores: María Asunción Fernández Camacho
Calificación: Sobresaliente “Cum Laude”
Centro: Universidad de Sevilla
Fecha: 4 de febrero de 2016

Título: **Control of microstructure in porous silicon coatings with closed porosity for functional applications**
Autor: Jaime Caballero Hernández
Directores: Vanda Cristina Fortio Godinho, María Asunción Fernández Camacho
Calificación: Sobresaliente “Cum Laude”
Centro: Universidad de Sevilla
Fecha: 20 de abril de 2016

Título: **Recubrimientos protectors nanoestructurados preparados por magnetron sputtering**
Autor: Santiago Domínguez Meister
Directores: Teresa Rojas Ruiz, Juan Carlos Sánchez López
Calificación: Sobresaliente “Cum Laude”
Centro: Universidad de Sevilla
Fecha: 17 de noviembre de 2016

Formación de Graduados / Master Degree Thesis

Título: **Obtención de cerámicas conductoras propuestas para su electromecanizado**
Autor: Jesús Guzmán Mínguez
Directoras: Dra. Rosalía Poyato Galán
Grado: Trabajo Fin de Master
Año Académico: 2015-2016 (29 de Junio 2016)

DOCENCIA / TEACHING

Máster "Láser, Plasma y Tecnología de Superficies"

Técnicas de caracterización de superficies y láminas delgadas

Cristina Rojas Ruiz

Funcionalización de superficies para aplicaciones mecánicas, protectoras y de bioactividad controladas

Juan Carlos Sánchez López

Lugar: Universidad de Córdoba, Universidad Politécnica de Madrid, Instituto de Ciencia de Materiales de Madrid

EQUIPAMIENTO CIENTÍFICO AVAILABLE EQUIPMENT

Laboratorio de síntesis y catálisis

Material básico de laboratorio químico: PHmetro, agitadores, calefactores, estufa de secado a vacío, centrífuga.

Reactores y material de vidrio convencional para síntesis de nanopartículas y catalizadores por via química.

Rotavapor, sistemas de filtrado.

Cámara seca MBRAUN.

Dos campanas extractoras.

Reactores catalíticos de lecho fijo para catálisis heterogenea solido-gas.

Reactores catalíticos para catálisis heterogenea sólido-líquido.

Sistemas controladores de flujo másico (gases y líquidos evaporados), bombas peristálticas para líquidos.

Tres cromatógrafos de gases, columnas y detectors FID y TCD.

Buretas automáticas para medición de gases.

Cámaras de deposición de recubrimientos y películas delgadas y tratamientos con plasma.

Sistema de tratamiento con plasmas Diener.

Tres cámaras de deposición por la técnica de pulverización catódica (magnetrón sputtering). Con una dotación total de 7 cabezas magnetrón, 2 fuentes DC, 2 fuentes RF y 1 fuente pulsada, portamuestras girables, calentables y "biased".

Una cámara de deposición por la técnica de pulverización catódica con fuente HIPIMS.

Equipamiento para microscopía electrónica

Sistema de preparación de muestras en película delgada TXP de Leica.

Pulidora, trípode y microscopio óptico.

TEM de 300kV Tecnai F30 dotado de modo STEM, detector HAADF, analizador EDX Oxford Max80 y filtro de energías GIF Quantum.

Propiedades mecánicas de superficies y recubrimientos

Tribómetro CSM (movimiento lineal y rotativo) para evaluación de coeficientes de fricción y desgaste.

Tribómetro de alta temperatura (hasta 800ºC) –Microtest

Calotest para medida de espesores y evaluación del desgaste.
Equipo de rayado (Scratch-test) hasta 200N (Tribotechnic).
Perfilómetro-rugosímetro (Mahr) de tipo táctil y resolución vertical nanométrica.

Propiedades eléctricas de materiales

Sistema de medida de espectroscopía de impedancia compleja, formado por un impedancímetro Agilent modelo 4294A, un horno Hobersal ST115020, y una celda de medida hermética para la realización de medidas en atmósfera.

UNIDAD EXTERNA DE INVESTIGACIÓN: FÍSICA DE MATERIALES

EXTERNAL UNIT: PHYSICS OF MATERIALS

PERSONAL / PERSONNEL

Catedrático

- Dr. Alejandro Conde Amiano
- Dra. Clara F. Conde Amiano
- Dr. Alberto Criado Vega
- Dr. Arturo Domínguez Rodríguez
- Dr. Victorino Franco García
- Dr. Antonio Muñoz Bernabé

Profesor Titular

- Dr. Javier S. Blázquez Gámez
- Dra. Josefa María Borrego Moro
- Dra. María Dolores Estrada de Oya
- Dra. M. del Carmen Gallardo Cruz
- Dra. Angela Gallardo López
- Dr. Felipe Gutiérrez Mora
- Dr. José María Martín Olalla
- Dra. Ana Morales Rodríguez
- Dr. Francisco Javier Romero Landa

Investigador Contratado

- Dr. Jhon J. Ipus Bados

Becario Predoctoral

- Ldo. Luis M. Moreno Ramírez

Proyectos de Investigación / Research Projects

Modelado y Control de la Histéresis en Materiales Magnetocalóricos para Refrigeración y Conversión de Energía TT

Código/Code:	MAT2016-77265-R
Periodo/Period:	30-12-2016 / 29-12-2019
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	##### €
Investigador responsable/Research head:	Victorino Franco García / Javier Sebastián Blázquez Gámez
Componentes/Research group:	Josefa María Borrego Moro, Alejandro Conde Amiano, Clara Francisca Conde Amiano, Jhon J. Ipus Bados

Materiales Magnéticos y Eficiencia Energética: Caracterización y Modelado Magnetic Materials and Energy Efficiency: Characterization and Models

Código/Code:	MAT2013-45165-P
Periodo/Period:	01-01-2014 / 31-12-2017
Organismo Financiador/Financial source:	Ministerio de Ciencia e Innovación
Importe total/Total amount:	118.530,61 €
Investigador responsable/Research head:	Alejandro Conde Amiano / Victorino Franco García
Componentes/Research group:	Clara F. Conde Amiano, Josefa María Borrego Moro, Javier S. Blázquez Gámez , Jhon J. Ipus Bados, Laszlo F. Kish

RESUMEN / ABSTRACT

Se estudiarán materiales magnéticos con aplicacabilidad en sistemas para un uso eficiente de la energía, centrando nuestra atención, fundamentalmente, en dos aspectos interrelacionados: la refrigeración magnética y los materiales compuestos para la recuperación de energía. En lo referente al efecto magnetocalórico, se estudiarán materiales con una transición de fase de primer orden, en los que el control de la microestructura, la composición y campos externos como la presión o el campo eléctrico pueden alterar la respuesta termomagnética. Se

abordará la problemática actual de dos familias de materiales magnetocalóricos con alto interés tecnológico: los compuestos del tipo LaFeSi y las aleaciones Heusler. Los primeros, especialmente los compuestos hidrogenados, son altamente prometedores para aplicaciones industriales, pero sufren una migración del hidrógeno que da lugar a una disminución del pico de respuesta magnetocalórica que se desdobla en dos picos menores. El control de la microestructura y la modificación de la composición debe dar lugar a una mejora en la estabilidad de las propiedades. Las aleaciones Heusler, a pesar de contar con una elevada respuesta magnetocalórica, presentan dificultades para su aplicación tecnológica, debido a la significativa irreversibilidad de la transición microestructural. Esfuerzos combinados de modificación de la composición y modelado de la transición de fase deben permitirnos minimizar esta histéresis térmica y mejorar la aplicabilidad de las aleaciones Heusler. El estudio experimental de materiales magnetocalóricos requiere de técnicas apropiadas para la caracterización de la variación de entropía magnética y de la variación de temperatura adiabática. La primera puede obtenerse mediante medidas indirectas de imanación, pero los protocolos de medida para materiales con una transición de fase de primer orden existentes en la actualidad implican procedimientos costosos en tiempo (y, consecuentemente, de alto coste económico). La modelización de las transiciones de fase nos permitirá diseñar algoritmos de medida más eficientes. Por otra parte, la caracterización de la respuesta dinámica de los materiales magnetocalóricos sólo puede hacerse mediante la medida directa de la variación de temperatura adiabática, existiendo un número muy limitado de dispositivos comerciales que permitan realizar estas medidas de manera fiable, especialmente para muestras de reducida masa. Realizaremos estudios para desarrollar y optimizar un equipo de medida directa de la variación de temperatura adiabática para el estudio de la respuesta dinámica de los materiales, lo que tendrá implicaciones directas en el análisis de la viabilidad de emplear estos materiales en dispositivos refrigeradores. En especial, se estudiará la posibilidad de realizar medidas con excitación AC para la detección de pequeñas respuestas. El tercer bloque de estudio de este proyecto se centra en el análisis de otros efectos cruzados en materiales con transiciones de fase magnéticas o magnetoestructurales. Mediante el desarrollo de materiales multiferroicos artificiales, preparados por procedimientos combinados de aleado mecánico y "spin coating", estudiaremos la viabilidad de la recuperación de energía por medios termomagnéticos.

In this project, we will study magnetic materials with applicability in systems for energy efficiency. Our interest will be focused in two interrelated topics: magnetic refrigeration and composite materials for energy harvesting. Concerning magnetocaloric effect, we will study materials with a first order phase transition, which thermomagnetic response can be controlled through tailoring microstructure and composition. Other external excitations, such as pressure and electric field, can also affect the thermomagnetic response. We will focus on some of the currently open problems of two families of magnetocaloric materials with high technological interest: LaFeSi-type compounds and Heusler alloys. In hydrogenated compounds of the former family, despite their promising industrial applicability, hydrogen migrates yielding a reduction of the peak of the magnetocaloric response, which splits in two smaller peaks. The control of the microstructure and the compositional tailoring should lead to an enhancement of the stability of the properties. Heusler alloys, despite their high magnetocaloric response, show a strong irreversibility at the microstructural transition, which hinders their technological application. Tailoring the composition along with modelling of the phase transition should allow us to minimize the detrimental thermal hysteresis and to enhance the applicability of Heusler alloys. The experimental study of magnetocaloric materials requires appropriate techniques for the characterization of the isothermal magnetic entropy change and the adiabatic temperature change. Although the former can be obtained from indirect measurements of magnetization, the measurement protocols for materials with a first order phase transition used nowadays demand long time measurements (and, consequently, have a high economical cost). Modeling of the phase transitions will allow us to design more efficient measurement algorithms. On the other hand, the characterization of the dynamic response of the magnetocaloric materials can only be performed by direct measurements of the adiabatic temperature change. However, there is a reduced number of commercial devices supplying reliable measurements of this magnitude, especially for samples with a low mass. We will carry out studies for developing and optimizing a setup for the characterization of the dynamic response of materials. This will have direct implications in the analysis of the viability to use these materials in refrigerators. Particularly, we will study the viability to perform AC measurements to detect weak reponses. Finally, the analysis of cross-effects in materials with magnetic or magnetostructural transitions will be undertaken. Especially, we will study the viability for thermomagnetic energy harvesting through the development of artificial multiferroic materials prepared by combined mechanical alloying and spin coating techniques.

