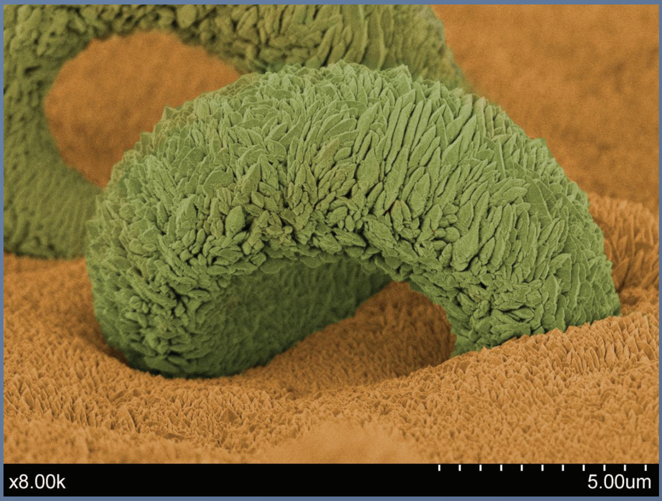
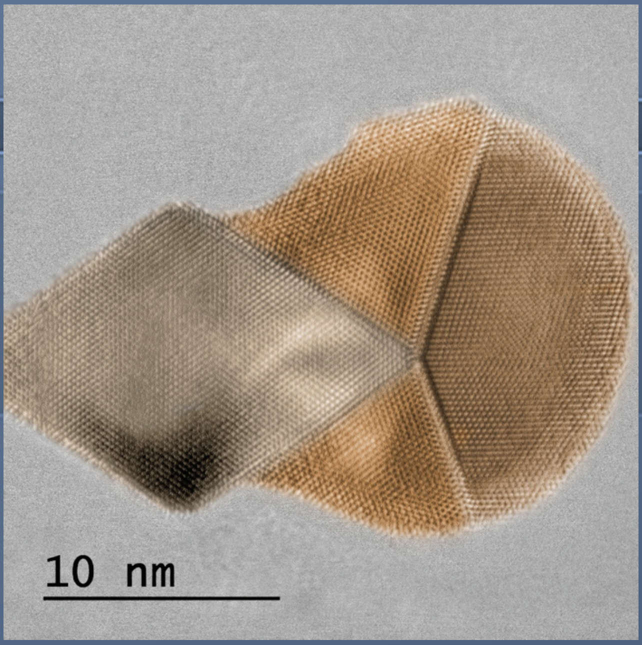
ICMS

Instituto de Ciencia de Materiales de Sevilla





Consejo
Superior de
Investigaciones
Científicas

Universidad
de Sevilla

Junta de
Andalucía

Memoria de Actividades
Annual Report

2013

Consejo Superior de Investigaciones Científicas
Universidad de Sevilla
Junta de Andalucía

INSTITUTO DE CIENCIA DE MATERIALES DE SEVILLA

Memoria de Actividades
Annual Report
2013

Centro de Investigaciones Científicas “Isla de la Cartuja” (cicCartuja)
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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)
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41092 Isla de la Cartuja, Sevilla
http://www.icms.us-csic.es

DISEÑO Y MAQUETACIÓN
José Carlos Rivero Cabello – Pedro José Sánchez Soto

FOTOGRAFÍAS DE PORTADA
“Snake on the sand” – Autor: Mariana Paladini San Martín
“Andalusian Delight” – Autor: Carlos García Negrete
Fotografías premiadas en el 2nd Al-NanoPhoto

COMISIÓN MEMORIA-ICMS
Gerardo Colón Ibáñez - Hernán R. Míguez García – Pedro José Sánchez Soto

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El ICMS en 2013
ICMS in 2013

Presentación Presentation

A través de esta Memoria 2013, 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 1985, está integrado por personal científico de la Universidad de Sevilla y el CSIC, cuenta en la actualidad con 141 personas, 48 de las cuales son científicos permanentes.

De acuerdo con la estructura establecida en el vigente Plan Estratégico 2010-2013, 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 2013, 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 1985, and integrated by scientific staff of the University of Seville and the CSIC, including today more than 141 people, 48 of which are permanent scientific staff.

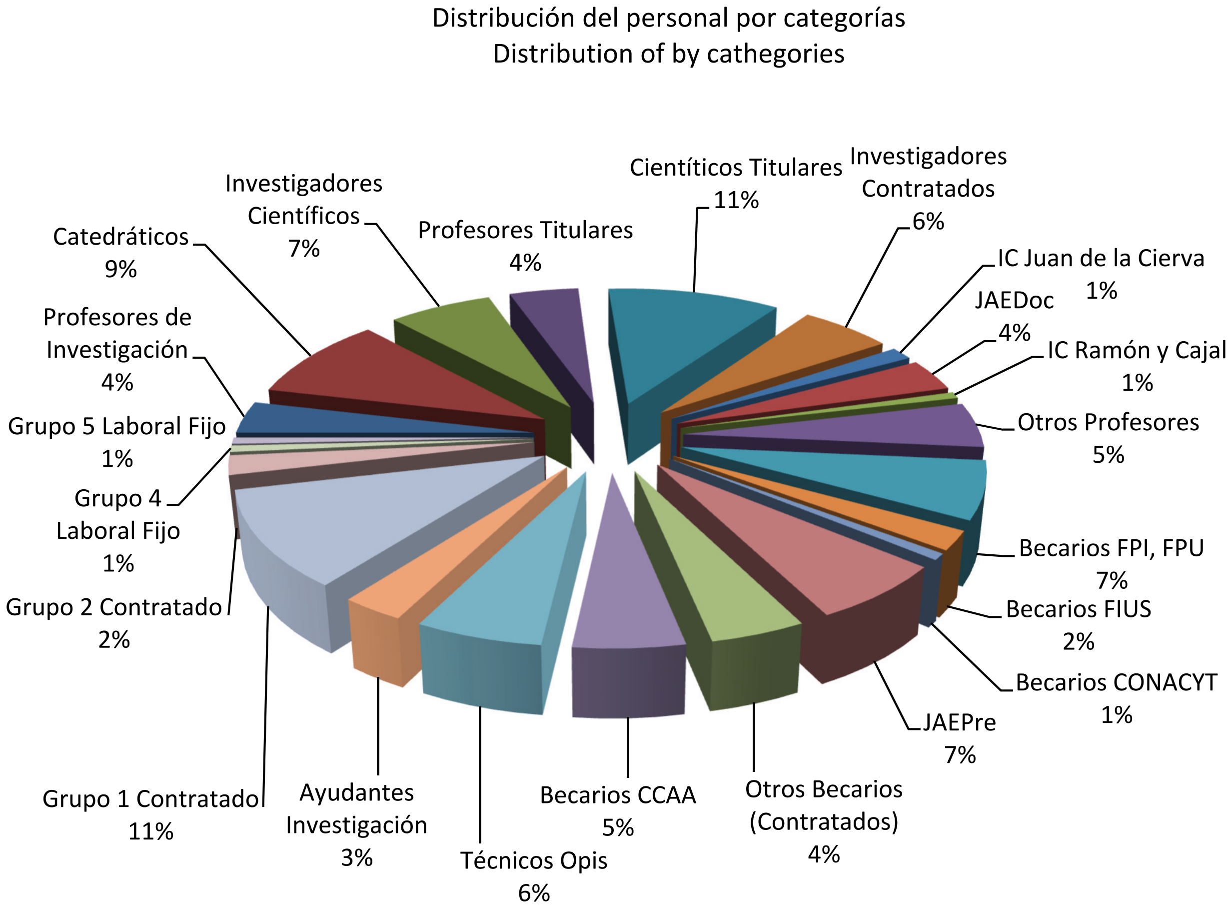
The current Strategic Plan 2010-2013 organizes our Center in 5 lines of research, which related with disciplines of solid state chemistry and physic, as well as materials engineering subjects, are intended to cover the most modern and innovative aspects of the current material science. Our basic commitment 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.

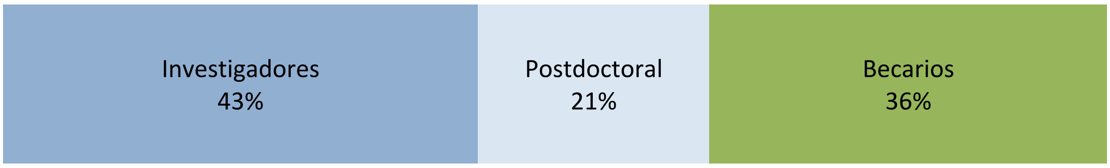
Dr. **Alfonso Caballero Martínez**
Director del Instituto de Ciencia de Materiales de Sevilla