Procesado, caracterización y propiedades mecánicas de cerámicos nanoestructurados reforzados con nanotubos de carbono

Código/Code:	P12-FQM-1079 (Proyecto de Excelencia)
Periodo/Period:	30-01-2014 / 16-02-2019
Organismo Financiador/Financial source:	Junta de Andalucía
Investigador responsable/Research head:	Arturo Domínguez Rodríguez
Componentes/Research group:	Luis María Esquivias Fedriani, Angela Gallardo López, Diego Gómez García, Felipe Gutiérrez Mora, Victor Morales Flórez, Ana Morales Rodríguez, Rodrígo Moreno Botella, Antonio Muñoz Bernabé, Rosalía Poyato Galán, Eugenio Zapata Solvas

PROYECTOS EN LOS QUE PARTICIPAN INTEGRANTES DE LA UNIDAD

Avalanchas en Biofísica, Materiales y Plasmas

Código/Code:	MAT2015-69777-REDT
Periodo/Period:	27-11-2015 / 28-11-2017
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total (UEI)/Total amount (UEI):	30.000 €
Investigador responsable/Research head:	Eduard Vives-Santa-Eulalia (U. Barcelona)
Investigador responsable (UEI) Research head (UEI):	María del Carmen Gallardo Cruz
Componentes/Research group:	José María Martín Olalla, Francsico Javier Romero Landa

RESUMEN / ABSTRACT

Bajo la denominación genérica de “avalanchas” se incluyen una serie de fenómenos físicos que consisten en la respuesta discontinua, estocástica e intermitente de un sistema cuando se conduce de forma suave y continua: variaciones repentinas del parámetro de orden ocurren separadas por intervalos sin ninguna actividad aparente. Existen ejemplos muy variados y a distintas escalas espaciales y temporales que incluyen la fractura de un material poroso nanoestructurado bajo compresión, las transiciones de fase estructurales en sólidos, el ruido Barkhausen en un material ferromagnético cuando se imanta, algunos fenómenos de condensación y ebullición, la respuesta de los tejidos neuronales, las verdaderas avalanchas (aludes) de nieve, los terremotos o incluso los plasmas de fusión y las llamaradas solares. Las dimensiones espaciales, energéticas y las duraciones de las avalanchas suelen presentar distribuciones estadísticas que, si no son de tipo ley de potencias, en general tienen colas largas. Es decir su comportamiento se aleja del comportamiento gaussiano esperable. El objetivo de esta red es discutir desde una aproximación multidisciplinar y holística las diferencias y similitudes entre estos fenómenos y la posibilidad que se puedan describir bajo un marco físico-estadístico común. Tambien se pretende importar y exportar las técnicas experimentales y estadísticas para el estudio de las correlaciones temporales y espaciales de las avalanchas entre las diferentes disciplinas:

física de materiales, biofísica, geofísica, física de plasmas. El reto final sería contribuir de forma importante al conocimiento y a la mejora de los métodos de caracterización, evaluación de riesgos, predicción y alarma de estos fenómenos, desde las fracturas en materiales hasta los terremotos y las llamaradas solares.

Under the name of “avalanches” we designate many physical phenomena that consist in the discontinuous, stochastic and intermittent response of a system under smooth and continuous external driving: sharp variations of the order parameter occur separated by intervals with apparently no activity. There are many examples at different spatial and temporal scales including: the failure of nanostructured porous materials under compression, the structural transitions in solids, Barkhausen noise in ferromagnetic materials, some condensation and boiling phenomena, the response of neural tissues, the real snow avalanches, earthquakes and even

fusion plasmas and solar flares. The spatial and energetic dimensions as well as the durations of the avalanches display often power-law behavior or, at least, fat tails. Their behaviour clearly differs from the expected gaussian behaviour. The goal of this network is to discuss from a multidisciplinar and holistic approximation the differences and similarities of these phenomena and the possibility to describe all of them in a comon statistical mechanics framework. We also pretend to export and import the experimental and statistical analysis techniques for the study of the spatial and temporal correlations of the avalanches in the different disciplines: materials physics, biophysics, geophysics and plasma physics. The final goal will be to contribute to the knowledge and improvement of the methods for characterization, risk evaluation, forecast and alarm of these phenomena, from materials failure to earthquakes and solar flares.

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

A hybrid silver-magnetite detector based on surface enhanced Raman scattering for differentiating organic compounds
Caro, C; Sayagues, MJ; Franco, V; Conde, A; Zaderenko, P; Gamez, F
Sensors and Actuators B-Chemical, **228** (2016) 124-133
DOI: 10.1016/j.snb.2016.01.003

In this work a cheap detector of organic molecules is developed. It comprises a cellulose fiber doped with a mixture of magnetite and reduced silver nanoparticles, the latter ones synthesized anew. The nanoparticles and the fiber were characterized with well-established spectroscopic, microscopic and magnetic techniques, namely infrared, UV-vis spectroscopies, vibrating sample magnetometry and electronic microscopies. The so-obtained doped-fibers were tested as surface enhanced Raman spectroscopy detector in aqueous samples with a diluted mixture of two pollutant models (rhodamine 6G and picric acid), being able to differentiate between both organic compounds. Hence, the nanoparticle-impregnated fiber is proposed as a reliable preliminary qualitative and semiquantitative test of the presence of specific organic molecules in solutions. Moreover, the magnetite nanoparticles provide the detector with a saturation magnetization value that enables the separation of the fiber from the solution with the aid of a commercial magnet.

Applicability of scaling behavior and power laws in the analysis of the magnetocaloric effect in second-order phase transition materials
Romero-Muniz, C; Tamura, R; Tanaka, S; Franco, V
Physical Review B, **94** (2016) art 134401
DOI: 10.1103/PhysRevB.94.134401

In recent years, universal scaling has gained renewed attention in the study of magnetocaloric materials. It has been applied to a wide variety of pure elements and compounds, ranging from rare-earth-based materials to transition metal alloys, from bulk crystalline samples to nanoparticles. It is therefore necessary to quantify the limits within which the scaling laws would remain applicable for magnetocaloric research. For this purpose, a threefold approach has been followed: (a) the magnetocaloric responses of a set of materials with Curie temperatures ranging from 46 to 336 K have been modeled with a mean-field Brillouin model, (b) experimental data for Gd has been analyzed, and (c) a 3D-Ising model-which is beyond the mean-field approximation-has been studied. In this way, we can demonstrate that the conclusions extracted in this work are model-independent. It is found that universal scaling remains applicable up to applied fields, which provide a magnetic energy to the system up to 8% of the thermal energy at the Curie temperature. In this range, the predicted deviations from scaling laws remain below the experimental error margin of carefully performed experiments. Therefore, for materials whose Curie temperature is close to room temperature, scaling laws at the Curie temperature would be applicable for the magnetic field range available at conventional magnetism laboratories (similar to 10 T), well above the fields which are usually available for magnetocaloric devices.

Time evolution of mechanical amorphization: A kinetic model
Blazquez, JS; Ipus, JJ; Conde, A
Scripta Materialia, **130** (2016) 260-263
DOI: 10.1016/j.scriptamat.2016.12.019

A model is proposed to describe the evolution of the amorphization process driven by mechanical grinding. This model considers the interface controlled growth of the disordered regions at the boundaries between the crystallites (unlike the dassical nucleation and growth theory, the growing particles are thus concave which affects the kinetic equation). The validity of this model has been tested for the amorphization process of mechanically alloyed Fe-Nb-B system at different frequencies. Predictions of the model are in agreement with the experimental results and values of the linear growth rate and the thickness of the starting disordered layer are obtained.

A unified approach to describe the thermal and magnetic hysteresis in Heusler alloys
Blazquez, JS; Franco, V; Conde, A; Gottschall, T; Skokov, KP; Gutfleisch, O
Applied Physics Letters, **109** (2016) art 122410
DOI: 10.1063/1.4963319

Different excitations, like temperature, magnetic field, or pressure, can drive a martensitic transition in Heusler alloys. Coupled phenomena in these materials lead to interesting magnetocaloric and barocaloric effects ascribed to this transition. In this work, we demonstrate that isothermal transformations induced by a magnetic field and isofield transformations induced by the temperature can be described using the same framework. By defining an effective temperature that relates field and temperature through the properties of the system (magnetic moment and entropy of the transition), both kinds of loops can be transformed into the other kind, therefore providing a more effective way of characterizing hysteretic samples. The validity of this effective temperature approach to describe the transition holds for martensite to austenite transformations as well as reversal ones, and thus, the hysteresis phenomena can be described using this single general excitation.

Large magnetocaloric effect in fine (GdO3)-O-2 nanoparticles embedded in porous silica matrix
Zelenakova, A; Hrubovcak, P; Kapusta, O; Zelenak, V; Franco, V
Applied Physics Letters, **109** (2016) art 122412
DOI: 10.1063/1.4963267

The magnetocaloric properties of a composite material consisting of isolated Gd2O3 nanoparticles with a diameter of 6-8 nm embedded in the pores of a mesoporous silica matrix have been studied. The fascinating nanostructure and composition were properly characterized by small angle X-ray scattering, X-ray absorption near edge structure, and TEM. Almost ideal paramagnetic behavior of the material was observed in the temperature range of 1.8-300 K. When compared to various nanosystems, the presented composite exhibits an extraordinarily large magnetic entropy change of 40 J/kgK for a field variation of 0-5 T at cryogenic temperature (3 K). Considering only the mass of the Gd2O3 nanoparticle fraction, this corresponds to 120 J/kg K. Calculated refrigerant capacities are 100 J/kg and 400 J/kg for the composite and nanoparticles, respectively. Our findings suggest that the combination of the unique porous structure of amorphous silica with fine gadolinium oxide nanoparticles and high value of magnetic entropy change enables to extend the application of the Gd2O3@SiO2 composite, to cryomagnetic refrigeration. In addition, the characteristics of the thermomagnetic behavior have been studied using the scaling analysis of the magnetic entropy change.