Datos Estadísticos del ICMS

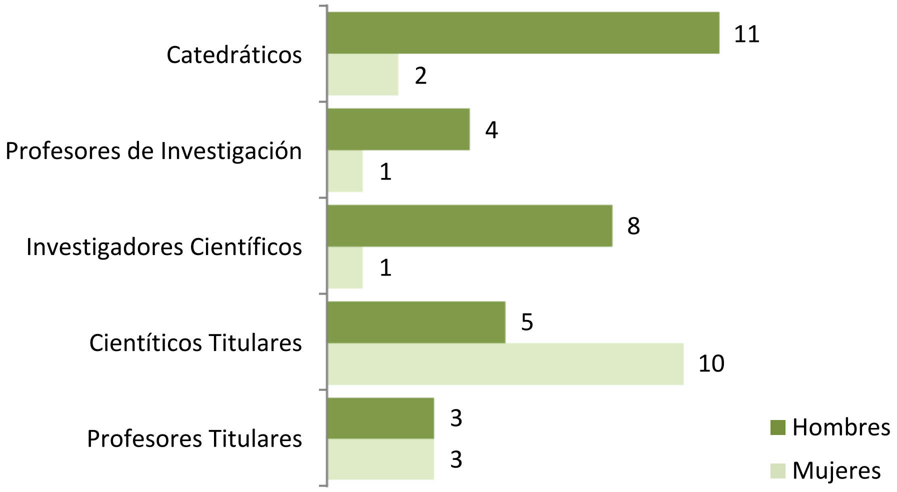
Statistical Data of ICMS

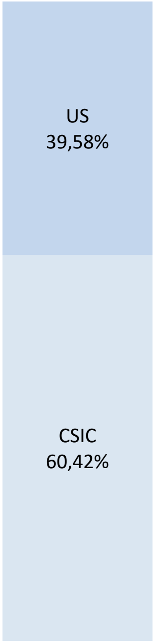
Recursos Humanos / Human Resources



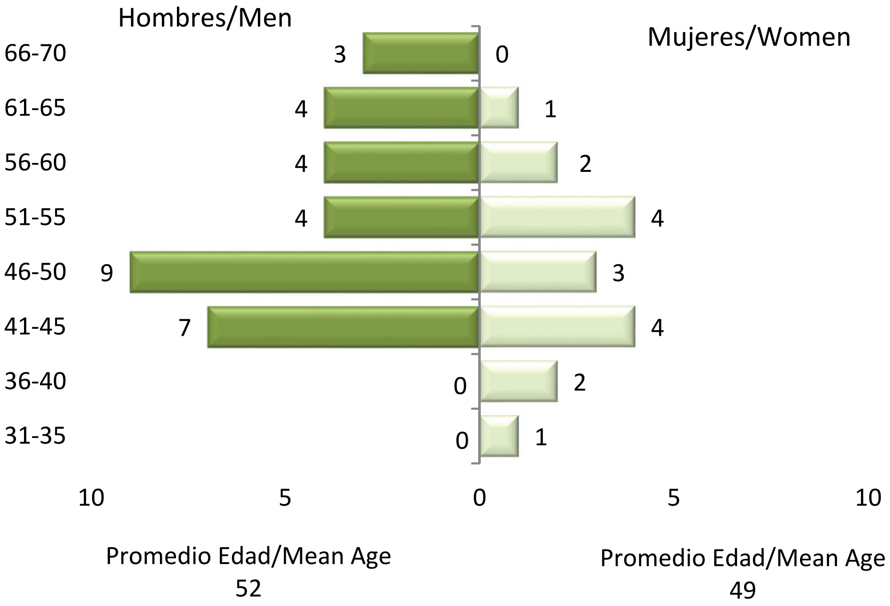


Distribución del personal Investigador por sexo
Scientific staff by sex



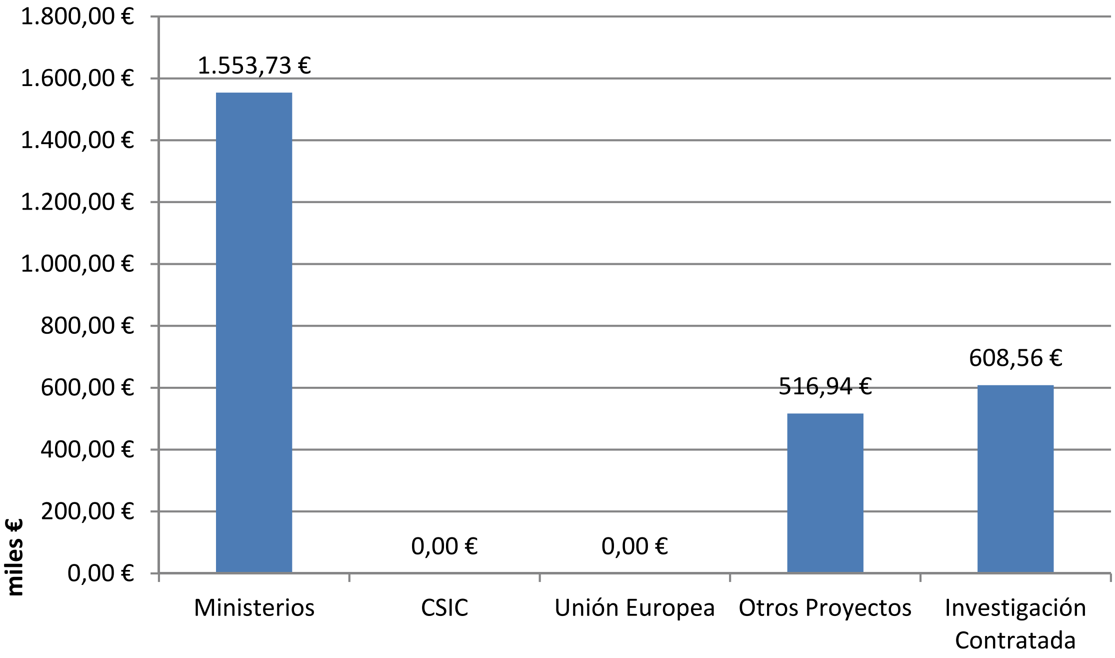


Distribución del personal investigador por edad
Scientific staff by age

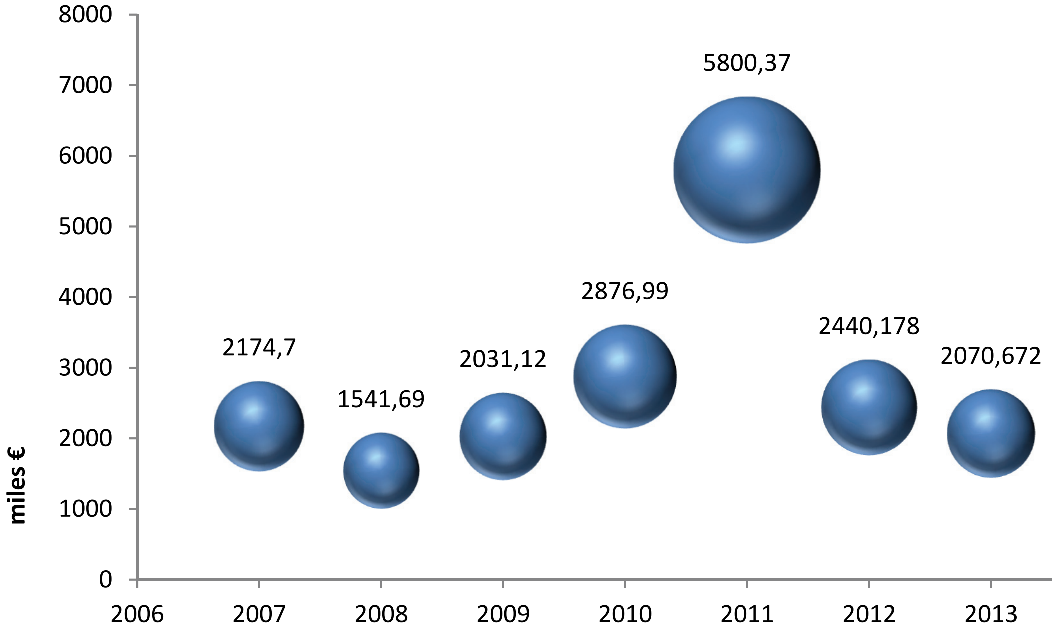


FINANCIACIÓN / FUNDING

Financiación del ICMS por Organismo Financiador 2013 (Sede-cicCartuja)
Funding of ICMS classified by source 2013 (Sede-cicCartuja)



Evolución 2007-2013 de la Financiación del ICMS (PCO)
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 (*)
CHEMICAL SOCIETY REVIEWS	1	30,43
ENERGY & ENVIRONMENTAL SCIENCE	1	15,49
ADVANCED MATERIALS	1	15,41
JOURNAL OF THE AMERICAN CHEMICAL SOCIETY	1	11,44
ADVANCED FUNCTIONAL MATERIALS	3	10,44
CHEMISTRY OF MATERIALS	1	8,54
PHYSICAL REVIEW LETTERS	1	7,73
ACS CATALYSIS	1	7,57
NANOSCALE	1	6,74
INTERNATIONAL MATERIALS REVIEWS	1	6,55
CARBON	1	6,16
JOURNAL OF CATALYSIS	1	6,07
APPLIED CATALYSIS B-ENVIRONMENTAL	3	6,01
ACS APPLIED MATERIALS & INTERFACES	2	5,90
CHEMISTRY-A EUROPEAN JOURNAL	1	5,70
APPLIED ENERGY	1	5,26
JOURNAL OF PHYSICAL CHEMISTRY C	3	4,84
INORGANIC CHEMISTRY	2	4,79
CATALYSIS SCIENCE & TECHNOLOGY	1	4,76
CRYSTAL GROWTH & DESIGN	1	4,56
LANGMUIR	2	4,38
JOURNAL OF HAZARDOUS MATERIALS	1	4,33
PHYSICAL CHEMISTRY CHEMICAL PHYSICS	3	4,20
DALTON TRANSACTIONS	1	4,10
CHEMICAL ENGINEERING JOURNAL	1	4,06
ENVIRONMENTAL POLLUTION	1	3,90
RSC ADVANCES	2	3,71
JOURNAL OF MOLECULAR CATALYSIS A-CHEMICAL	1	3,68
APPLIED CATALYSIS A-GENERAL	1	3,67
NANOTECHNOLOGY	4	3,67
CHEMICAL GEOLOGY	1	3,48
CHEMPHYSCHEM	1	3,36

REVISTA JOURNAL	ARTÍCULOS PAPERS	FACTOR DE IMPACTO IMPACT FACTOR (*)
CATALYSIS TODAY	5	3,31
FOOD CHEMISTRY	1	3,26
COMPOSITES PART A-APPLIED SCIENCE AND MANUFACTURING	1	3,01
SCRIPTA MATERIALIA	2	2,97
EUROPEAN JOURNAL OF INORGANIC CHEMISTRY	2	2,97
INTERNATIONAL JOURNAL OF HYDROGEN ENERGY	3	2,93
RESPIRATION	1	2,92
JOURNAL OF THE ELECTROCHEMICAL SOCIETY	1	2,86
JOURNAL OF BIOMEDICAL MATERIALS RESEARCH PART A	1	2,84
JOURNAL OF BIOMATERIALS APPLICATIONS	1	2,76
ENERGY & FUELS	1	2,73
JOURNAL OF ALLOYS AND COMPOUNDS	3	2,73
APPLIED CLAY SCIENCE	1	2,70
RESOURCES CONSERVATION AND RECYCLING	1	2,69
PHOTOCHEMISTRY AND PHOTOBIOLOGY	1	2,68
APPLIED GEOGRAPHY	1	2,65
RAPID COMMUNICATIONS IN MASS SPECTROMETRY	1	2,64
POLYMER DEGRADATION AND STABILITY	1	2,63
CHEMICAL ENGINEERING SCIENCE	1	2,61
APPLIED SURFACE SCIENCE	4	2,54
JOURNAL OF PHYSICS D-APPLIED PHYSICS	1	2,52
JOURNAL OF THE AMERICAN CERAMIC SOCIETY	3	2,43
COLLOIDS AND SURFACES A-PHYSICOCHEMICAL AND ENGINEERING ASPECTS	1	2,35
JOURNAL OF THE EUROPEAN CERAMIC SOCIETY	3	2,31
JOURNAL OF MATERIALS SCIENCE	3	2,31
JOURNAL OF PHOTOCHEMISTRY AND PHOTOBIOLOGY A-CHEMISTRY	1	2,29
JOURNAL OF NANOPARTICLE RESEARCH	2	2,28
POWDER TECHNOLOGY	1	2,27
CONSTRUCTION AND BUILDING MATERIALS	1	2,27
JOURNAL OF THERMAL ANALYSIS AND CALORIMETRY	3	2,21
SURFACE & COATINGS TECHNOLOGY	1	2,20
JOURNAL OF APPLIED PHYSICS	2	2,19
JOURNAL OF ARCHAEOLOGICAL SCIENCE	1	2,14

REVISTA JOURNAL	ARTÍCULOS PAPERS	FACTOR DE IMPACTO IMPACT FACTOR (*)
MATERIALS CHEMISTRY AND PHYSICS	2	2,13
SOLID STATE IONICS	1	2,11
THERMOCHIMICA ACTA	1	2,11
CHEMICAL PHYSICS LETTERS	1	2,09
AMERICAN MINERALOGIST	2	2,06
JOURNAL OF OPTICS	1	2,01
SURFACE SCIENCE	2	1,87
THIN SOLID FILMS	3	1,87
WEAR	1	1,86
GOLD BULLETIN	1	1,84
POLYMER TESTING	3	1,82
INTERNATIONAL JOURNAL OF REFRACTORY METALS & HARD MATERIALS	1	1,76
MATERIALS SCIENCE IN SEMICONDUCTOR PROCESSING	1	1,76
CHEMISTRY CENTRAL JOURNAL	1	1,66
PHYSICA STATUS SOLIDI A-APPLICATIONS AND MATERIALS SCIENCE	1	1,53
NANOSCIENCE AND NANOTECHNOLOGY LETTERS	1	1,44
COLD REGIONS SCIENCE AND TECHNOLOGY	1	1,44
ADVANCES IN APPLIED CERAMICS	1	1,11
JOURNAL OF ADVANCED OXIDATION TECHNOLOGIES	1	1,11
DESALINATION AND WATER TREATMENT	1	0,99
PHYSICS OF THE SOLID STATE	3	0,78
BOLETIN DE LA SOCIEDAD ESPAÑOLA DE CERAMICA Y VIDRIO	3	0,34
JOURNAL OF MATERIALS CHEMISTRY C	1	0,00
Total	134	3,69

(*) Factor de Impacto correspondiente al año 2013
Journal Citation Reports of 2013