The influence of magnetocrystalline anisotropy on the magnetocaloric effect: A case study on Co2B
Fries, M; Skokov, KP; Karpenkov, DY; Franco, V; Ener, S; Gutfleisch, O
Applied Physics Letters, **109** (2016) art 232406
DOI: 10.1063/1.4971839

The influence of magnetocrystalline anisotropy on the magnetocaloric effect (MCE) was studied on single crystals of Co2B and compared to measurements on polycrystalline samples. Large differences in adiabatic temperature change Delta T-ad and magnetic entropy change Delta S-M were found along the different crystallographic directions. The magnetocaloric effect differs by 40% in the case of Delta T-ad in a field change of 1.9 T when applying the field along the hard axis and easy plane of magnetization. In the case of Delta S-M, the values differ 50% and 35% from each other in field changes of 1 and 1.9 T, respectively. It was found that this anisotropy effect does not saturate in fields up to 4 T, which is higher than the anisotropy field of Co2B (approximate to 2 T). A simple model was developed to illustrate the possible effect on magnetocrystalline anisotropy, showing large differences especially in application relevant fields of about 1 T. The results strongly suggest that the MCE could be maximized when orienting single crystalline powders in an easy axis parallel to the applied field in active magnetocaloric regenerator structures, and therefore the overall device efficiency could be increased.

Anisotropy field distribution in soft magnetic Hitperm alloys submitted to different field annealing processes
Blazquez, JS; Marcin, J; Andrejka, F; Franco, V; Conde, A; Skorvanek, I
Journal of Alloys and Compounds, **658** (2016) 367-371
DOI: 10.1016/j.jallcom.2015.10.210

The magnetic anisotropy field distribution is discussed for Hitperm alloys annealed under different field conditions leading to different induced magnetic anisotropies: zero (ZF), transversal (TF), and longitudinal (LF) field annealing and compared to that of as-quenched (AQ) melt-spun amorphous ribbon. In order to accurately use the present method, the demagnetizing factor has been obtained by analyzing the field dependence of the inverse of the field derivative of the magnetization. The coherence of the analysis is supported by testing the normalization of the complete distribution of anisotropy fields. Independently of the composition, two groups can be distinguished among the studied samples: those with mainly perpendicular anisotropy field contributions (ZF and TF samples) and those with mainly longitudinal anisotropy field contributions (LF and AQ samples). Behavior of TF samples is well reproduced using Stoner-Wohlfarth model and, in the case of as-quenched amorphous samples, the anisotropy field depends almost linearly on the thickness of the ribbon.

Gd plus GdZn biphasic magnetic composites synthesized in a single preparation step: Increasing refrigerant capacity without decreasing magnetic entropy change
Law, JY; Moreno-Ramirez, LM; Blazquez, JS; Franco, V; Conde, A
Journal of Alloys and Compounds, **675** (2016) 244-247
DOI: 10.1016/j.jallcom.2016.03.130

Biphasic Gd + GdZn composites (Gd(50+x)Zn(50-x); x = 0, 5, 15 and 25 at.%) were successfully obtained in a single fabrication step by induction melting. X-ray microdiffraction results reveal the homogeneity of all the prepared samples and indicate a combination of GdZn and Gd phases with different proportions. With this method, the main drawbacks of preparing composites have been avoided, providing the additional advantage of enhanced thermal conductivity between phases. The biphasic Gd + GdZn composite shows an enhanced refrigerant capacity in comparison to Gd (11%) as well as to single phase GdZn (45%). Heat capacity measurements provide an adiabatic temperature change of around 3.5 K for 20 kOe for the optimal composite.

Influence of nanocrystallization on the magnetocaloric properties of Ni-based amorphous alloys: Determination of critical exponents in multiphase systems
Sanchez-Perez, M; Moreno-Ramirez, LM; Franco, V; Conde, A; Marsilius, M; Herzer, G
Journal of Alloys and Compounds, **686** (2016) 717-722
DOI: 10.1016/j.jallcom.2016.06.057

Traditionally the study of the magnetocaloric effect (MCE) in amorphous and nanocrystalline alloys has been focused on Fe and rare earth based compositions. However, they present problems that hinder their application such as the high price of rare earths or the necessary addition of a large fraction of nonmagnetic elements in Fe-based alloys in order to tune their response to temperatures close to room temperature. Alternatively, Ni-based amorphous alloys have the Curie temperature (TC) well below room temperature and with a good glass forming ability. For these alloys, Fe addition provides a good tool to tune TC close to room temperature. In this work, the magnetocaloric properties of Ni82-xFexSi2B16 (x = 8, 12, 13.2, 16) have been studied in the amorphous and nanocrystalline state. Besides compositional dependence of MCE in this series, a detailed analysis of the critical exponents of the magnetic transition of both amorphous and nanocrystalline alloys has been performed using the magnetic field dependence of MCE. Results were compared with those derived from the Kouvel-Fisher method, requiring a deconvolution of the different magnetization contributions in the Kouvel-Fisher case for nanocrystalline alloys.

Nanocrystallization kinetics understood as multiple microprocesses following the classical theory of crystallization
Manchon-Gordon, AF; Blazquez, JS; Conde, CF; Conde, A
Journal of Alloys and Compounds, **675** (2016) 81-85

DOI: 10.1016/j.jallcom.2016.03.087

In this work, we propose a model for using the classical Johnson-Mehl-Avrami-Kolmogorov (JMAK) crystallization theory to analyze nanocrystallization processes as a set of multiple microprocesses. This model is based on the well-known microstructure observations of nanocrystalline systems for which, although the number of crystallites increases along the process, the growth of each crystallite is limited to a much shorter time than the needed one for completing the process. The very low values of the Avrami exponent, n similar to 1, are well reproduced assuming a set of multiple classical JMAK processes with constant nucleation rate and diffusion controlled growth, n(i) = 2.5 for each i individual microprocess. It is shown that the values of Avrami exponent experimentally observed and lying out of the theoretical range can be assumed as effective values derived from a complex process consisting of multiple microprocesses, which can be individually treated as classical ones.

Influence of hot compaction on microstructure and magnetic properties of mechanically alloyed Fe(Co)-based amorphous compositions
Ipus, JJ; Borrego, JM; Blazquez, JS; Stoica, M; Franco, V; Conde, A
Journal of Alloys and Compounds, **653** (2016) 546-551
DOI: 10.1016/j.jallcom.2015.09.074

Amorphous Fe75Nb10B15 and (Fe70Co30)(75)Nb10B15 alloys were prepared by mechanical alloying from the elemental constituents and hot compacted at different temperatures within the supercooled liquid region. After compaction, the microstructure studied by X-ray diffraction shows an increase in the crystalline fraction for both compositions. Magnetic properties and magnetic entropy change of compacted samples of the Co-free alloy were enhanced with respect to the powder sample, meanwhile, a deterioration in these properties was observed for the Co containing alloy after compaction. Both changes could be ascribed to an enrichment in boron in the amorphous phase.

Magnetocaloric response of amorphous and nanocrystalline Cr-containing Vitroperm-type alloys
Moreno-Ramirez, LM; Blazquez, JS; Franco, V; Conde, A; Marsilius, M; Budinsky, V; Herzer, G
Journal of Magnetism and Magnetic Materials, **409** (2016) 56-61
DOI: 10.1016/j.jmmm.2016.02.0787

The broad compositional range in which transition metal (TM) based amorphous alloys can be obtained, yields an easily tunable magnetocaloric effect (MCE) in a wide temperature range. In some TM-based alloys, anomalous behaviors are reported, as a non-monotonous trend with magnetic moment (e.g. FeZrB alloys). Moreover, in certain Cr-containing Vitroperm alloys anomalously high values of the magnetic entropy change were published. In this work, a systematic study on MCE response of Cr-containing amorphous alloys of composition Fe74-xCrxCu1Nb3Si15.5B6.5 (with x=2, 8, 10, 12, 13, 14 and 20) has been performed in a broad Curie temperature range from 100 K to 550 K. Curie temperature and magnetic entropy change peak of the amorphous alloys decrease with the increase of Cr content at rates of -25.6 K/at% Cr and -54 mJ kg(-1) K-1/at% Cr, respectively, following a linear trend with the magnetic moment in both cases. The presence of nanocrystalline phases has been considered as a possible cause in order to explain the anomalies. The samples were nanocrystallized in different stages, however, the magnetocaloric response decreases as crystallization progresses due to the large separation of the Curie temperatures of the two phases.

Assessment of the magnetocaloric effect in La,Pr(Fe,Si) under cycling
Kaeswurm, B; Franco, V; Skokov, KP; Gutfleisch, O
Journal of Magnetism and Magnetic Materials, **406** (2016) 259-265
DOI: 10.1016/j.jmmm.2016.01.045

The response of a magnetocaloric material to periodic variations of magnetic field and temperature corresponding to those occurring during a magnetic refrigeration process is studied. A series of simple measurement protocols are suggested which are used to obtain a value for the cyclic response of the magnetic entropy change associated with the magnetic transition. The entropy values are compared to direct measurements of the temperature change under adiabatic conditions. The procedure is illustrated on the first order magnetocaloric material La0.6Pr0.4Fe11.6Si1.4 and provides a basis for comparison of the suitability of different hysteretic magnetocaloric materials for application in a magnetic refrigerator. For the alloy studied here the peak magnetic entropy change of -28 +/- 11 kg(-1) K-1 in a field change of 2 T is not affected by cycling, but the full width at half maximum of the peak decreases from 8.7 K to 3.8 K.