COMPOSICIÓN Y ESTRUCTURA
STRUCTURE AND ORGANISATION

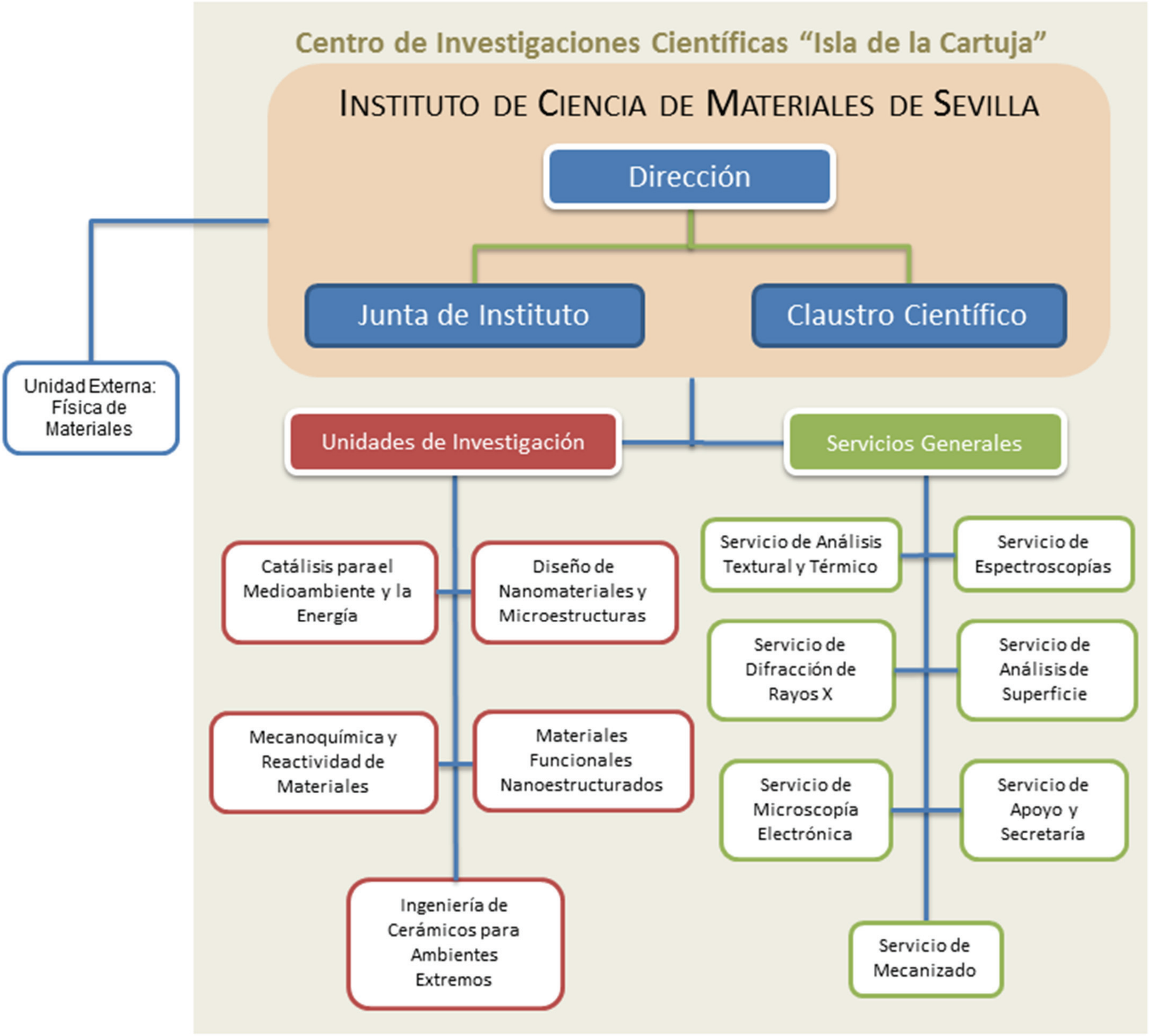


EL INSTITUTO / THE INSTITUTE

El Instituto de Ciencia de Materiales (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 (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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TAILORED NANOMATERIALS AND MICROSTRUCTURE

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UNIDADES DE INVESTIGACIÓN

RESEARCH UNITS

CATÁLISIS PARA EL MEDIOAMBIENTE Y LA ENERGÍA
CATALYSIS FOR ENVIRONNMENT AND ENERGY

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Dr. José Antonio Odriozola Gordón

Investigadores Científicos

Dr. Miguel Angel Centeno Gallego
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Científicos Titulares

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

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Dra. Anna Dimitrova Penkova
Dra. Fátima Ternero Fernández

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Lda. Sandra Palma del Valle
Ldo. Alberto Rodríguez Gómez
Lda. Miriam González Castaño
Ldo. Antonio Martínez Pascual
Ldo. Sebastián Murcia López
Ldo. Sergio Alberto Obregón Alfaro
Ldo. Tomás Ramírez Reina

PROYECTOS DE INVESTIGACIÓN / RESEARCH PROJECTS

Unión Europea
Fondo Europeo de Desarrollo Regional
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MINISTERIO DE ECONOMÍA Y COMPETITIVIDAD

Aprovechamiento de gas no convencional: Reactores de microcanales en GTL Valorization of Non-conventional gas: microchannel reactors in GTL

Código/Code:	ENE2012-37431-C03-01
Periodo/Period:	01-01-2013 / 31-12-2015
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	234.000 €
Investigador responsable/Research head:	José Antonio Odriozola Gordón
Componentes/Research group:	Svetlana Ivanova, Anna Dimitrova Penkova, Tomás Ramírez Reina, Sandra Palma del Valle, Ara Muñoz Murillo, María Isabel Domínguez Leal, Francisca Romero Sarria

RESUMEN / ABSTRACT

Existen en la actualidad fuentes de gas que podríamos agrupar bajo el calificativo de no-convencional que incluyen el que se encuentra confinado en formaciones geológicas de baja permeabilidad, el gas asociado al crudo, los pequeños yacimientos en lugares remotos, el biogas producido en la digestión anaerobia de residuos y los denominados product gas generados en la gasificación de biomasa y en la combustión de alquitran. La composición de todas estas fuentes de gas es similar estando constituidas por una mezcla de metano y dióxido de carbono con cantidades inferiores de otros gases permanentes. La concentración de CO2 puede llegar a ser de hasta el 40% en volumen como ocurre con el gas asociado de algunos campos off-shore y el biogas producido por fermentación de residuos agrícolas. La valorización de este gas mediante el proceso GTL (Gas to Liquid) es una alternativa cuando su localización remota o distante de los gaseoductos no permite ser agregado a las fuentes convencionales o no puede ser consumido in situ, ya que los combustibles líquidos son más fáciles de almacenar y transportar y tienen aplicación directa en el transporte.

La tecnología GTL convencional no es aplicable ya que su viabilidad económica exige instalaciones y suministros a una escala elevadísima. Por ello, se está desarrollando esa misma tecnología en reactores de microcanales de pared catalítica que consiguen incrementar de forma notable el rendimiento de las unidades de producción de gas de síntesis y síntesis de Fischer-Tropsch (SFT), al poder trabajar con elevadas velocidades espaciales, mejorando el control de la temperatura y con ello de la selectividad y la seguridad del proceso; además, la naturaleza modular basada en la replicación de unidades simplifica de forma considerable el escalado del proceso, adaptándose bien a unidades de producción de gas no convencional que, por lo general, no son grandes.

En el presente proyecto se pretende desarrollar la tecnología de microcanales para el proceso GTL utilizando mezclas metano-dióxido de carbono para simular las fuentes de gas no convencional. Los estudios que hemos venido realizando sobre reactores de microcanales de-

berán ampliarse a condiciones de presión y temperatura más drásticas, lo que debe permitir validar y mejorar la selección de materiales para la construcción y las técnicas de unión.

La aplicación de esta tecnología requiere el desarrollo de nuevos catalizadores activos, selectivos y estables que se adapten a los procesos GTL en reactores de microcanales. Se diseñarán catalizadores para el reformado al vapor, el reformado seco y la oxidación parcial de metano para la producción de gas de síntesis, así como catalizadores SFT. Se construirán reactores de microcanales para el ensayo de dichos catalizadores, se obtendrán las ecuaciones cinéticas de los catalizadores seleccionados y se modelarán y simularán los reactores construidos.

Apart from the large reserves, natural gas is present in a wide variety of sources that can be grouped as non-conventional gas, including non-conventional natural gas confined in low-permeability geological deposits, associated gas, biogas produced by anaerobic digestion of residues and product gas a result of biomass and tar gasification. Most of them are, in general, far from marketplaces and transport infrastructures, present in small or medium fields that does not allow large-scale GTL plants and, as in the associated gas in oil fields contribute to increase GHG. These gases have a similar composition, they mainly contain methane and carbon dioxide, the later may reach 40% by volume as in some off-shore oild fields and biogas produced by digestion of crop residues. Recent trends in the use of syngas are dominated by the conversion of inexpensive remote natural gas into liquid fuels (“gas to liquids” or GTL) forecasts the use of non-conventional gas in compact syngas units for GTL processes resulting in liquid fuels of easier storage and transportation having direct aplication for transport.

GTL technology developed for microchannel reactors notably increases the production yield of syngas and Fischer-Tropsch synthesis (FTS) units on running the reaction under high space velocities and improving temperature control, therefore enhancing selectivity and process safety. Modularity, which is based on unit replication, simplifies the scaling-up and allows an easier adjust to small and medium size production units.

In this Project, we aim at developing microchannel technologies for GTL using CH4-CO2 mixtures that simulate non-conventional gas resources. Our previous studies on microchannel reactor technology will be put forward to adapt to the elevated temperatures and pressures required for the GTL process. This will allow validation of the bonding techniques as well as improve materials selection to fit these drastic requirements.

New catalysts adapted to the GTL process in microchannels will be developed for steam and dry reforming of methane as well as for the partial oxidation of methane and the FTS. These catalysts must be active, selective and stable under reaction conditions and will be tested in powder, in structured form (micromonoliths) and in microchannels units. For them kinetic equations will be developed and the built microchannel reactor will be modeled and simulated.

Desarrollo de sistemas catalíticos nanoestructurados preparados mediante sol-gel y de deposición fotoquímica para aplicaciones energéticas y medioambientales (NanoFotoCat) Development of nanostructured catalyticsystems prepared by sol-gel and fotoassisted deposition (PAD) methods for energy and environmental applications

Código/Code: ENE2011-24412
Periodo/Period: 01-01-2012 / 31-12-2014
Organismo Financiador/Financial source: Ministerio de Economía y Competitividad
Importe total/Total amount: 102.850 €

Investigador responsable/Research head: Alfonso Caballero Martínez
Componentes/Research group: Gerardo Colón Ibáñez, Juan Pedro Holgado Vázquez, Sergio Obregón Alfaro, Rosa María Pereñíguez Rodríguez, Fátima Ternero Fdez.

RESUMEN / ABSTRACT

En el presente proyecto se plantea el desarrollo de una serie de catalizadores nanoestructurados basados en metales de transición tales como Ni, Cu, Au o Pd, y depositados en soportes activos (TiO_2, CeO_2, WO_3, Fe_2O_3 y soportesmesoporosos como SBA-15 dopado con titania y ceria). Se utilizarán métodos de preparación convencionales (impregnación, deposición-precipitación, etc.), junto con procedimientos de síntesis de desarrollo más reciente, como métodos sol-gel y, muy especialmente, el denominado Fotodeposición Asistida Fotoquímicamente (Photochemical Assisted Deposition, PAD). De esta forma, esperamos controlar de manera rigurosa a la escala nanométrica tanto el tamaño de la partícula metálica y/o bimetálica, como la interacción metal-soporte. En el caso concreto del método PAD, uno de los objetivos principales del proyecto es el estudio y la optimización de las variables del proceso de deposición de manera que, además de controlar el tamaño de la partícula metálica desde diámetros en torno a 15nm hasta sistemas atómicamente dispersos sobre soportes activos como ceria o titania, nos permita diseñar la distribución de los metales en la partícula metálica, haciendo uso de procesos controlados de fotodeposición consecutivos y/o simultáneos de los metales. Esta metodología debe permitir la obtención de distribuciones metálicas de tipo core-shell o aleadas, lo que como es conocido, puede llegar a afectar de manera fundamental a las prestaciones catalíticas. Estas prestaciones serán comprobadas en diferentes reacciones de interés energético y/o medioambiental, tanto en fase gas como en fase líquida. Así, los sistemas basados en niquel y oro se utilizarán en las reacciones de reformado húmedo y seco de metano (Steam and Dry Methane Reforming, SRM/DRM) y la oxidación selectiva de CO (Preferential Oxidation of CO, PROX), respectivamente. Los sistemas mono y bimetálicos basados en paladio y paladio-oro serán utilizados para la optimización de la reacción de síntesis directa de agua oxigenada a partir de hidrógeno y oxígeno, realizada en fase líquida a alta presión. La correlación entre el estado físico-químico y la reactividad de los sistemas catalíticos nos permitirá aclarar aspectos fundamentales de los mecanismos de las reacciones heterogéneas propuestas.

In the present project we propose the development of a series of nanostructured catalysts based on transition metals such as Ni, Cu, Au or Pd deposited in active supports (TiO_2, CeO_2, WO_3, Fe_2O_3 and mesoporous supportslike SBA-15 doped with titania and ceria). Conventional methods of preparation will be used (impregnation, deposition-precipitation, etc.), along with procedures of synthesis of more recent development, like sol-gel and, very specially, the denominated Photochemical Assisted Deposition (PAD). In this way, we expect to control at the nanometric scale the size of the metallic and/or bimetallic particle, along with the interaction between the metal and support surface. In the case of the PAD method, one of the primary targets of the project is the study and optimization of the different parameters affecting the deposition process. So that, besides the control of the metallic particle size from diameters around 15nm to atomic dispersed systems on active supports like ceria or titania, it would allow us to design the distribution of metals in bimetallic particles, making use of consecutive and/or simultaneous controlled processes of fotodeposition of metals. Using this methodology, we will try to obtain metallic distributions of different kinds: core-shell, alloys, etc., which as it is well-known, can strongly affect the catalytic performances. These benefits will be verified in different catalytic reactions of energetic and/or environmental interest, both in gas and liquid phase. Thus, the systems based on nickel and gold will be used in the steam and dry reforming reactions of methane and the selective oxidation of CO (Preferential Oxidation of CO, PROX), respectively. The bimetallic catalytic systems based on palladium and palladium-gold will be used for the optimization of the reaction of direct synthesis of hydrogen peroxide from hydrogen and oxygen, made in liquid phase at high pressure. The correlation between the physical-chemistry state and the reactivity of the catalytic systems will allow us to clarify fundamental aspects of the mechanisms of the proposed heterogeneous reactions.

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Desarrollo de nuevos procesos industriales basados en sistemas catalíticos para la obtención sostenible de ingredientes base en fragancias y aromas Development of new industrial processes based on catalytic systems for Sustainable production of base compounds of fragrances and aromas

Código/Code: IPT-2011-1553-420000
Periodo/Period: 04-05-2011 / 31-12-2014
Organismo Financiador/Financial source: Programa INNPACTO. Ministerio de Economía y Competitividad
Importe total/Total amount: 183.423 €
Investigador responsable/Research head: Juan Pedro Holgado Vázquez
Componentes/Research group: Alfonso Caballero Martínez, Víctor Manuel González de la Cruz, Rosa Pereñíguez Rodríguez, Gerardo Colón Ibáñez

RESUMEN / ABSTRACT

En la actualidad, los procesos industriales utilizados para la transformación de muchos compuestos utilizados en el campo de las fragancias y los aromas ofrecen bajos rendimientos y generan una enorme cantidad de residuos peligrosos, que requieren varias etapas de segregación y tratamiento de los mismos durante el proceso. La mayor parte de estos procesos se basan en reacciones de oxidación/reducción de compuestos estequiometricos, o están basados en sistemas de catálisis homogénea, los cuales presentan inconvenientes asociados con la corrosión, la recuperación del catalizador de la mezcla de reacción y su regeneración para su posible reutilización. En este contexto “eco-amigable”, existe un interés creciente para el uso de oxidantes menos contaminantes, tales como el peróxido de hidrógeno o el oxígeno molecular, y la integración de dichos oxidantes en sistemas de catálisis heterogénea. Obviamente uno de los mayores retos que presentan las reacciones basadas en sistemas catalíticos es lograr el máximo rendimiento (producto de conversión por selectividad) con objeto de reducir el consumo de reactivos (materias primas), y minimizar los procesos de separación y eliminación de subproductos no deseados fruto de la ineficacia del proceso. En este tipo de reacciones (con compuestos principalmente orgánicos, muchos de origen natural), no es, en general, difícil obtener una alta conversión, pero dado que estos compuestos presentan multiples funcionalidades y/o puntos susceptibles de ser oxidados, el reto se centra en la obtención de una alta selectividad, generalmente incluso a nivel enantiomérico.

En el marco del proyecto, se han seleccionado procesos de interés en la industria cosmética y alimentaria, con objeto de conseguir el desarrollo a escala industrial de procesos basados en sistemas de catálisis heterogénea para la obtención de compuestos intermedios de gran valor añadido en el mercado de los perfumes y aromas, como son, entre otros, el proceso de obtención de la l-carvona a partir de la oxidación catalítica del d-limoneno.

Nowadays, most of the industrial processes used for transformations of many compounds used in the field of fragrances and aromas have low yields, and generate a lot of environmentally noxious products, being necessary to accomplish several stages of segregation and treatment during the process of production of these chemicals. Most of these processes are done by reduction or oxidation reactions using stoichiometric compounds, or are based in homogeneous catalysis, that present associated hitches associated with corrosion, recovery of the catalysts from reaction media and its regeneration for its possible recycle. In this “environmentally friendly” context, there is a growing interest in the use of oxidants less contaminants, such as molecular oxygen or hydrogen peroxide, and the integration of these oxidants into heterogeneous catalysis processes. Obviously, one of the big challenges for catalytic systems is to maximize the yield (conversion times selectivity), in order to reduce the consumption of reactants (raw material), and minimize the separation and elimination of undesired sub-products obtained as consequence of process inefficacy. In these type of reactions (with mainly organic products, many from natural sources), it is not, as a general rule, difficult to obtain a high conversion, but as the starting materials present many functionalities and/or points susceptible to be oxidized, the main challenge is to obtain a (very) high selectivity, in many cases even at enantiomer level.

In this project, we have selected processes and reactions with a direct interest in the food and cosmetic industry, with the scope to develop processes, at industrial scale, based on heterogeneous catalysts to obtain compounds with high added value in the aromas and fragances fields, such as the production of l-carvone from catalytic oxidation of d-limonene.

Nuevos fotocatalizadores basados en compuestos de Bi^{3+} altamente fotoactivos en el visible New Bi^{3+} based photocatalysts highly active in the visible

Código/Code:	P09-FQM-4570 (Proyecto de Excelencia)
Periodo/Period:	11-03-2011 / 31-03- 2015
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	82.000 €
Investigador responsable/Research head:	Gerardo Colón Ibáñez
Componentes/Research group:	Carmen Hidalgo López, J. Antonio Navío Santos, Manuel Macías Azaña, Sebastián Murcia

RESUMEN / ABSTRACT

El objetivo principal de este proyecto es el desarrollo de una nueva generación de materiales nanoestructurados alternativos al TiO_2 que presenten una alta fotoactividad en la región del visible y que puedan ser utilizados de forma competitiva y eficiente en procesos de tratamiento de efluentes líquidos y gaseosos mediante el aprovechamiento de la luz solar. El presente proyecto pretende desarrollar nuevos sistemas de nanocatalizadores heterogéneos basados en Bi^{3+} (Bi_2WO_4, Bi_2MoO_6, $BiVO_4$, Bi_3O_4Cl, $CaBi_2O_4$, $PbBi_2Nb_2O_9$,…) que presenten unas propiedades optoelectrónicas adecuadas para el aprovechamiento de la energía solar en el rango del visible (Fotocatálisis Solar) y que al mismo tiempo exhiban unas propiedades fisicoquímicas mejoradas que optimicen el proceso fotocatalítico desde el punto de vista de difusión y transferencia de portadores de carga fotogenerados.

The main objective of this project is the development of a new generation of nanostructured materials alternative to TiO_2 with high photoactivity in the visible region that could be efficiently used in liquid or gaseous effluent treatment. The present project intent to develop new heterogeneous nanocatalytic systems based on Bi^{3+} (Bi_2WO_4, Bi_2MoO_6, $BiVO_4$, Bi_3O_4Cl, $CaBi_2O_4$, $PbBi_2Nb_2O_9$,…) exhibiting appropriated optoelectronic properties for the solar light use in the visible range (Solar Photocatalysis). Moreover, from the point of view of the photoinduced charge carriers diffusion and transfer, the improved physicochemical properties would optimize the photocatalytic process.

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 / 01-02- 2016
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.

Convenios y Contratos / Contracts and Agreements

Protocol for XPS analysis of different samples form Group of Inorganic Chemistry and Catalysis

Código/Code:	20132331
Periodo/Period:	22-03-2013 / 22-05-2015
Organismo Financiador/Financial source:	University Utrecht
Importe total/Total amount:	3.000 €
Investigador responsable/Research head:	Juan Pedro Holgado Vázquez

Desarrollo de sistemas catalíticos compactos para aplicaciones energéticas

Código/Code:	1211/0017
Periodo/Period:	25-04-2011 / 10-12-2013
Organismo Financiador/Financial source:	Técnicas Reunidas, SA
Importe total/Total amount:	415.041,40 €
Investigador responsable/Research head:	José Antonio Odriozola Gordón

Patentes / Patents

Método para la fabricación pulvimetalúrgica de imanes
Inventores: Fátima Ternero Fernández
Tipo de Patente: Internacional
Número de presentación: 201400015
Solicitud número PCT: PCT/ES2012/070717
Fecha Solicitud: 30 Diciembre 2013
Entidad/es Titular/es: CSIC y Universidad de Huelva

Material heteroestructurado de $BiVO_4$ dopado con Er+, Procedimiento de obtención y sus aplicaciones
Inventores: Sergio Obregón y Gerardo Colón
Tipo de Patente: Nacional
Número de Solicitud: 201330539
Fecha Solicitud: 16 Abril 2013
Entidad/es Titular/es: CSIC

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 Palmas de Gran Canaria es el Prof. Jesús Pérez Peña, Catedrático de la Universidad de las Palmas de Gran Canaria, y por parte del C.S.I.C en el Instituto, es el Dr. José Antonio Navío Santos, Profesor Titular de la Universidad de Sevilla.

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 Electrochemistry Applied to the Environment", of the University of Las Palmas de Gran Canaria through the Material Science Institute of Seville.

The person in charge of this Associated Unit is Prof. Dr. Jesús Pérez Peña 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

Promotional effect of the base metal on bimetallic Au-Ni/CeO2 catalysts prepared from core-shell nanoparticles
Holgado, JP; Ternero, F; Gonzalez-delaCruz, VM; Caballero, A
ACS Catalysis, **3** (2013) 2169-2180
Septiembre, 2013 | **DOI**: 10.1021/cs400293b

A set of three catalysts (a Au–Ni bimetallic and their corresponding Au and Ni monometallics) has been prepared by impregnation of previously prepared suspensions of monodisperse metallic particles to ensure the precise control of their physicochemical characteristics (size and composition). The Au–Ni/CeO_2 bimetallic catalysts present better reactivity toward CO oxidation than monometallic Au/CeO_2 and Ni/CeO_2 prepared under identical conditions. “operando-like” characterization of Ni and Au atoms into the bimetallic particles using, among other techniques, ambient-pressure photoelectron spectroscopy and diffuse reflectance infrared Fourier transform spectroscopy has allowed us to determine that under oxidative conditions the samples present a Au@NiO core–shell distribution, where Ni surface atoms are affected by an electronic effect from inner Au atoms.

A single-source route to bulk samples of C_3N and the co-evolution of graphitic carbon microspheres
King, TC; Matthews, PD; Holgado, JP; Jefferson, DA; Lambert, RM; Alavi, A; Wright, DS
Carbon, **64** (2013) 6-10
Noviembre, 2013 | **DOI**: 10.1016/j.carbon.2013.04.043

The thermolysis of commercially available m-phenylenediamine (1,3-(NH2)2C6H4) at 800 °C under a static vacuum in a sealed quartz tube provides the first bulk synthesis of C3N, whose properties have only been predicted theoretically previously. Hollow carbon microspheres (CMSs) which do not contain significant nitrogen doping (1–3 μm diameter) are co-produced in the reaction and readily separated from the C3N flakes. The separate C3N flakes and CMSs have been characterized by electron microscopy, X-ray spectroscopy and X-ray diffraction. These studies show that the samples of C3N and CMSs both possess multi-layered turbostratic graphitic structures. A new mechanism for the template-free assembly of CMSs is proposed on the basis of electron microscopy that involves bubble evolution from a static carbonized layer.

High-performance Er^{3+}–TiO_2 system: Dual up-conversion and electronic role of the lanthanide
Obregon, S; Kubacka, A; Fernandez-Garcia, M; Colon, G
Journal of Catalysis, **299** (2013) 298-306
Marzo, 2013 | **DOI**: 10.1016/j.jcat.2012.12.021

Erbium-doped TiO_2 materials are synthesized by means of a surfactant-free hydrothermal method having good photoactivities for the liquid-phase degradation of phenol and MB and the gas phase of toluene. From the structural and morphological characterization, it has been stated that the presence of Er3+ induces a progressive anatase cell expansion due to its incorporation in the TiO_2 lattice. The best photocatalytic performance was attained for the samples with 2 at% of Er^{3+} irrespective of the chemical degradation reaction essayed. From activity and optical studies under different irradiation excitation conditions, a dual-type mechanism is proposed to be at the origin of the photocatalytic activity enhancement. On one hand, the improvement observed under UV irradiation occurs by the effective charge separation promoted by Er^{3+} species which would act as electron scavenger. Besides, the up-conversion luminescence process of Er^{3+} allows profiting the NIR range of the lamp and transferring energy in the UV range to the TiO_2. The dual action of Er ions located at anatase networks will open up a wide roadway for the developing of an integral solar active photocatalyst.