Optimal temperature range for determining magnetocaloric magnitudes from heat capacity
Moreno-Ramirez, LM; Blazquez, JS; Law, JY; Franco, V; Conde, A
Journal of Physics D-Applied Physics, **49** (2016) art. 495001
DOI: 10.1088/0022-3727/49/49/495001

The determination of the magnetocaloric magnitudes (specific magnetic entropy change, Delta s(M), and adiabatic temperature change,Delta T-ad) from heat capacity (c(H)) measurements requires measurements performed at very low temperatures (similar to 0 K) or data extrapolation when the low temperature range is unavailable. In this work we analyze the influence on the calculated Delta s(M) and Delta T-ad of the usually employed linear extrapolation of cH from the initial measured temperature down to 0 K. Numerical simulations have been performed using the Brillouin equation of state, the Debye model and the Fermi electron statistics to reproduce the magnetic, lattice and electronic subsystems, respectively. It is demonstrated that it is not necessary to reach experimentally temperatures very close to 0 K due to the existence of certain starting temperatures of the experiments, the same for Delta s(M) and Delta T-ad, that minimize the error of the results. A procedure is proposed to obtain the experimental magnitudes of Delta s(M) and Delta T-ad with a minimum error from cH data limited in temperature. It has been successfully applied to a GdZn alloy and results are compared to those derived from magnetization measurements.

Structural, magnetic properties and magnetocaloric effect of Mn1.2Fe0.8P1-xSixB0.03 compounds
Zheng, ZG; Tan, ZC; Yu, HY; Zhang, JL; Zeng, DC; Franco, V
Materials Research Bulletin, **77** (2016) 29-34
DOI: 10.1016/j.materresbull.2016.01.017

Structural, magnetic, and magnetocaloric properties of the Mn1.2Fe0.8P1-xSixB0.03(x = 0.4, 0.5 and 0.55) phases have been investigated. The experimental results show that the critical behavior of the paramagnetic to ferromagnetic phase transition can be tuned through the P/Si ratio. An increase in the Si concentration not only raises the Curie temperature from 176 K to 280 K, but also changes the nature of the magnetic transition from the first order (for x = 0.4) to the second order (for x = 0.55) and, thereby, affects the magnetocaloric properties. The peak values of the thermal hysteresis and magnetic entropy change for a magnetic field change of 0-2T are 10 K, 4K, 0.8'K and 163, 8.7, 5.6 J kg(-1) K-1 for x = 0.4, 0.5, and 0.55, respectively. Mechanisms for the magnetic hysteresis loss and possibilities for the room temperature magnetic refrigeration are discussed.

Study of the Induced Anisotropy in Field Annealed Hitperm Alloys by Mossbauer Spectroscopy and Kerr Microscopy

Blazquez, JS; Marcin, J; Andrejka, F; Franco, V; Conde, A; Skorvanek, I

Metallurgical and Materials Transactions A-Physical Metallurgy and Materials Science, **47A** (2016) 4301-4305

DOI: 10.1007/s11661-016-3562-z

Samples of Fe39Co39Nb6B15Cu1 alloy were nanocrystallized under zero field annealing (ZF) and transverse field annealing (TF) conditions. A reduction in coercivity for TF samples with respect to ZF sample (16 and 45 A/m, respectively) is observed. Kerr microscopy images show a well-defined parallel domain structure, transversally oriented to the ribbon axis for the TF sample unlike for the ZF sample, for which a complex pattern is observed with large and small domains at the surface of the ribbon. Although Mossbauer spectra are clearly different for the two studied samples, Mossbauer studies confirm that there is no significant difference between the hyperfine field distributions of TF and ZF samples but only the relative intensity of the 2nd and 3rd lines A (23) (related to the angle between the gamma radiation and the magnetic moments, alpha). However, for TF annealed samples alpha = 90 deg (A (23) = 4), indicating that the magnetic moments lay on the plane of the ribbon in agreement with the well-defined domain structure observed by Kerr microscopy, ZF annealed samples show A (23) = 1.8. This value is close to that of a random orientation (A (23) = 2) but smaller, indicating a slight preference for out of plane orientations. Moreover, it is clearly smaller than that of the as-cast amorphous samples A (23) = 2.8, with a preference to in-plane orientations. The application of the law of approach to saturation yields a larger effect of the inhomogeneities in ZF sample with respect to TF one.

A New Method for Determining the Curie Temperature From Magnetocaloric Measurements

Moreno-Ramirez, LM; Blazquez, JS; Franco, V; Conde, A; Marsilius, M; Budinsky, V; Herzer, G

IEEE Magnetics Letters, **7** (2016) art 6102004

DOI: 10.1109/LMAG.2016.2533481

A new method is proposed for determining the Curie temperature from magnetocaloric measurements. It is based on the field dependence of the magnetic entropy change close to the Curie temperature. The main advantages over other methods are that the obtained temperature is field independent, and the process is noniterative and does not require a fitting procedure nor prior knowledge of the critical exponents of the transition. The reliability of the method is demonstrated using both simulated and experimental data for pure Ni and an Fe-based amorphous alloy.

First-Order Reversal Curve (FORC) Analysis of Magnetocaloric Heusler-Type Alloys
Franco, V; Gottschall, T; Skokov, KP; Gutfleisch, O
IEEE Magnetics Letters, **7** (2016) art 6602904
DOI: 10.1109/LMAG.2016.2541622

The thermomagnetic hysteresis loops of a Ni45.7Mn36.6In13.5Co4.2 Heusler-type alloy exhibiting inverse magnetocaloric effect were studied with the help of first-order reversal curves (FORC). These have been measured using two different protocols (either upon heating or cooling the sample) and using different applied magnetic fields. For proper comparison, FORC distributions were shifted according to the field dependent center of the M(T) hysteresis loop, which follows a linear trend. The qualitative behavior of FORC distributions remains the same, allowing their use for fingerprinting the transition, while there is a shift of their maxima along the hysteretic temperature axis and their distributions also get broader along the interaction temperature axis with increasing magnetic field. This was evidence that FORC distributions are dependent on the intensive variables temperature and field. As a consequence, it is necessary to obtain them for different temperatures and fields in order to accurately model the transition.

DOCENCIA / TEACHING

Máster Profesorado de ESO y Bachillerato, FP
Complementos de formación disciplinar en Física y Química
Dr. Javier S. Blázquez
Lugar: Universidad de Sevilla

EQUIPAMIENTO CIENTÍFICO AVAILABLE EQUIPMENT

- Calorímetro de barrido diferencial (Perkin-Elmer DSC7)
- Criostato para espectrómetro Móssbauer
- Balanza termogravimétrica (Perkin-Elmer TGA-7)
- Espectrómetro Mössbauer (Wissel MB-500) con horno y criostato.
- Coercímetro (desarrollado en el laboratorio)
- Magnetómetro de muestra vibrante (LakeShore 7000) con horno y criostato.
- Equipo de medida directa de temperatura adiabática (Advanced Magnetic Technologies).
- Molino Planetario (Fritszch Pulverisette Vario 4)
- Equipo de solidificación por enfriamiento ultrarrápido (melt spinning, Bühler)
- Equipo de fusión por arco (MAM1, Bühler)
- Calorímetro de conducción, resolución en la medida del flujo de calor mejor que 0,1▯W, fluctuaciones en temperatura del orden de 10-6 K, velocidad de barrido

menor de 0,01K/h, rango 80-320K, es posible aplicar tensión uniaxial hasta 30 kg/cm2 y campo eléctrico hasta 800V/cm

- Calorímetro de conducción, rango 80-400K, campo eléctricos 2000V/cm
- Medida de constante dieléctrica, en los mismos calorímetros,
- Medida de ciclo de histéresis en Ferroeléctricos.
- Analizador de Impedancia

SERVICIOS GENERALES
GENERAL SERVICES

SERVICIO DE ESPECTROSCOPIAS / SPECTROSCOPY SERVICE

El Servicio de Espectroscopias incluye las Unidades de Espectroscopía de Resonancia Magnética Nuclear, Espectroscopía Raman, Espectroscopía Infrarroja y Espectroscopía Ultravioleta-Visible. Este servicio está dedicado a la determinación de la estructura molecular de los compuestos químicos y la caracterización de materiales.

This Service consists of four different spectroscopies: Nuclear Magnetic Resonance Spectroscopy, Raman Spectroscopy, Infrared Spectroscopy and Ultraviolet-Visible Spectroscopy. It is devoted to the determination of molecular structure of chemical compounds and materials.

ESPECTROSCOPÍA MICRO-RAMAN / MICRO-RAMAN SPECTROSCOPY

La espectroscopía Raman se basa en un proceso fotónico en el que la radiación incidente es dispersada por la muestra, produciéndose transiciones de tipo vibracional y rotacional. En general, el espectro Raman se interpreta como un espectro vibracional que ofrece información muy similar al espectro de infrarrojo, aunque las vibraciones que se ven reflejadas en el espectro Raman no son siempre las mismas que en aquél. Para que un modo vibracional sea activo en espectroscopía Raman es necesario que se produzcan cambios en la polarizabilidad de los enlaces químicos o la molécula considerada, lo que conlleva la producción de momentos dipolares inducidos. Su campo de aplicación es muy extenso: semiconductores, compuestos del carbono (grafito, diamante, nanotubos, fibras…), catalizadores, pigmentos, etc.

Raman spectroscopy is based on a photonic process in which the incident radiation is dispersed by the sample. This latter is perturbed leading to vibrational and rotational transitions. In general, the Raman spectrum is interpreted like a vibrational one, providing information very similar to the infrared spectroscopy, although the Raman active vibrations are not always the same as those excited with infrared radiation. A Raman vibration mode is active if there is a change of polarizability of the chemical bonds or the considered molecule, which in turn results in the generation of induced dipolar momentam. Its application fields are very broad: semiconductors, carbon compounds (graphite, diamond, nanotubes, fibers…), catalysts, pigments, etc.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- LabRAM Horiba Jobin Yvon dotado de un microscopio confocal y 3 longitudes de excitación (785 cm^{-1} rojo, 532 cm^{-1} verde, y 325 cm^{-1} UV)
  LabRAM Horiba Jobin Yvon equipped with a confocal microscope and 3 excitation lasers (785 cm^{-1} red, 532 cm^{-1} green, and 325 cm^{-1} UV)

Responsables Científicos/ Scientific Responsibles: Dr. Juan Carlos Sánchez López, Dr. Hernán Míguez García y Dr. Miguel Angel Centeno Gallego
Personal Técnico/ Technical Assistant: Dr. Miguel Angel Avilés Escaño

ESPECTROSCOPÍA INFRARROJA / INFRARED SPECTROSCOPIES

La espectroscopía de Infrarrojos (FT-IR) se basa en la absorción de radiación infrarroja por parte de los materiales. Esta absorción supone un cambio en la energía vibracional de los enlaces, siempre que se produzca un cambio en la polarización de dicho enlace. El resultado obtenido es un espectro en el que se representa la radiación absorbida o transmitida en función del número de onda de la radiación, lo cual permite identificar el enlace correspondiente.
El equipo en el ICMS cubre un rango de número de ondas que va desde 5000 a 250 cm-1 (óptica de CsI) y se puede trabajar con purga o en vacío. Se halla equipado con accesorios para trabajar en los modos de Reflectancia Difusa (DRIFT), Reflectancia Total Atenuada (ATR) y Reflexión Especular. Dispone de un microscopio de Infrarrojos que tiene una resolución lateral de 10 µm.