Erbium doped TiO_2–Bi_2WO_6 heterostructure with improved photocatalytic activity under sun-like irradiation

Obregon, S; Colon, G

Applied Catalysis B: Environmental, **140-141** (2013) 299-305

Agosto, 2013 | **DOI**: 10.1016/j.apcatb.2013.04.014

Erbium doped TiO_2–Bi_2WO_6 have been synthesized by means of a surfactant free hydrothermal method having good photoactivities under sun-like excitation for the degradation of Rhodamine B. From the structural and morphological characterization it has been stated that the presence of Er^{3+} induces a progressive russelite cell contraction due to its incorporation in the Bi_2WO_6 lattice in substitutional sites. The best photocatalytic performance was attained for the samples with 1 at% of Er. From the study of the photocatalytic activity under different irradiation conditions it can be inferred that Er^{3+} presence induces a significant improvement of the photoactivity in the UV range. The evolution of band-gap values seems to be similarly related with the reaction rate progression. Thus, the higher band-gap values in lower Er doped systems would be the cause of a better electron hole separation under UV irradiation.

In situ FT-IR study of the adsorption and photocatalytic oxidation of ethanol over sulfated and metallized TiO_2

Murcia, JJ; Hidalgo, MC; Navio, JA; Arana, J; Dona-Rodriguez, JM

Applied Catalysis B: Environmental, **142-143** (2013) 205-213

Octubre, 2013 | **DOI**: 10.1016/j.apcatb.2013.05.022

TiO_2 Degussa P25, TiO_2 prepared by sol–gel submitted to sulfation pre-treatment and some metallized catalysts obtained by photodeposition of Au or Pt over the sulfated TiO_2, were evaluated in the reaction of ethanol photo-oxidation. FT-IR spectroscopy was used to investigate the surface features of the photocatalysts, identifying adsorbed species and following the evolution of intermediate products in the ethanol photo-oxidation reaction. Nature of surface acidity in terms of Brönsted and Lewis centers was also studied.

Results showed that sulfation pre-treatment and metallization were important factors influencing the selectivity. Acetaldehyde was the main oxidation product on sulfated TiO_2; in the case of P25 also acetates production was observed. The photodeposition of metals had a detrimental effect on the selectivity to acetaldehyde; on metallized catalysts the formation of stable secondary intermediates was detected.

Based on these findings, a reaction pathway for the ethanol photo-oxidation over the different photocatalysts, via acetaldehyde or via acetate formation is proposed.

Preferential oxidation of CO in excess H_2 over CuO/CeO_2 catalysts: Characterization and performance as a function of the exposed face present in the CeO2 support

Gamarra, D; Camara, AL; Monte, M; Rasmussen, SB; Chinchilla, LE; Hungria, AB; Munuera, G; Gyorffy, N; Schay, Z; Corberan, VC; Conesa, JC; Martinez-Arias, A

Applied Catalysis B: Environmental, **130-131** (2013) 224-238

Febrero, 2013 | **DOI**: 10.1016/j.apcatb.2012.11.008

A series of oxidised copper-cerium nanostructured catalysts prepared by impregnation of copper over ceria supports synthesized by different methods (hydrothermal with varying preparation parameters, microemulsion/precipitation), in order to achieve different specific morphologies (nanocubes, nanorods and nanospheres), have been examined with respect to their catalytic properties for preferential oxidation of CO in excess H2 (CO-PROX). The catalysts have been characterized in detail by XRD, Raman, SBET measurement, HREM, XPS, TPR and EPR, which allows establishing a model of structural characteristics of the catalysts. The characterization results have been correlated with analysis of CO-PROX catalytic properties by means of catalytic activity measurements complemented by operando-DRIFTS. Structural dependence of the CO oxidation reaction on the dispersed copper oxide entities as a function of the exposed face present at the surface of the different ceria supports is revealed. An important overall enhancement of the CO-PROX performance is detected for the sample supported on ceria nanocubes which is proposed to be a consequence of the interaction between copper oxide and (1 0 0) faces of the ceria support.

Multiple Zeolite Structures from One Ionic Liquid Template

Blanes, JMM; Szyja, BM; Romero-Sarria, F; Centeno, MA; Hensen, EJM; Odriozola, JA; Ivanova, S

Chemistry-A European Journal, **19** (2013) 2122-2130

Febrero, 2013 | **DOI**: 10.1002/chem.201202556

This study reports the use of 1-butyl-3-methyl imidazolium methanesulfonate ionic liquid as a template in the synthesis of zeolites. It is found that the silicon source determines the formation of beta (BEA), mordenite framework inverted (MFI), or analcime (ANA) zeolites. Depending on this source, different preorganized complexes are obtained that drive the formation of the different zeolite structures. In the presence of ethanol, the ionic liquid form preorganized complexes that drive the formation of MFI. In its absence, BEA is obtained. Whereas, the large amount of sodium present when using sodium metasilicate leads to ANA formation. A molecular simulation study of the relative stability of the template-framework system and location of the template provides further insight into the mechanism of synthesis.

Monoclinic Tetragonal Heterostructured $BiVO_4$ by Yttrium Doping with Improved Photocatalytic Activity

Usai, S; Obregon, S; Becerro, AI; Colon, G

Journal of Physical Chemistry C, **117** (2013) 24479-24484

Octubre, 2013 | **DOI:** 10.1021/jp409170y

Yttrium-doped BiVO4 has been synthesized by means of a surfactant free hydrothermal method having good photoactivities under sun-like excitation for the degradation of Methylene Blue (MB). From the structural and morphological characterization it has been stated that the presence of Y3+ induces the progressive stabilization of the tetragonal phase and the slight higher surface area values. By following the tetragonal cell parameters, the substitutional incorporation of Y3+ into the BiVO4 tetragonal lattice might be considered. Best photocatalytic performances were attained for the samples with Y3+ content of 3.0 at. % for which the MB degradation rate constant appears 2-fold higher. Furthermore, photoactivities for visible-light-driven O2 evolution demonstrate that the photocatalytic performance of the best Y-doped system (initial rate of O2 evolution, 285 μmol g–1 h–1) was more than 5 times that of undoped m-BiVO4 (initial rate of O2 evolution, 53 μmol g–1 h–1). The occurrence of Y3+ doping and a monoclinic–tetragonal heterostructured BiVO4 system induces the higher photocatalytic activities. PL analysis provides a clear evidence of the lower charge carriers recombination in heterostructured yttrium-doped systems.

Impact of Ce–Fe synergism on the catalytic behaviour of Au/CeO_2–FeO_x/Al_2O_3 for pure H_2 production

Reina, TR; Ivanova, S; Idakiev, V; Delgado, JJ; Ivanov, I; Tabakova, T; Centeno, MA; Odriozola, JA
Catalysis Science & Technology, **3** (2013) 779-787
Marzo, 2013 | **DOI**: 10.1039/C2CY20537H

In this work the development of a series of gold catalysts, essentially based on γ-alumina promoted with a small superficial fraction of Ce–Fe mixed oxides, is reported. The catalytic behaviour is evaluated in the water gas shift reaction. The formation of a Ce–Fe solid solution is evidenced by XRD and related to the catalytic activity where the importance of the Ce–Fe interaction is demonstrated. The best catalyst reached CO conversions very close to the equilibrium limit. A long-term stability test is performed and the loss of activity is observed and attributed to reaction intermediates. Almost complete recovery of the initial conversion is achieved after oxidation treatment, suggesting that the problem of stability could be overcome by a suitable change in the reaction parameters thus leading to a highly efficient catalyst for future applications in H_2 production and clean-up.

Gold(III) stabilized over ionic liquids grafted on MCM-41 for highly efficient three-component coupling reactions

Bobadilla, LF; Blasco, T; Odriozola, JA
Physical Chemistry Chemical Physics, **39** (2013) 16927-16934
Octubre, 2013 | **DOI**: 10.1039/C3CP52924J

Two alkoxysilyl-modified ionic liquids (ILs) have been synthesized and further grafted onto mesoporous silica, MCM-41; these ionic liquids were used for dispersing AuCl3 catalysts that activate C–H bonds as required for the synthesis of propargylamines by coupling alkyne, aldehyde and amine (A3 coupling) species. 29Si NMR experiments demonstrate the formation of covalent bonds between alkoxysilyl-modified Bmim IL and the MCM-41 surface through silanol groups. The catalytic activities of Au(III)-supported MCM-41 and Au(III) homogeneous catalysts are lower than those obtained for the IL functionalized Au–MCM-41 solids when the same gold loading is considered. An interaction between Au(III) species and the IL is proposed for explaining the stabilization of gold(III) species. However, successive reaction cycles result in a decrease in the catalytic activity that has been explained on the basis of gold leaching.

A low-temperature single-source route to an efficient broad-band cerium(III) photocatalyst using a bimetallic polyoxotitanium cage
Lv, YK; Yao, MM; Holgado, JP; Roth, T; Steiner, A; Gan, LH; Lambert, RM; Wright, DS
RSC Advances, **3** (2013) 13659-13662
Septiembre, 2013 | **DOI**: 10.1039/C3RA41524D

Aqueous hydrolysis of a series of cerium-containing polyoxotitanium cages gives Ce(III)-doped TiO_2 [TiO_2(Ce)] or TiO_2-supported Ce(III)$_2$$Ti_2O_7$, depending on the starting Ti : Ce ratio of the precursor. TiO_2-supported $Ce_2Ti_2O_7$ exhibits superior photocatalytic activity to the Ce-doped TiO_2 materials and unusual broad-band absorption behaviour across the visible and near-infrared regions.

Role of activated carbon on the increased photocatalytic activity of AC/Bi_2WO_6 coupled materials
Murcia-López, S; Navío, J.A.; Hidalgo, M.C.
Applied Catalysis A: General, **466** (2013) 51-59
Septiembre, 2013 | **DOI**: 10.1016/j.apcata.2013.06.022

The photocatalytic activities of several Bi_2WO_6 and TiO_2/Bi_2WO_6 materials with different activated carbon (AC) contents were studied for Rhodamine B (RhB) (and Phenol) photodegradation under UV–vis and vis illumination. A wide characterization of the materials was carried out. The addition of AC strongly affected the Bi2WO6 morphology although not the crystalline phase. Even in the material with the lowest AC content (2 wt% nominal content) a structure with hierarchical porosity was formed. AC presence increased the initial reaction rates in the degradation of RhB. An important improvement in the photoactivity under both UV–vis and vis illumination conditions was obtained with the lowest AC content (2 wt%) when compared to the pristine material Bi2WO6 or to the systems with higher AC additions. AC/TiO_2/Bi_2WO_6 materials were also improved in comparison to the TiO_2/Bi_2WO_6 heterostructure without carbon. The improvement cannot be only ascribed to adsorption capability and surface area effects. A mechanism explaining the role of AC on the photocatalytic activity improvement is proposed.

Cyclohexane photocatalytic oxidation on Pt/TiO_2 catalysts
Murcia, JJ; Hidalgo, MC; Navio, JA; Vaiano, V; Sannino, D; Ciambelli, P
Catalysis Today, **209** (2013) 164-169
Junio, 2013 | **DOI**: 10.1016/j.cattod.2012.11.018

Gas-solid heterogeneous photocatalytic oxidation (PCO) of cyclohexane in humidified air over TiO2 and Pt/TiO_2 catalyst was studied.

Pt/TiO_2 photocatalysts were synthesized by photodeposition method at different Pt loadings (0.5–2 wt.%). The addition of 0.5 wt.% Pt does not significantly modify the TiO_2 properties. The increase in Pt loading induces to an aggregation of metallic particles on TiO_2 surface.
The cyclohexane PCO was performed in a fluidized bed photoreactor at 60 and 100 °C. Pure TiO_2 was more active than 1 and 2 wt.% Pt/TiO_2 samples at 60 °C. Nevertheless, the conversion level increases with temperature on Pt/TiO_2 photocatalysts. The cyclohexane was mineralized into CO_2, water and low amount of CO. A beneficial effect of Pt addition was found, since total CO_2 selectivity was obtained. The Pt/TiO_2 photocatalysts prepared by photodeposition provide the total cyclohexane PCO without CO production. Photocatalysts deactivation was not observed in any performed test. Evidence of an opportune tuning of temperature is highlighted.