Infrared spectroscopy (FT-IR) is based on the selective absorption of the infrared radiation by the materials. This absorption means a change in the vibrational energy of the chemical bonds, whenever it occurs a change in the polarization. The result is a spectrum showing the absorbed or transmitted radiation as a function of the wavenumber of the radiation, which can be assigned to the corresponding chemical bound.
The equipment at the ICMS works in a wavenumber range from 5000 to 250 cm^{-1} (CsI optic), and can operate with a gas purge or in vacuum. It is equipped with several accessories to do Diffuse Reflectance (DRIFT), Attenuated Total Reflectance (ATR) or Specular Reflectance. It has got an Infrared Microscope with a lateral resolution of 10 µm.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- JASCO FT/IR-6200 IRT-5000
  JASCO FT/IR-6200 IRT-5000

Responsables Científicos/ Scientific Responsibles: Dr. Manuel Ocaña Jurado y Dr. Ángel Barranco Quero
Personal Técnico/ Technical Assistant: Dr. Miguel Angel Avilés Escaño

ESPECTROSCOPÍA ULTRAVIOLETA / ULTRAVIOLET-VISIBLE SPECTROSCOPIES

La espectroscopía Ultravioleta-Visible (UV-Vis) informa sobre las diferencias de energía existentes entre los niveles electrónicos ocupados más externos y los desocupados más próximos.
Se dispone en el ICMS de dos equipos que cubren el rango de longitudes de onda que va desde 190 nm hasta 900 nm. Se puede trabajar tanto en el modo de Transmisión como en el de Reflectancia Difusa.

The Ultraviolet-Visible Spectroscopy (UV-Vis) reports on the existing energy differences between the more external occupied electronic levels and the nearer unoccupied ones.
There are two equipments in the laboratory, which work in the wavelength range of 190 nm to 900 nm. It can operate in the Transmission mode or in Diffuse Reflectance Modes.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- SHIMADZU UV-2101 PC
  SHIMADZU UV-2101 PC
- Perkin Elmer Lambda 12
  Perkin Elmer Lambda 12

Personal Técnico/ Technical Assistant: Dr. Miguel Angel Avilés Escaño

SERVICIO DE ANÁLISIS TEXTURAL Y TÉRMICO / TEXTURAL AND THERMAL ANALYSIS SERVICE

Este servicio incluye las siguientes Unidades: Análisis Térmico, Fisi-quimisorción, Análisis de Tamaño de Partícula y Potencial Z. Está dedicado a la determinación de la textura, estructura y comportamiento térmico de los materiales.

This Service includes the following units: Thermal Analysis, Physisorption and Chemisorption, Particle Size and Z-potential determination. It is devoted to the characterization of texture, microstructure and thermal behavior of advanced materials.

FISI-QUIMISORCIÓN / PHYSISORPTION-CHEMISORPTION

Este servicio constituye una herramienta básica para la caracterización microestructural de sólidos pulverulentos de distinta naturaleza, en cuanto a porosidad, superficie específica y superficie químicamente activa.

En el servicio se dispone de un analizador de adsorción de gases (Micromeritics, ASAP 2020) que proporciona isotermas de adsorción y desorción, a partir de los cuales se obtienen de ellas la superficie específica y distribución del tamaño de poro y de microporo de estos materiales, incorporando también los accesorios necesarios para medidas de quimisorción.

This service constitutes a basic tool for the microstructural characterization of powdered solids of different natures, regarding to their porosity, specific surface area and chemically active surface.

This service is composed by a phyisisorption analyser (Micromeritics, ASAP 2020) which provides the complete adsorption/desorption isotherms, from which the specific surface area, pore and micropore size distribution and concentration of reactive sites are obtained. The instrument is also equipped for carrying out chemisorption of different reactive molecules, as O_2, H_2, CO, etc.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- Analizador científico de fisisorción ASAP2010 (Micromeritics)
  Physisorption analyser ASAP 2010 (Micromeritics)
- Analizador de quimisorción ASAP2010 (Micromeritics)
  Chemisorption analyser ASAP 2010 (Micromeritics)
- Analizador de fisisorción multimuestra TRISTAR II (Micromeritics)
  Multisample physisorption analyser TRISTAR II (Micromeritics)
- Analizador de fisisorción multimuestra TRISTAR II-Kr (Micromeritics)
  Multisample physisorption analyser TRISTAR II-Kr (Micromeritics)

Responsables Científicos/ Scientific Responsibles: Dr. Gerado Colón Ibáñez y Dr. Miguel Angel Centeno Gallego
Personal Técnico/ Technical Assistant: Dª Cristina Gallardo López

ANÁLISIS TÉRMICO / THERMAL ANALYSIS

Las técnicas de análisis térmico permiten estudiar aquellos cambios físicos o químicos que ocurren en los sólidos en función de la temperatura y que conlleven modificaciones en su masa o intercambios de calor con su entorno.
En el servicio se pueden realizar experimentos desde temperatura ambiente hasta 1500ºC, tanto en atmósfera inerte (N_2) como reactiva (aire, O_2,…).
Se dispone de dos técnicas: Análisis Termogravimétrico (TG) y Análisis Térmico Diferencial (ATD).

Thermal analysis techniques allow to studying physical or chemical changes occurring in solid in samples as a function of the temperature. Those changes should involve either a mass change or a heat flow.
The experiments can be performed in the range from room temperature to 1500ºC, both under inert (N_2), or reactive (air, O_2,…) atmospheres.
Two different techniques are available: Thermogravimetry (TG) and Differential Thermal Analysis (DTA)

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- Equipo de análisis térmico simultáneo TG/ATD/CDB TA Instruments Q600
  TA Instruments Q600 Simultaneous TG/DTA/DSC instrument
- Equipo termogravimétrico TG, TA Instruments Q5000
  Thermogravimetric instrument TG, TA Instruments Q5000

Responsable Científico/ Scientific Responsible: Dr. Luis A. Pérez Maqueda
Personal Técnico/ Technical Assistant: Dª Cristina Gallardo López

TAMAÑO DE PARTÍCULAS Y POTENCIAL Z / PARTICLE SIZE AND Z POTENTIAL ANALYSIS

Se dispone de las técnicas de Dispersión Dinámica de Luz y de Difracción Láser (LD), que permiten determinar la distribución de tamaños de partícula de sistemas coloidales en suspensión (disolvente acuoso u orgánico) en los rangos que van de 3 a 3000 nanómetros (DLS) o de 0.05 a 900 micras (LD).

Así mismo, se dispone de la técnica de Análisis de Movilidad Electroforética para la evaluación del potencial "Z" de sistemas coloidales en suspensión (disolvente acuoso u orgánico).

Dynamic light scattering (DLS) and Laser diffraction (LD) are available for the determination of particle size distributions of colloidal systems (dispersed in aqueous or organic dispersions solutions) in the range 3-3000 nanometers (DLS) and 0.05-900 microns (LD).

Electrophoretic mobility measurements can be also performed for the evaluation of Z potential in colloidal systems (aqueous or organic dispersions).

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- Malvern modelo Zetamaster (DLS) y MalvernSizer (LD)
  Malvern model Zetamaster (DLS) and MalvernSizer (LD)

Responsable Científico/ Scientific Responsible: Dr. Manuel Ocaña Jurado
Personal Técnico/ Technical Assistant: Dª Cristina Gallardo López

SERVICIO DE MICROSCOPÍA ELECTRÓNICA / ELECTRON MICROSCOPY SERVICE

El servicio está dedicado a la caracterización química y estructural de muestras sólidas mediante técnicas de microscopía electrónica. Las técnicas de caracterización disponibles en el servicio son la Microscopía Electrónica de Transmisión (TEM) y la Microscopía Electrónica de Barrido (SEM), con el equipamiento anexo de preparación de muestras para TEM y SEM.

This Service is devoted to the chemical and structural characterization of solid samples by means of electron microscopies. The characterization techniques available at ICMS are Transmission Electron Microscopy (TEM) and Scanning Electron Microscopy (SEM), with additional equipment for TEM and SEM sample preparation.

MICROSCOPÍA ELECTRÓNICA DE BARRIDO / SCANNING ELECTRON MICROSCOPY

La microscopía electrónica de barrido proporciona información microestructural, morfológica y de composición química en escala microscópica. Se puede aplicar a todo tipo de materiales y problemáticas de estudio en ciencia de materiales: cerámicas, plásticos, metales, minerales, catalizadores, muestras de patrimonio histórico, capas finas, recubrimientos, interfases, nanopartículas, etc. El equipo SEM es un microscopio de emisión de campo de cátodo frío que permite realizar imágenes de la morfología y textura superficial de las muestras con una resolución de 1 nm a 15kV. También permite trabajar a bajo voltaje en muestras sin metalizar y en modo transmisión (STEM-in-SEM) en muestras electrón-transparentes. Acoplado al detector de rayos-X (EDX) permite análisis elementales y mapas composicionales.