Effect of the alloy on micro-structured reactors for methanol steam reforming
Echave, FJ; Sanz, O; Velasco, I; Odriozola, JA; Montes, M
Catalysis Today, **213** (2013) 145-154
Septiembre, 2013 | **DOI**: 10.1016/j.cattod.2013.02.027

Micro-monoliths and foams made of aluminium, Fecralloy® and brass were studied as substrates for structured systems for methanol steam reforming (MSR). All the alloys exhibited very adherent oxide layer produced by pre-treatment to improve the adhesion between substrate and catalyst. 2.5% Pd/ZnO catalyst was prepared and deposited on structured substrates. Both, good catalyst adhesion and stable catalytic performance were achieved in the case of brass micro-monoliths. The Fecralloy® and aluminium substrates reacted with the catalytic active components resulting in catalyst modification. The aluminium based substrates promoted dimethyl ether (DME) formation. Aluminium foam produced better performance than aluminium micro-monoliths that could be related to improved mass and heat transfer properties in foams.

In situ characterization of iron-promoted ceria–alumina gold catalysts during the water-gas shift reaction
Reina, TR; Xu, WQ; Ivanova, S; Centeno, MA; Hanson, J; Rodriguez, JA; Odriozola, JA
Catalysis Today, **205** (2013) 41-48
Abril, 2013 | **DOI**: 10.1016/j.cattod.2012.08.004

In this work an in situ XRD and XANES study of two gold catalysts supported on iron-promoted ceria–alumina carriers was carried out during the water-gas shift reaction (WGS). The first catalyst, $Au/CeO_2–FeO_x/Al_2O_3$, was prepared using a commercial alumina support in order to obtain a Ce–Fe oxide solid solution and in the second one, $Au/FeO_x/CeO_2–Al_2O_3$, an iron oxide monolayer was deposited onto a ceria–alumina commercial support to promote its redox properties. Catalytic activities in the WGS were remarkably different for both systems. The catalytic activity of the $Au/CeO_2–FeO_x/Al_2O_3$ catalyst was higher than the one shown by the $Au/FeO_x/CeO_2–Al_2O_3$ catalyst that resulted active at much higher temperatures. In situ XRD demonstrates the formation of magnetite (Fe_3O_4) during the WGS reaction and the presence of big gold particles, ca. 21 nm in diameter, in the low-activity system. This in contrast to the high-activity system that shows undetectable gold nanoparticles and the absence of diffraction peaks corresponding to magnetite during the WGS. The data obtained using in situ XANES states that Ce^{4+} species undergo reduction to Ce3+during the WGS for both catalysts, and also confirms that in the high-activity catalyst iron is just present as Fe^{3+} species while in the low-activity catalyst Fe^{3+} and Fe^{2+} coexist, resulting in iron spinel observed by XRD. These results allow us conclude that the $Au/CeO_2–Fe_2O_3/Al_2O_3$ catalyst is a suitable catalyst for WGS when avoiding the formation of magnetite, in such a case Fe^{3+} species favors reduction and water splitting increasing the catalytic activity in the WGS reaction.

Influence of the O_2/CO ratio and the presence of H_2O and CO_2 in the feed-stream during the preferential oxidation of CO (PROX) over a CuOx/CeO_2-coated microchannel reactor

Laguna, OH; Dominguez, MI; Oraa, S; Navajas, A; Arzamendi, G; Gandia, LM; Centeno, MA; Montes, M; Odriozola, JA
Catalysis Today, **203** (2013) 182-187
Marzo, 2013 | **DOI**: 10.1016/j.cattod.2012.04.021

The catalytic performance of a CuOx/CeO_2 powder catalyst and that of a microchannel reactor or microreactor (MR) coated with the same solid was determined and compared. The catalytic activity measurements were carried out with varying O_2/CO molar ratios in the feed-stream. In addition, the influence of the presence of CO_2 and H_2O in the reaction mixture was studied. Some discrepancies were observed between the performances of the powder catalyst and the MR depending on the O_2/CO ratio. The MR presented a very good performance with a superior selectivity for CO conversion. This behaviour was due to a more efficient heat removal in the case of the MR that inhibited the H_2 oxidation reaction and the r-WGS. The isothermicity of the microreactor during the process was demonstrated through the monitoring of the MR inlet and outlet temperatures.
Concerning the presence of CO_2 or H_2O in the feed-stream, both compounds gave rise to a decrease of the CO conversion. The negative effect on the catalytic performance was more marked when both compounds were fed together, although the principal inhibitor effect was associated to the CO_2. This seems to be related with the formation of stable carbonates at the catalyst surface.

Preferential oxidation of CO over Au/CuOx–CeO_2 catalyst in microstructured reactors studied through CFD simulations

Uriz, I; Arzamendi, G; Dieguez, PM; Laguna, OH; Centeno, MA; Odriozola, JA; Gandia, LM
Catalysis Today, **216** (2013) 283-291
Noviembre, 2013 | **DOI**: 10.1016/j.cattod.2013.04.023

A computational fluid dynamics (CFD) simulation study of the preferential oxidation of CO (CO-PROX) in microstructured reactors consisting in square and semicircular microchannels coated with an Au/CuOx–CeO_2 catalyst is presented. The CO content of the feed stream was set at 1 vol.%. A parametric sensitivity analysis has been performed under isothermal conditions revealing that an optimal reaction temperature exists that leads to a minimum CO content at the microreactor exit. The influence of the space velocity, CO_2 concentration and oxygen-to-CO molar ratio in the feed stream (λ), catalyst loading, and microchannel characteristic dimension (d) on the microreactor performance has been investigated. Under suitable conditions, the CO concentration can be reduced below 10 ppm at relatively low temperatures within the 155–175 °C range. A negative effect of the increase of d from 0.35 mm to 2.8 mm on the CO removal efficiency has been found and attributed to a more detrimental effect of the mass transport limitations on the oxidation of CO than that of H_2. Non-isothermal CFD simulations have been performed to investigate the cooling of the CO-PROX reactor with air or a fuel cell anode off gas surrogate in parallel microchannels. Due to the very rapid heat transfer allowed by the microreactor and the strong influence of the reaction temperature on the exit CO concentration, a careful control of the coolant flow rate and inlet temperature is required for proper reactor operation. The microreactor behavior is virtually isothermal.

Efficient and affordable hydrogen production by water photo-splitting using TiO_2-based photocatalysts

Melian, EP; Diaz, OG; Mendez, AO; Lopez, CR; Suarez, MN; Rodriguez, JMD; Navio, JA; Hevia, DF; Pena, JP

International Journal of Hydrogen Energy **38** (2013) 2144-2155

Febrero, 2013 | **DOI**: 10.1016/j.ijhydene.2012.12.005

TiO_2-based photocatalyst materials were synthesized through a sol–gel method, followed either by: (1) hydrothermal treatment (150 °C/24 h), or (2) heat treatment (calcination) in a temperature range between 400 and 900 °C. The resulting materials were characterized through BET, XRD, TEM, FTIR, RAMAN, laser diffraction and UV–Vis Diffuse Reflectance Spectroscopy. Photoactivity of the various materials was checked against photocatalytic water-splitting for hydrogen production and a relationship between TiO_2 structure and hydrogen production capacity was identified. Optimum results were obtained for anatase-rutile mixtures in a ratio of 87:13. The activity of the home-made photocatalysts was also compared (under the same conditions) with the best commercially available materials which have been widely described in the literature: Hombikat UV100, Millenium PC100, Kronos vlp7000, Degussa P25 and Kemira 625.

Hydrogen production using Pt-loaded TiO_2 photocatalysts

Melian, EP; Lopez, CR; Mendez, AO; Diaz, OG; Suarez, MN; Rodriguez, JMD; Navio, JA; Hevia, DF

International Journal of Hydrogen Energy, **38** (2013) 11737-11748

Septiembre, 2013 | **DOI**: 10.1016/j.ijhydene.2013.07.006

A series of synthesised TiO_2-based and commercial photocatalysts were modified by Pt photodeposition and a study made of their photocatalytic activity in hydrogen production. The modified commercial photocatalysts were Evonik P25, Kronos vlp7000 and Hombikat UV-100, and the other modified photocatalysts were synthesised by our group using sol–gel and sol–gel hydrothermal processes (SG400, SG750 and HT). Pt weight percentages used in the study were 0.5, 1.0 and 2.1 wt.% (Pt/TiO2). The photocatalysts were extensively characterised by X-ray diffraction (XRD), UV–vis diffuse reflectance, Brunauer–Emmett–Teller (BET) surface area measurement, transmission electron microscopy (TEM), scanning electron microscopy (SEM–EDX), Fourier transform infrared spectroscopy (FTIR) and laser light dispersion. Methanol (25% vol.) was used as sacrificial agent over the 8 h of the hydrogen production tests and measurements were taken of the final concentrations of formaldehyde and formic acid as well as initial and final TOC. Photoactivity of all photocatalysts increased in the presence of Pt. The most efficient of the synthesised photocatalysts was SG750 and of the commercial photocatalysts P25. Maximum production of SG750 was 1846 μmol h−1 at 1.0 wt.% Pt and its production per surface unit was notably higher than that of P25.

Steam reforming of methanol over supported Ni and Ni–Sn nanoparticles
Bobadilla, LF; Palma, S; Ivanova, S; Dominguez, MI; Romero-Sarria, F; Centeno, MA; Odriozola, JA
International Journal of Hydrogen Energy, **38** (2013) 6646-6656
Mayo, 2013 | **DOI**: 10.1016/j.ijhydene.2013.03.143

The influence of the synthesis method and Sn addition on Ni/CeO_2–MgO–Al_2O_3 catalyst is correlated to its catalytic behavior in the reaction of methanol steam reforming. The catalysts prepared by impregnation method are compared to samples obtained by deposition of previously obtained nanoparticles by the polyol method. X-ray diffraction (XRD), specific surface area measurements and H_2-temperature programmed reduction (TPR) were used to characterize the catalysts. The differences of the structure, phase transformation and reduction behavior are discussed and related to the catalytic performance of the samples as well as the nature of the carbonaceous deposits formed during the reaction.

Degradation of Rhodamine B/Phenol Mixtures in Water by Sun-Like Excitation of a Bi_2WO_6–TiO_2 Photocatalyst
Murcia-López, S; Navío, J.A.; Hidalgo, M.C.
Photochemistry and Photobiology, **89** (2013) 832-840
Julio, 2013 | **DOI**: 10.1111/php.12054

Bi_2WO_6 and Bi_2WO_6–TiO_2 (5% molar Ti) nano-heterostructures were synthesized by a hydrothermal method. The properties of the synthesized catalysts were characterized, having high photoactivity for Rhodamine B degradation under sun-like illumination, explained by a synergetic mechanism previously proposed through UV and visible induced processes, in which the photosensitization effect of Rhodamine B is considered. We now report that using Phenol, a molecule which does not lead the photosensitization process, the photoactivity decreased considerably, thus emphasizing how important is the model molecule selected as degradation substrate for evaluating the photoactivity. The photocatalytic properties of the synthesized catalysts have been evaluated by exposing a mixture of Rhodamine B and Phenol in water, to different illumination conditions. It can be confirmed that the photoinduced mechanism via the photosensitization of Rhodamine B is a key factor responsible for the increase on the photocatalytic activity showed by the Bi_2WO_6–TiO_2 compound and that the degradation mechanism of Rhodamine B is not changed by the simultaneous presence of other transparent substrate as Phenol.

Au/TiO_2 supported on ferritic stainless steel monoliths as CO oxidation catalysts
Milt, VG; Ivanova, S; Sanz, O; Dominguez, MI; Corrales, A; Odriozola, JA; Centeno, MA
Applied Surface Science, **270** (2013) 169-177
Abril, 2013 | **DOI**: 10.1016/j.apsusc.2012.12.159

Metallic supported structured catalysts were obtained by washcoating AluchromYHf monoliths with an Au/TiO_2 catalyst. The powder catalyst was synthesized by DAE (direct anionic exchange) method. Using this catalyst, a stable slurry was prepared and used to washcoat the monoliths. TEM and SEM studies revealed that gold nanoparticles in the Au/TiO_2 powder catalyst had an average diameter of 3–4 nm, but during the preparation of the structured catalyst, aggregate Au particles of the slurry reached diameters of 9 nm. Before coating, Aluchrom YHf monoliths were thermally treated to generate a homogeneous and well-adhered oxide rough surface layer, mainly composed of α-Al_2O_3 whiskers, which favored the anchoring of the catalyst. The catalytic layer deposited was well attached and contained not only the Au/TiO_2 catalyst but also metallic oxides formed from stainless steel components that diffused through the oxide scale. The structural characterization was performed by XRD, XRF, TEM, SEM, GD-OES and SBET.