The scanning electron microscopy provides information about the microstructure, morphology and chemical composition at the microscopic scale of solid samples. It can be applied to all type of materials including ceramics, polymers, metals, minerals, catalysts, samples from cultural heritage, thin films, coatings, interfaces, nanoparticles, etc. The SEM microscope is a field emission cold cathode equipment which enables images of the surface morphology and texture of samples with a resolution of 1 nm at 15kV. It also allows working at low voltages with non-metalized samples and in transmission mode for electron-transparent samples (STEM-in-SEM). Coupled to the X-ray detector (EDX) enables compositional analysis and elemental mapping.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- Microscopio SEM, modelo Hitachi S4800 SEM-FEG: cañón de emisión de campo de cátodo frío y voltaje de 0.5-30 kV, resolución de 1 nm a 15kV. Dotado de analizador EDX Bruker-X Flash-4010 con una resolución de 133 eV (en la línea MnKα) y detector con portamuestras para trabajar en modo transmisión (STEM-in-SEM).
  Hitachi S4800 SEM-FEG microscope: cold cathode field emission gun with voltage from 0.5 to 30 kV, resolution of 1nm at 15 kV. Equipped with a Bruker-X Flash-4010 EDX detector with a resolution of 133 eV (at the MnKα line), and a detector with sample holder to work in transmission mode (STEM-in- SEM).
- Equipamiento adicional en el "laboratorio de preparación de muestras para microscopía electrónica" (ver sección abajo)
  Additional equipment in the "electron microscopy samples preparation laboratory" (see the section below)

Responsable Científico/ Scientific Responsible: Dra. Asunción Fernández Camacho
Personal Técnico/ Technical Assistant: Dra. M. Carmen Jiménez de Haro

MICROSCOPÍA ELECTRÓNICA DE TRANSMISIÓN / TRANSMISSION ELECTRON MICROSCOPY

La microscopía electrónica de transmisión es una técnica ampliamente utilizada para la caracterización estructural y química de materiales a escala microscópica y nanoscópica, proporcionando imágenes bidimensionales de la textura de la muestra, forma y tamaño de grano y/o de partícula, grado de homogeneidad a escala microscópica, grado de cristalinidad de la muestra, identificación de fases cristalinas, e imágenes de alta resolución que identifican dominios cristalinos. El equipo está dotado de un analizador EDX para el análisis composicional. Puede aplicarse a todo tipo de materiales y campos de estudio en ciencia y tecnología de materiales trabajando sobre muestras electrón-transparentes preparadas en su caso ad-hoc para este fin. El servicio realiza microscopía en modo transmisión: Imágenes en campo claro y campo oscuro, difracción de electrones de área selecta y microscopía electrónica de alta resolución así como análisis elemental de area selecta. No se dispone de modo STEM.

The transmission electron microscopy is a widely used technique for the microstructural and chemical characterization at micro and nanoscales, providing two-dimensional images of the sample texture and shape as well as grain and/or particle size, degree of homogeneity at the microscopic scale, degree of crystallinity of the sample, identification of crystalline phases, and high resolution images to identify the crystalline domains. The microscope is equipped with an EDX analyzer for compositional analysis. It can be applied to all type of materials and research topics in materials science and technology working with electron-transparent samples prepared ad-hoc for this end. The service performs transmission electron microscopy: Imaging in bright and dark field, selected area electron diffraction and high resolution electron microscopy, as well as elemental analysis of selected areas. It does not provide STEM mode.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- Microscopio Philips CM20 (200kV) con una resolución estructural de 0.14 nm entre líneas y 0.23 nm entre puntos, portamuestras de uno y dos giros y de calentamiento. Acoplados al equipo se dispone de un sistema de análisis por Energías Dispersivas de Rayos X (EDX X-Max 80T, Oxford Instruments) y una cámara CCD (Gatan) para registro de imágenes.
  Philips CM200 microscope (200kV) with a structural resolution of 0.14 nm between lines and 0.23 nm between points. Sample holders with one and two angles and heating. It is equipped with a X-ray Energy Dispersive Analyzer (EDX X-Max 80T, Oxford Instruments) and a CCD GATAN camera for image acquisitions.
- Equipamiento adicional en el “laboratorio de preparación de muestras para microscopía electrónica” (ver sección abajo)
  Additional equipment in the “electron microscopy samples preparation laboratory” (see the section below)

Responsable Científico/ Scientific Responsible: Dra. Asunción Fernández Camacho
Personal Técnico/ Technical Assistant: Dª Olga Montes Amorín (CicCartuja) y Dª María Inmaculada Roja Cejudo

LABORATORIO DE PREPARACIÓN DE MUESTRAS PARA MICROSCOPÍA ELECTRÓNICA / ELECTRON MICROSCOPY SAMPLES PREPARATION LABORATORY

El laboratorio de preparación de muestras para TEM y SEM dispone de metalizador de oro, evaporador de carbón, metalizador de Cr y carbón, cortadora de disco, pulidora, "disc-grinder", cortadora ultrasónica, pulidora cóncava (dimple) y adelgazador iónico (Fischione 1010).

The laboratory for TEM and SEM samples preparation has a gold coater, a carbon evaporator, a metallization system for Cr and carbon, a diamond wheel cutter, a grinder with disc-grinder device, an ultrasonic cutter, a concave polishing (dimple) and ion thinning (Fischione 1010).

Responsable Científico/ Scientific Responsible: Dra. Asunción Fernández Camacho
Personal Técnico/ Technical Assistants: Dª María Inmaculada Roja Cejudo, Dª Olga Montes Amorín (CicCartuja) y Dra. M. Carmen Jiménez de Haro

SERVICIO DE DIFRACCIÓN DE RAYOS X
X-RAY DIFFRACTION LABORATORY SERVICE

La difracción de rayos-X permite la identificación cualitativa y cuantitativa de sustancias cristalinas y su caracterización microestructural y textural.
El servicio dispone en la actualidad de cuatro difractómetros independientes, configurados específicamente para abordar el análisis de muestras policristalinas de muy distinta naturaleza, en lo referente a su composición, estabilidad química, cristalinidad, etc.
Asimismo, con alguno de ellos se pueden llevar a cabo, además de los análisis rutinarios (θ-2θ), otros varios mas avanzados, como pueden ser:

- Seguir las transformaciones de fase "in situ" provocadas por calentamientos en atmósfera inerte (vacio, Ar) o reactiva (H2, O2,..).
- Caracterizar materiales en la nanoescala (1-100 nm) mediante el estudio de la dispersión de rayos-X a ángulos bajos (SAXS).
- Determinar el grosor, densidad y rugosidad de películas delgadas, mediante Reflectometría de rayos-X.
- Obtener la estructura cristalina de materiales inestables a la atmósfera o muy transparentes a los rayos-X, mediante el empleo de capilares.

X-ray diffraction allows the qualitative and quantitative identification of crystalline substances and their microstructural and textural characterization.
At present, four independent diffractometers are available in this service, specifically configured to analyze the composition, chemical stability, crystallinity and many other properties in polycrystalline samples of a varied nature. Besides ordinary analyses (θ-2θ), part of the equipment can perform some advanced studies as:

- Direct monitoring of transformations undergone in materials under heating, such as phase changes, under inert o reactive atmosphere.
- To characterize materials at the nanoscale (1-100 nm) through X-ray scattering at low angles, using the SAXS technique.
- To measure some physical parameters of layers such as density, thickness and surface roughness with the reflectometry setup.
- To obtain the diffraction patterns of samples either sensitive to the atmosphere or highly transparent to X-rays (organic compounds) employing the capillary configuration.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- Difractómetro Panalytical X'PERT PRO con cargador automático de muestras
  Diffractometer PANALYTICAL X'PERT PRO with automatic sample charger
- Difractómetro PHILIPS X'PERT PRO con cámara de alta temperatura (1200 ºC) ANTON PAAR HTK 1200
  Diffractometer PHILIPS X'PERT PRO with high temperature chamber (1200ºC) ANTON PAAR HTK 1200
- Difractómetro Panalytical X'PERT PRO (reflectometría, SAXS, ángulo rasante y capilares)
  Diffractometer PANALYTICAL X'PERT PRO (reflectometry, SAXS, low angle scattering and capillary)
- Difractómetro de polvo SIEMENS D5000 DUAL (reflexión y transmisión)
  Diffractometer SIEMENS D5000 DUAL (reflection and transmission)

Responsable Científico/ Scientific Responsible: Dra. Concepción Real Pérez
Personal Técnico/Technical Assistant: D. José María Martínez Blanes

SERVICIO DE ANÁLISIS DE SUPERFICIE/ SURFACE ANALYSIS SERVICE

El Servicio de Análisis de superficie consta de un espectrómetro de Espectroscopía de Fotoelectrones de Rayos X (XPS). Este servicio está dedicado al análisis químico y electrónico de superficies sólidas. También permiten conocer la composición en profundidad (desde la superficie hacia el interior) de los sólidos.

The surface analysis service consists of an X-ray Photoelectron Spectrometer (XPS). This service is devoted to the electronic and chemical analysis of solid surfaces. It also provides information about the compositional depth profile of solids (from their surface toward their bulk).

ESPECTROSCOPÍA DE FOTOEMISIÓN DE ELECTRONES / X-RAY PHOTOELECTRON SPECTROSCOPY

Las "Espectroscopías de Fotoelectrones" (XPS/ESCA y AES) son unas poderosas técnicas de análisis cuantitativo no destructivo, sensibles exclusivamente a las primeras capas de la superficie de los sólidos (20-30 Å), lo que permite obtener información sobre las propiedades químicas, físicas y electrónicas de las mismas.
El interés técnico de esta información es enorme en campos tales como corrosión, catálisis, tratamientos de superficies, fenómenos de flotación y adherencia, segregación de fases, etc.
La característica más importante de la Espectroscopía de Fotoelectrones (XPS/ESCA) es que permitir diferenciar distintos estados de oxidación y/o situaciones del entorno (coordinación) de los átomos en las muestras sólidas analizadas. El límite de detección es del 0.5% para cada especie química.
El servicio dispone actualmente de dos instrumentos independientes.

Typically, “photoelectron spectroscopies” are a powerful set of non-destructive analysis techniques, exclusively sensitive to the more superficial few atomic layers (20-30 Å), allowing to obtain valuable information about their chemical, physical and electronic properties.
The technical interest of the resulting information is huge in fields such as catalysis, corrosion, surface treatments,, floating and adhesion phenomena, or segregation processes, among others. The most remarkable characteristic of X-Ray Photoelectron Spectroscopy (XPS/ESCA) is that it allows to discriminate, for a given element, between different oxidation states or chemical surroundings (coordination).