The catalytic activity of the powder and structured catalysts was tested in the oxidation of the CO reaction. Catalysts demonstrated to be active at room temperature. After a first activation run, and in spite of their larger gold particle size, the catalytic activities of the structured catalysts overcame those of the powder catalyst. This improvement is probably due to the segregation of the transition metal oxides toward the surface oxide scale.

Improved photocatalytic activity of g-C_3N_4/TiO_2 composites prepared by a simple impregnation method

Miranda, C; Mansilla, H; Yanez, J; Obregon, S; Colon, G

Journal of Photochemistry and Photobiology A: Chemistry, **253** (2013) 16-21

Febrero, 2013 | **DOI**: 10.1016/j.jphotochem.2012.12.014

g-C_3N_4 and TiO_2 hybrid structures are synthesized by means of a simple impregnation method having good photoactivities for the degradation of phenol under UV irradiation. From the wide structural and surface characterization we have stated that the presence of g-C_3N_4 notably affect the surface feature of TiO_2 (surface area and pore size distribution). Enhanced photoactivities have been obtained for composites systems. The best result was obtained for 2 wt% loading of g-C_3N_4 leading to a 70% of improvement with respect to bare TiO_2 in the reaction rate. The effective charge carrier separation was proposed as the responsible of such improved photoactivity.

Au/CeO_2 metallic monolith catalysts: influence of the metallic substrate

Tejada, LMM; Dominguez, MI; Sanz, O; Centeno, MA; Odriozola, JA

Gold Bulletin, **46** (2013) 221-231

Diciembre, 2013 | **DOI**: 10.1007/s13404-013-0102-0

Ceria-based gold catalysts were successfully deposited on ferritic stainless steel (Fecralloy) and aluminium monoliths. The prepared monolithic and reference powder catalysts were characterized by means of S-BET, X-ray diffraction, glow discharge optical emission spectroscopy and scanning electron microscopy-energy dispersive X-ray analysis techniques and tested in the CO oxidation reaction. Characterization results put in evidence the diffusion of cations from the catalytic layer on the surface of the monoliths to the metallic oxide scale and inversely, from the oxide scale to the catalysts, thus altering the catalytic formulation and affecting the CO oxidation properties of the catalytic device. The extension and nature of the modifications produced depend on the nature of the catalysts and the metallic substrate, as well as the reaction conditions applied. These facts must be considered when gold catalysts are supported on metallic-structured devices.

Gas-phase Photocatalytic Partial Oxidation of Cyclohexane to Cyclohexanol and Cyclohexanone on Au/TiO_2 Photocatalysts
Sannino, D; Vaiano, V; Ciambelli, P; Murcia, JJ; Hidalgo, MC; Navio, JA
Journal of Advanced Oxidation Technologies, **16** (2013) 71-82
Enero, 2013

The heterogeneous photocatalytic partial oxidation of cyclohexane in gas-phase as an alternative green process for fine chemicals synthesis was successfully achieved on Au/TiO_2 photocatalysts prepared by photodeposition technique. Different gold loadings ranging between 0.5 and 2 wt.% of photodeposited Au on TiO_2 synthesized by sol-gel method were obtained by changing the concentration of gold precursor at fixed illumination intensity and time. The cyclohexane partial photoxidation was conducted in a gas-solid photocatalytic fluidized bed reactor at high illumination efficiency. Main observed reaction products were cyclohexanol, cyclohexanone and CO_2. The resulting selectivity was dramatically influenced by the gold content. The reaction temperature was a critical parameter to reach the photocatalysts stability, avoiding deactivation phenomena while the tuning of Au content of the photocatalysts, resulted in the promotion of the formation of cyclohexanol or cyclohexanone with high selectivity. In particular, by increasing Au content, the process selectivity is completely reversed, passing from high cyclohexanol selectivity (75%) to high selectivity to cyclohexanone (80%). These promising results evidenced that Au/TiO_2 catalysts in the selected operating conditions, are effective materials for the synthesis of cyclohexanone and cyclohexanol in gas-phase by photocatalysis, at very low reaction temperatures and without the additional step of catalyst recovering needed in the liquid partial oxidation of cyclohexane.

On the different photocatalytic performance of $BiVO_4$ catalysts for Methylene Blue and Rhodamine B degradation
Obregon, S; Colon, G
Journal of Molecular Catalysis A: Chemical, **376** (2013) 40-47
Septiembre, 2013 | **DOI**: 10.1016/j.molcata.2013.04.012

$BiVO_4$ hierarchical structures were synthesized by means of a surfactant free hydrothermal method having good photoactivities for the degradation of Methylene Blue and Rhodamine B under UV–vis irradiation. From the structural and morphological characterization it has been stated that BiVO4 present the monoclinic crystalline phase with different morphologies depending on the pH value. For Methylene Blue the photodegradation rate is strongly affected by the crystallite size and higher (0 0 4) facet exposition. On the contrary, for Rhodamine B, the ζ-potential of the surface clearly determines the photocatalytic performance of BiVO4 catalyst.

Preparation of Titanium Oxide/Silicon Oxide (TiO_2/SiO_2) systems through the solvothermal method for applications in photocatalysis

Galeano, L.; Navío, J.A.; Restrepo, G.M.; Marín, J.M.

Información Tecnológica, **24** (2013) 81-92

DOI: 10.4067/S0718-07642013000500010

Sistemas Óxido de Titanio/Óxido de Silicio (TiO_2/SiO_2) fueron obtenidos por anclaje de TiO_2 en SiO_2. El fotocatalizador TiO_2 se obtuvo por alcohólisis del TiCl4 con 2-propanol y posterior cristalización a presión autógena a 200 °C, excluyendo etapas de calcinación a altas temperaturas. Se emplearon diferentes relaciones SiO2/TiCl4 para determinar su influencia en la estabilidad, propiedades y fotoactividad de los sistemas TiO_2/SiO_2. La actividad fotocatalítica fue evaluada por la fotodegradación de metanol en fase gaseosa. El TiO_2 cristalizó como fase anatasa o como una mezcla rutilo/anatasa, dependiendo por la relación $SiO_2/TiCl_4$ inicial. Los resultados muestran que se producen materiales compuestos con alta cristalinidad del TiO_2. Se encontró también que hay una fuerte relación entre la actividad fotocatalítica con las propiedades fisicoquímicas y de estas con las composiciones iniciales de síntesis.

Low-temperature CO oxidation on multicomponent gold based catalysts

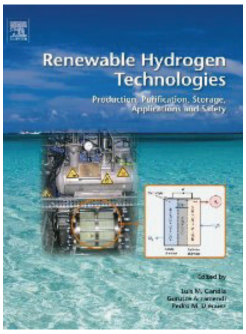
Tomás Ramírez Reina; Svetlana Ivanova; Miguel A. Centeno; José A. Odriozola

Frontiers, **1** (2013)

DOI: 10.3389/fchem.2013.00012#sthash.gw90OSot.dpuf

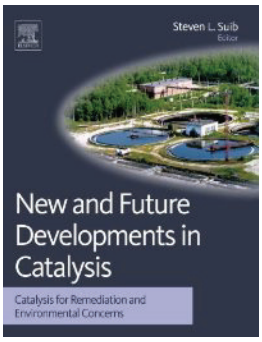
Sistemas Óxido de Titanio/Óxido de Silicio (TiO_2/SiO_2) fueron obtenidos por anclaje de TiO_2 en SiO_2. El fotocatalizador TiO_2 se obtuvo por alcohólisis del TiCl4 con 2-propanol y posterior cristalización a presión autógena a 200 °C, excluyendo etapas de calcinación a altas temperaturas. Se emplearon diferentes relaciones SiO2/TiCl4 para determinar su influencia en la estabilidad, propiedades y fotoactividad de los sistemas TiO_2/SiO_2. La actividad fotocatalítica fue evaluada por la fotodegradación de metanol en fase gaseosa. El TiO_2 cristalizó como fase anatasa o como una mezcla rutilo/anatasa, dependiendo por la relación $SiO_2/TiCl_4$ inicial. Los resultados muestran que se producen materiales compuestos con alta cristalinidad del TiO_2. Se encontró también que hay una fuerte relación entre la actividad fotocatalítica con las propiedades fisicoquímicas y de estas con las composiciones iniciales de síntesis.

LIBROS Y OTRAS PUBLICACIONES / BOOKS AND OTHER PUBLICATIONS



Advances in structured and microstructured catalytic reactors for hydrogen production
Oihane Sanz; Francisco J. Echave; Francisca Romero-Sarria; Jose Antonio Odriozola; Mario Montes
En: Renewable Hydrogen Technologies: Production, Purification, Storage, Applications and Safety, (2013) 201-224 (Editorial Elsevier Scientific Tecnology, United Kingdom)
ISBN: 978-0-444-56352-1

Microprocess technology for hydrogen purification
Svetlana Ivanova; Oscar Hernando Laguna; Miguel Angel Centeno; Asier Eleta; Mario Montes; Jose Antonio Odriozola
En: Renewable Hydrogen Technologies: Production, Purification, Storage, Applications and Safety, (2013) 225-243 (Editorial Elsevier Scientific Tecnology, United Kingdom)
ISBN: 978-0-444-56352-1



Structured Catalysts for Volatile Organic Compound Removal
Svetlana Ivanova; Alejandro Pérez; Miguel Ángel Centeno; José Antonio Odriozola
En: New and Future developments in Catalysis. Catalysis for Remediation and Environmental Concerns, (2013) 233-256 (Editorial Elsevier Scientific Tecnology, United Kingdom)
ISBN: 978-0-444-53874-1

Photocatalytic nano-oxides; the case of TiO_2 and ZnO
A. Iglesias-Juez; A. Kubacka; G. Colón; M. Fernández-García
En: New and Future developments in Catalysis. Catalysis for Remediation and Environmental Concerns, (2013) 245-266 (Editorial Elsevier Scientific Tecnology, United Kingdom)
ISBN: 978-0-444-53874-1

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

COMUNICACIONES EN CONGRESOS Y REUNIONES / COMMUNICATIONS IN CONGRESS AND MEETINGS

IV Congreso Internacional y XIII Congreso Mexicano de Catálisis

16 – 19 abril [Puerto Vallarta, México]

Desarrollo de microreactores de pared catalítica para tecnologías energéticas
O.H. Laguna, M. Montes, G. Arzamendi, L.M. Gandía, M.A. Centeno, J.A. Odriozola
Comunicación oral

7th World Congress On Oxidation Catalysis (7WCOC)

9 – 12 junio [Saint Louis, Missouri, Estados Unidos de América]

1-Octanol Selective Oxidation over Supported Gold Catalysts: Influence of the Support Nature, Metal Load and Reaction Conditions
Vicente Cortés Corberán, Almudena Gómez-Avilés, Susana Martínez-González, Svetlana Ivanova, Maria I. Dominguez, Roberto Fernandez-Lafuente
Comunicación oral

H_2 oxidation as criteria for PROX catalyst selection
Tomás Ramírez Reina, Alejandro Pérez Flórez, Miguel Ángel Centeno, José Antonio Odriozola, Svetlana Ivanova
Comunicación oral

Heterogeneous selective oxidation of fatty alcohols: Oxidation of 1-Tetradecanol as a model substrate
Vicente Cortés Corberán, Susana Martínez-González, Almudena Gómez-Avilés, Maria I. Dominguez, Svetlana Ivanova, M. Elena González-Pérez
Comunicación oral

Influence of methanol concentration on CO oxidation over CuOx/CeO2 catalysts
Sandra Palma del Valle, Alejando Pérez Flores, Francisca Romero-Sarria, Miguel Ángel Centeno Gallego, Óscar H. Laguna Espitia, Jose Antonio Odriozola Gordon
Comunicación oral

4th International Conference on Semiconductor Photochemistry – SP4

23 – 27 junio [Praga, República Checa]

Photocatalytic Propylene Epoxidation with Bi2WO6-Based Photocatalysts
Murcia López, S., Hidalgo, M.C., Navío, J.A., Vaiano, V., Ciambelli, P., Sannino, D.