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

Espectrómetro de Fotoelectrones VG ESCALAB 210, compuesto de:
Photoelectron Spectrometer VG ESCALAB 210, consisting on:

- Cámara de análisis, analizador hemiesférico multicanal VG ESCALAB 210, manipulador de cuatro ejes, y fuentes de excitación de rayos X (dual, AlKα y MgKα, acromático), de luz ultravioleta y de haces de electrones, lo que permite realizar análisis superficiales mediante técnicas de XPS, UPS y REELS, así como estudios angulares.
  Analysis Chamber, equipped with a hemispheric multichannel analyser VG ESCALAB 210, a four axis manipulator, a dual X-ray source (achromatic AlKα, Mg Kα), a UV lamp, and a electron gun, allowing to perform surface analysis by XPS, UPS and REELS, including angular resolved studies.
- Dos Precámaras de tratamientos, con vacío residual de 10^{-8} y 10^{-9} mbar respectivamente, en las que es posible someter a las muestras a tratamientos diversos como: calentamientos a alta temperatura (T<800ºC) bajo atmósfera controlada, desbastado iónico con gases inertes o reactivos, exposición a plasmas, iluminación con laser, deposición de metales, óxidos y compuestos sencillos, exfoliación in situ, etc.
  Two prechambers for different treatments, with ultimate vacuum levels of 10-8 and 10-9 mbar respectively, where samples can be subjected to diverse treatments and transferred to the analysis chamber without exposure to the atmosphere. The possible treatments include heating at high temperature (< 800C) under controlled atmosphere, ion

sputtering with inert or reactive gases, exposure to plasma, laser treatments, deposition of metals, oxides or simple compounds, exfoliation, etc.

Espectrómetro de Fotoelectrones SPECS, compuesto de:
Photoelectron Spectrometer SPECS, consisting on:

- Cámara de análisis, dotada de analizador hemiesférico multicanal PHOIBOS 100, manipulador de tres ejes, y fuentes de excitación de rayos X (dual, Alkα y MgKα).
  Analysis Chamber, equipped with a hemispheric multichannel analyser PHOIBOS 100, three axis manipulator and dual X-ray source (achromatic Al Kα, Mg Kα).
- Precámara de tratamiento de alta Presión y alta Temperatura (HPHT Cell). En esta Cámara es posible someter a las muestras a tratamientos térmicos en presencia de gases hasta una presión de 20 atm y 800 ºC, tanto en estático como en dinámico (simultáneamente).
  Pre-chamber for High Pressure/High Temperature treatments (HPHT Cell).Samples can be subjected to treatments in the presence of gases up to 20 bar and 800ºC (simultaneously). These treatments can be performed either under static or flowing gas conditions. After treatments, samples can be transferred to the analysis chamber without exposure to the atmosphere.
- Una cámara de inserción rápida dotada de sistema de aparcamiento/ desgasificado, que permite evacuar las muestras a temperatura reducida (T < 150ºC). También es posible la realización de tratamientos de desbatado iónico o la incorporación de otros sistemas (iluminación con luz Uv-Vis, evaporación de metales, u otros compuestos, etc.)
  A Fast entry chamber, equipped with a parking and degassing system, allowing the samples to be evacuated at moderate temperature (T < 150ºC). It is also possible to sputter the samples under an accelerated ion beam (0.5- 5.0 kV) using inert or reactive gases. Incorporation of some other equipment (Visible light illumination, metal evaporators) is also contemplated.

Responsables Científicos/ Scientific Responsibles: Dr. Juan Pedro Espinós Manzorro y Dr. Juan Pedro Holgado Vázquez

SERVICIO DE MECANIZADO/ MECHANIZED WORKSHOP

Se trata de un servicio horizontal fundamental para el Instituto y unidades externas adscritas al mismo, puesto que, además del mantenimiento del material y equipamiento científico, permite su mejora y adaptación a los distintos experimentos en curso, y de acuerdo con las necesidades de los investigadores y/o los proyectos que se llevan a cabo.

The Machining workshop is a basic and essential service that provides technical support to the maintenance, change, fitting and development of simple scientific equipments, according with the current requirements of the experiments and projects carried out by the researchers at the ICMS.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

El servicio cuenta con herramientas manuales y herramientas eléctricas para la conformación y unión de piezas de materiales muy diversos.
Para el proceso de mecanizado por arranque y herramienta de viruta cuenta con las siguientes máquinas herramientas:
Besides other minor tools for cutting, soldering, folding, drilling, etc, this workshop is equipped with:

- Centro de mecanizado, modelo HASS TM1P
  A computer numeric control turing machine, model HASS TM1P
- Taladro vertical, modelo ERLO TSAR32
  A vertical drill, model ERLO TSAR32
- Torno paralelo semiautomático PINACHO SMART TURN180
  A couple of turning machines: models PINACHO TURN180

Responsable/ Responsible: Dra. Ana Isabel Becerro Nieto
Personal Técnico: D. Juan Carlos Sánchez Martín y D. Manuel Perea Domínguez

ACTIVIDADES DIVULGATIVAS Y FORMATIVAS

OUTREACH AND TEACHING ACTIVITIES

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Organizado por la Universidad de Sevilla
Coordinador: Dr. Joaquín Ramírez Rico
Fecha de Celebración: Curso Académico 2015-16
Créditos necesarios: 60
Dirigido a: Licenciados en Química, Física, así como, los Titulados en Ingeniería afines (Ingenieros Químicos, de Materiales, etc.)
Especialidades: Materiales para la Energía y el Medio Ambiente. Ingeniería de Superficies, Materiales Estructurales y Funcionales.
Mención de Calidad

Química del Estado Sólido (Créditos: 5) | Dra. Pilar Malet Maenner
Fisica del Estado Sólido (Créditos: 5) | Dra. Angela Gallardo López | Dra. Josefa Borrego | Dr. Manuel Jiménez Melendo
Técnicas de Caracterización de Materiales (Créditos: 8) | Dr. Julián Martínez Fernández | Dr. Juan M. Montes Martos (US)
Síntesis de Materiales y Nanoestructuras (Créditos: 7) | Dra. María Dolores Alcalá | Dra. Svetlana Ivanova | Dr. Francisco J. Gotor Martínez
Catalizadores para la Energía y el Medio Ambiente (Créditos: 5) | Dr. José Antonio Odriozola Gordón
Materiales con Funcionalidad Superficial (Créditos: 5) | Dra. Rosa María Pereñiguez Rodríguez
Recuperación y Transformación de Materiales (Créditos: 5) | Dra. Svetlana Ivanova
Procesado de Materiales Estructurales (Créditos: 5) | Dr. Alfonso Bravo León | Dr. José Antonio Rodríguez Ortíz
Comportamiento Mecánico (Créditos: 5) | Dr. Arturo Domínguez Rodríguez | Dr. Diego Gómez García
Comportamiento Magnético (Créditos: 5) Dr. Javier S. Blázquez Gámez | Dr. Victorino Franco García
Comportamiento Térmico, Dieléctrico y Óptico de Materiales (Créditos: 5) | Dr. Hernán Míguez García | Dr. Francisco Javier Romero Landa | Dra. Carmen Gallardo Cruz
Computación en Ciencia de Materiales (Créditos: 5) | Dra. Regla Ayala Espinar | Dr. Antonio Córdoba Zurita (US) | Dr. Felipe Gutiérrez Mora

Asimismo, el personal del ICMS ha impartido cursos regulares de primer y segundo ciclos de las correspondientes Licenciaturas y/o Grados en Química, Física y Farmacia e Ingeniería de Materiales y Biología en la Universidad de Sevilla.

CONFERENCIAS INVITADAS IMPARTIDAS POR PERSONAL DEL ICMS
INVITED CONFERENCES BY PERSONNEL OF THE ICMS

8 de febrero | **Catalytic Devices for Fuel Processing. Noble Metal/Ceria Catalysts for the WGS Reaction. Why Gold and Platinum Behave Differently?**
Dr. José Antonio Odriozola Gordón
Lugar: Universidad de Barcelona

24 de junio | **Solution Processed Optical Materials for Optoelectronic Devices**
Dr. Hernán R. Míguez García
Lugar: Seminarios 2016 del Departamento de Física de la Materia Condensada. Facultad de Ciencias, Universidad de Zaragoza.

4 de octubre | **Solution processed optical materials for optoelectronic devices**
Dr. Hernán R. Míguez García
Lugar: Adolphe Merkle Institute: Autumn Semester 2016. University of Fribourg (Alemania)

9 de noviembre | **Tailoring the absorption and emission properties of nanomaterials through their photonic environment**
Alberto Jiménez Solano
Lugar: Max Planck Institute for Solid State Research (Alemania)

5 de diciembre | **Optical modelling of ABX3 perovskites for solar cells with improved performance**
Miguel Anaya Martín
Lugar: Center for Excitonics, Harvard-MIT (Estados Unidos de América)

CONFERENCIAS Y SEMINARIOS IMPARTIDOS EN EL ICMS
CONFERENCES AND SEMINAR IN THE ICMS

18 de febrero | **Nanoparticles as Building Blocks for Multicomponent Materials and Films**
Prof. Dr. Markus Niederberger
Laboratory for Multifunctional Materials, ETH Zurich, Suiza

Nanostructures including nanoparticles, nanowires and nanosheets are the ideal building blocks for the bottom-up fabrication of functional materials. They offer an immense variety of interesting properties, which not only depend on the composition, but also on the crystal structure, the particle size and shape and on the surface chemistry. Accordingly, potential synthesis routes have to provide full control over all these parameters. In addition, for most applications the nanoparticles have to be assembled and processed into useful geometries, including 2-dimensional arrangements like films or 3-dimensional bodies like composites, foams or aerogels. The talk will give a short introduction to the synthesis of metal oxide nanoparticles by nonaqueous sol-gel chemistry, and it will briefly discuss the underlying chemical mechanisms leading to nanoparticle formation. Moreover, several examples will be shown, how the nanoparticles can be processed into films and nanostructured bulk materials for specific applications in gas sensing, dye adsorption, or energy storage and conversion.

21 de abril | **Electrochromics and thermochromics: Towards a new paradigm for energy efficient buildings**
Prof. Dr. Claes-Göran Granqvist
Department of Engineering Sciences, The Ångström Laboratory, Uppsala University, Uppsala, Suecia

About forty per cent of the World’s primary energy is used for heating, cooling, lighting and ventilating buildings. New nanotechnologies are able to decrease the use of energy significantly at the same time as the comfort and amenities of the building are improved. This talk surveys a number of options, mostly based on work in the speaker’s laboratory. Foci lie on windows and glass facades with electrochromic and thermochromic properties. Functional principles, thin film preparation and properties, new plasmonic nanomaterials, and technological prospects are discussed.

13 de septiembre| **2D or not 2D? Two-dimensional nanostructures for sensing and energy conversion**
Prof. Dr. Bettina V. Lotsch
Max Planck Institute for Solid State Research, Stuttgart, Alemania

A central paradigm of nanochemistry is the rational synthesis, manipulation and assembly of nanoscale building blocks into hierarchical structures with tailor-made properties. Owing to their chemical diversity and wide scope of physical properties, 2D nanostructures lend themselves particularly well as versatile building blocks in miniaturized devices and for the directed self-assembly of hybrid superlattices with engineered functionalities. Our recent progress in the synthesis and integration of novel 2D bulk and nanosheet materials will be reviewed and their emerging applications in photonic sensing, solar energy conversion and as solid electrolytes will be discussed.

18 de noviembre | **On-surface synthesis and properties characterization of novel low-dimensional materials**
Prof. Dr. Oliver Gröning
Empa, Swiss Federal Laboratories for Materials Testing and Research

In recent years on-surface chemical synthesis routes have succeeded in producing atomically precise nanostructures, whose synthesis cannot be achieved by standard wet chemical processes. This circumstance is due to a great extend to the usually insoluble nature of the products. In this respect, particular attention has been devoted to graphene derived carbon nanostructures such as graphene nanoribbons (GNR) and regular 2D carbon networks[1,2]. In our presentation we will review the recent developments in the field of on-surface chemical synthesis with a particular emphasis on the importance UHV-analytical and computational tools in understanding the physicochemical processes involved in the synthesis and in assessing the electronic properties of the produced nanostructures. The first part of the presentation will mainly touch the role of specific molecular precursors for the synthesis of graphene derived 2D and 1D nanostructures. We will discuss the electronic properties of these novel nanomaterials and their prospects to be used in future electronic devices.
In the second part the question, how the chemical synthesis can be guided by specific atomic surface structures will be addressed. In this context, we will turn our attention to the PdGa compound, which has been found to combine high selectivity and activity in acetylene semi-hydrogenation [3]. We will discuss the non-trivial atomic structure of the (111) and (-1-1-1) PdGa surface, which differ significantly in the local structure of the top most Pd atoms and therefore are model surfaces to study active site isolation and ensemble effects on catalyst selectivity [4]. We will then explore the possibilities of chiral selective adsorption and synthesis on these surfaces made possible by the intrinsic chiral nature of the P213 space group PdGa belongs to [5]. We show that the intrinsically chiral surfaces of PdGa can induce 99% chiral adsorption selectivity of prochiral 9-Ethynylphenantrene (9-EP) at room temperature. Similarly high chiral selectivity can be achieved in producing prochiral 9-EP trimers, which shows the potential of achieving highly asymmetric chemical synthesis on a high temperature stable metallic catalyst.

[1] J. Cai, et al.,Nature 466, 470 (2010)
[2] M. Bieri, et al., JACS 132 16669, (2010)
[3] M. Armbrüster, M. Kovnir, M. Behrens, D. Teschner, Y Grin, and R. Schlögl, JACS 132, 14745 (2010)

Otras Actividades / Other Activities

Feria de la Ciencia / Fair of Science

La 14ª Feria de la Ciencia (del 5 al 7 de mayo de 2016, Palacio de Congreso y Exposiciones de Sevilla, FIBES) constituye un punto de encuentro donde se desarrollaron actividades de divulgación de la Ciencia y la Tecnología, realizando demostraciones y experimentos para facilitar la comprensión de contenidos científicos. El Instituto de Ciencia de Materiales de Sevilla presentó la actividad: "Tecnología del Plasma: Aplicaciones Funcionales".
Coordinadora: T Cristina Rojas Ruiz
Participaron un total de 19 monitores

The Fair of Science (5 to 7 May 2016, in Seville) constituted a meeting point where many activities for spreading of science and technology were carried out. Demonstrations and experiments where presented to facilitate the understanding of scientific aspects. The Materials Science Institute of Seville presented the activity: "Tecnología del Plasma: Aplicaciones Funcionales"
Coordinadora: T Cristina Rojas Ruiz
Participaron un total de 19 monitores

Semana de la Ciencia y la Tecnología / Science and Technology Week

Jornadas de puertas abiertas. La Semana de la Ciencia y la Tecnología está dirigida a todos los públicos. Este evento de carácter europeo está diseñado para demostrar cómo la ciencia y la tecnología nos afectan y cómo éstas pueden ser utilizadas para mejorar nuestras vidas y el mundo que nos rodea.

Coordinación: Dra. T. Cristina Rojas Ruiz

Café con Ciencia

El café con ciencia es una actividad que acerca de forma original y atractiva la ciencia y sus protagonistas. Esta actividad de divulgación genera un punto de contacto entre profesionales de la ciencia y alumnos de secundaria, promoviendo la reflexión sobre diversos asuntos en un entorno cercano y participativo.

En esta edición se compartirán desayuno con estudiantes de bachillerato para dialogar sobre temas concretos de sus estudios y sobre sus respectivas trayectorias profesionales. Esta actividad cumple el triple objetivo de comunicar la ciencia a través de sus propios protagonistas, promover la cultura científica y fomentar vocaciones investigadoras.

Las mesas de encuentro tienen una hora de duración, y se desarrollan con grupos reducidos de quince alumnos.

Mesa: Dra. Rosalía Poyatos Galán. Tema: “¿Podemos tener cerámicas a la carta manipulando el nanomundo?”

IES Hipatia e IES Caura (35 alumnos)

PARTICIPACION EN LA NOCHE EUROPEA / European Researchers’ Night

Dos Investigadores del Instituto han participado en la European Researchers’ Night, una iniciativa promovida por la Comisión Europea en el marco del programa Horizonte 2020. El evento se celebró el 29 de septiembre de forma simultánea en trescientos cincuenta ciudades europeas.

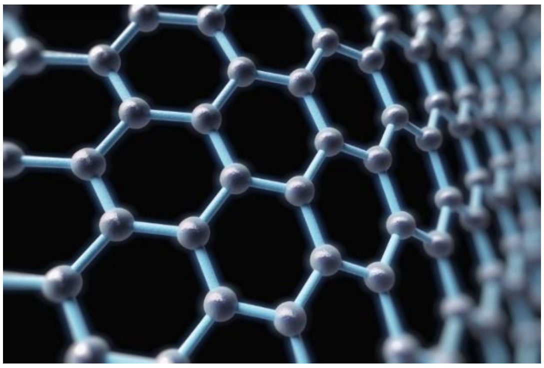
En Sevilla han unido esfuerzos el Consejo Superior de Investigaciones Científicas (CSIC), la Universidad de Sevilla (US), la Universidad Pablo de Olavide (UPO) y el Ayuntamiento de Sevilla para ofrecer un programa conjunto compuesto por talleres, microencuentros y espectáculos diseñados para buscar un acercamiento distendido entre la ciudadanía y la comunidad científica, mediante la diversión y el lenguaje divulgativo.

Este singular formato ofrece una oportunidad a los ciudadanos de acercarse a los investigadores y vencer algunos tópicos sobre la profesión científica. Y a los investigadores ofrece una oportunidad de salir de sus laboratorios y demostrar que no viven ajenos a la realidad exterior.

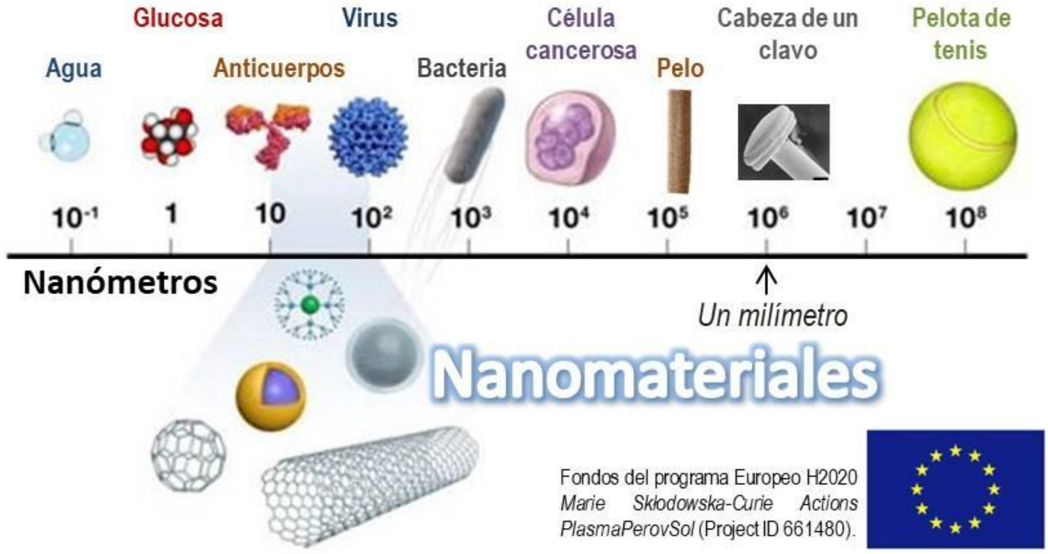
LA NOCHE EUROPEA
DE L@S INVESTIGADOR@S
MUJERES Y HOMBRES QUE HACEN CIENCIA PARA TI

Mauricio Calvo Roggiani. Cristales Fotónicos, materiales nanotecnológicos para el control de la luz

Rosalía Poyato Galán. Nanoplaquetas de grafeno: cuando el tamaño importa



Juan Ramón Sánchez Valencia. Nanomateriales, mucho más que miniaturación.



Instituto de Ciencia de Materiales de Sevilla
CSIC/US
c/ Américo Vespucio, nº 49
41092 Isla de la Cartuja. Sevilla
http://www.icms.us-csic.es
buzon@icmse.csic.es