AI-NanoFunc Workshops 2013
1 – 2 julio [Sevilla, España]

Chlorination of toluene over Ionic liquid grafted in carbon nanofiber
A.Martínez-Pascual, S. Ivanova, P. Losch, M. Boltz, B. Louis, F. Montilla J.A. Odriozola
Comunicación oral

CARBON 2013
14 – 19 julio [Rio de Janeiro, Brasil]

Photocatalytic Performance of Hybrid Bi_2WO_6-Carbon composites under UV and Visible Light
Rocio J. Carmona, Sebastian Murcia-Lopez, J.A. Navio, M.Carmen Hidalgo, Conchi O. Ania

XVII International Sol-Gel Conference
25 – 30 agosto [Madrid, España]

Modified Sol-Gel synthethic route to layered Nb_2O_3 and TiO_2
A. Martínez-Pascual, S. Ivanova, M.A. Centeno, J.A. Odriozola
Comunicación oral

11th European Congress on Catalysis, EuropaCat-XI
1 – 6 septiembre [Lyon, Francia]

Could an excellent WGS catalyst be useful in the PROX reaction?
T.R. Reina, E. Papadopoulou, S. Ivanova, M.A. Centeno, T. Ioannides, J.A. Odriozola
Comunicación oral

Effect of Rh/Ru addition on the catalytic properties of CeZr(Co) mixed oxides for glycerol steam reforming reaction. An in situ Raman spectroscopy study
L. M. Martínez T., M. A. Araque, M. A. Centeno, A. C. Roger
Poster

Influence of niobium oxide protection ligands on the hydrolysis of cellulose
A. Martínez-Pascual, S. Ivanova, M.A. Centeno, J.A. Odriozola
Poster

Kinetic modelling of glycerol steam reforming over micro-structured catalyst
L.F. Bobadilla, A. Álvarez, M.I. Domínguez, F. Romero-Sarria, M.A. Centeno, J.A. Odriozola
Poster

Noble Metal doped Ru/TiO_2 catalysts for Selective Methanation
A. Muñoz-Murillo; A. Pérez; M. A. Centeno; J.A. Odriozola
Poster

Platinum vs. Gold Catalyst for WGS: Could Platinum takes the Gold?
M.González; T.R. Reina; S. Ivanova; M.A. Centeno; J.A. Odriozola
Poster

5th Conference on Carbon for Energy Storage/Conversion and Envrironment Protection

23 – 26 septiembre [Mülheim an der Ruhr, Alemania]

Application of Bi_2WO_6-Carbon composites as visible light-photocatalyst
R.J.Carmona, S. Murcia López, J.A.Navio, M.C.Hidalgo, C.O. Ania
Poster

4 th International Conference on Structured Catalysts and Reactors, ICOSCAR-4

25 – 27 septiembre [Beijing, China]

Effect of enameling steel monoliths on the activity of PROX catalysts
A. Pérez, M.I. Domínguez, M.A. Centeno, M. Montes, J. A. Odriozola
Comunicación oral

Kinetics of the preferential oxidation of CO (CO-PrOx) and CFD simulations: comparison between CuOx-CeO_2 and Au/CuOx-CeO_2 catalysts in microstructured reactors
I. Uriz, G. Arzamendi, P. M. Diéguez, O. H. Laguna, M. A. Centeno, J. A. Odriozola, L. M. Gandía
Comunicación oral

12èmes Journées Algériennes de Catalyse

11 – 12 diciembre [Sétif, Argelia]

Microreactors technology for hydrogen production and purification
José Antonio Odriozola
Conferencia Plenaria

CONGRESOS Y REUNIONES NACIONALES / NATIONAL CONGRESSES AND MEETINGS

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

Reunión de la Sociedad Española de Catálisis SECAT 2013

26 – 28 junio [Sevilla, España]

José Antonio Odriozola Gordón [Comité Científico]
Miguel Angel Centeno Gallego [Comité Organizador]
Svetlana L. Ivanova - Antonio Martínez Pascual - Alejandro Pérez Flórez - Ara Muñoz Murillo
Miriam González Castaño - Sandra Palma del Valle - Tomás Ramírez Reina - Alicia Corrales Miralles - Andrea Álvarez Moreno - Oscar Hernando Laguna Espitia - Leidy Marcela Martínez Tejada - Anna D. Penkova

COMUNICACIONES EN CONGRESOS Y REUNIONES / COMMUNICATIONS IN CONGRESS AND MEETINGS

Reunión de la Sociedad Española de Catálisis SECAT 2013

26 – 28 junio [Sevilla, España]

Microrreactores para la oxidación preferencial de CO en presencia de H2: consideración de limitaciones al transporte de materia
O.H. Laguna; G. Arzamendi; L.M. Gandía; M.A. Centeno; J.A. Odriozola
Comunicación oral

Caracterización de un microrreactor tras su operación en la oxidación preferencial de CO en presencia de H_2
O.H. Laguna; A. Corrales; M.A. Centeno; J.A. Odriozola
Poster

Catalizadores metálicos estructurados: Influencia del substrato en la composición y actividad del catalizador soportado
A. Pérez, M.I. Domínguez, L.M. Martínez T., M.A. Centeno, J.A. Odriozola
Poster

Correlación entre la conversión de CO en WGS y la producción de CO en el reformado oxidativo de etanol con vapor, empleando un catalizador de Co promovido con Ce
H.M. Espitia, T. R.Reina, M.A. Centeno, J.A. Odriozola, S. Moreno, R.A. Molina
Poster

Efecto de la adición de Au sobre un catalizador CuOx-CeO2 para la oxidación preferencial de CO en presencia de H2 (PROX)
O.H. Laguna, W.Y. Hernández, G. Arzamendi, L.M. Gandía, M.A. Centeno, J.A. Odriozola
Poster

Efecto de la adición de metales nobles a catalizadores Ru/TiO_2 para metanación selectiva de CO
A. Muñoz-Murillo, A. Pérez, M. A. Centeno, J. A. Odriozola
Poster

Empleo de catalizadores bimetálicos Au/Cu soportados para la oxidación total de CO

W.Y. Hernández, O. H. Laguna, S. Ivanova, M. I. Domínguez, M.A. Centeno, J.A. Odriozola
Poster

Estudio de la reacción de PROX sobre sistemas Au/CeO_2-ZnO/Al_2O_3
T.R. Reina, A. Pérez, S. Ivanova, M.A. Centeno, J.A. Odriozola
Poster

Incremento de la estabilidad de un catalizador de Ni mediante la incorporación de CO para el reformado de etanol con vapor oxidativo
M. Muñoz, A. Alvarez, M.A. Centeno, F. Mondragón, J. Gallego, S. Moreno, R. Molina
Poster

Influencia de la concentración de metanol en la oxidación selectiva de CO en presencia de hidrógeno sobre catalizadores CuOx/CeO_2
S. Palma, F. Romero-Sarria, M.A. Centeno, J.A. Odriozola
Poster

Influencia del anión en la función plantilla de líquido iónico en la síntesis de zeolitas tipo MFI y BEA
J.M. Martínez Blanes, R. Ayala Espinar, S. Ivanova, F. Romero Sarria, J.A. Odriozola
Poster

Influencia del contenido de zeolita USY en el craqueo de 1,3,5 triisopropilbenceno
Y. Agámez-Pertuz, L.A. Oviedo, U. Navarro, M.A. Centeno, J.A. Odriozola
Poster

Reducibilidad del catalizador Co/CeO_2-ZrO_2 y su efecto en reacciones de reformado de hidrocarburos
E. Ambroise; L. M. Martínez T; V. López-Flores; X. Carrier; E. Marceau; V. Briois; M. A. Centeno; A. C. Roger
Poster

Oxidación selectiva heterogénea de alcanoles sobre catalizadores nanoparticulados de oro: efecto del soporte y la carga metálica
A. Gómez-Avilés; S. Martínez González; S. Ivanova; M. I. Dominguez; R. Fernández-Lafuente; V. Cortés Corberán
Poster

La importancia de la estructura en la reactividad de catalizadores Cu-Fe/PtAl en la reacción de PROX
A. Penkova, S. Palma, M.A. Centeno, J.A. Odriozola
Poster

PROX y WGS: Dos reacciones, un catalizador
T.R. Reina; S. Ivanova; E. Papadopoulou; T. Tabakova; V. Idakiev; T. Ioannides; M. A. Centeno; J.A. Odriozola
Poster
Pt vs Au en WGS

M. González-Castaño; T.R. Reina; S. Ivanova; M.A. Centeno; J.A. Odriozola
Poster

Reformado con vapor de agua de la mezcla glicerina-metano: efecto de la dispersión del Ni en catalizadores Ru-Ni
A. Álvarez; M.A. Centeno; F. Romero-Sarria; J.A. Odriozola
Poster

Síntesis y reactividad de óxido de niobio laminar en la hidrólisis de celulosa y la deshidratación de glucosa
A. Martínez-Pascual; S. Ivanova; F. Romero Sarria; J.A. Odriozola
Poster

Oxidacion Fotocatalitica de Etanol sobre Catalizadores de TiO2 y M-TiO2 (M=Au, Pt). Estudio FTIR
J.J. Murcia, M.C.Hidalgo, J.A.Navio, J.Araña
Poster

Evaluacion de Materiales Basados en Bi2WO6 para la Reduccion Fotocatalitica de CO_2
S. Murcia-Lopez, M.C. Hidalgo, J.A.Navio, V. Vaiano, P. Ciambelli, D. Sannino
Poster

Preparacion de Sistemas de Bi_2WO_6 Acoplados con Carbón Activado con Propiedades Fotocataliticas Mejoradas
S. Murcia-Lopez, M.C. Hidalgo, J.A.Navio
Poster

FORMACION / TRAINING

TESIS DOCTORALES/ DOCTOR DEGREE THESIS

Título:	**Sistemas Catalíticos basados en Nanopartículas Mono y Bimetálicas Activos en Procesos de interés medioambiental**
Autor:	Fátima A. Ternero Fernández
Directores:	Alfonso Caballero Martínez y Juan Pedro Holgado Vázquez
Calificación:	Sobresaliente "Cum Laude"
Centro:	Universidad de Sevilla

Título:	**Desarrollo de materiales basados en Bismuto con alta eficiencia fotocatalítica para aplicaciones de química verde**

Autor: Sebastián Murcia López
Directores: José Antonio Navío Santos y M. Carmen Hidalgo López
Calificación: Sobresaliente "Cum Laude"
Centro: Universidad de Sevilla

Título: **Control de la nanoestructura de sistemas M-TiO_2 (M=Pt y Au) preparados por fotodeposición con propiedades fotocatalíticas optimizadas**
Autor: Julie Joseane Murcia Mesa
Directores: José Antonio Navío Santos y M. Carmen Hidalgo López
Calificación: Sobresaliente "Cum Laude"
Centro: Universidad de Sevilla

FORMACIÓN DE GRADUADOS / MASTER DEGREE THESIS

Título: **Obtención de cementos belíticos a partir de residuos industriales**
Autor: Juan Miguel Figueroa Yañez
Directores: María Isabel Dominguez Leal, Francisco A. Corpas Iglesias
Grado: Trabajo Fin de Master
Año Académico: 2012-2013 (11 Julio 2013)

Título: **Catalizadores Heterogéneos Cu/ZnO/Al2O3 para la reacción de Water Gas Shift**
Autor: José Luis Santos Muñoz
Directores: José Antonio Odriozola Gordón
Grado: Trabajo Fin de Master
Año Académico: 2012-2013 (19 Julio 2013)

Título: **Síntesis, caracterización y actividad catalítica de líquidos iónicos soportados sobre nanofibras de carbono**
Autor: Antonio Samuel Martínez Pascual
Directores: José Antonio Odriozola Gordón, Svetlana Ivanova
Grado: Trabajo Fin de Master
Año Académico: 2012-2013 (24 Septiembre 2013)

Título: **Hormigones resistentes al fuego aditivados**
Autor: Blas González González
Directores: Svetlana Ivanova, Francisco A. Corpas Iglesias
Grado: Trabajo Fin de Master
Año Académico: 2012-2013 (30 Septiembre 2013)

Título:	**Valorización de residuos de construcción y demolición generados en obras de edificación**
Autor:	Alberto Domínguez Martín
Directores:	Svetlana Ivanova, María Isabel Domínguez Leal
Grado:	Trabajo Fin de Master
Año Académico:	2012-2013 (12 Diciembre 2013)

DOCENCIA / TEACHING

El Plan Nacional de I+D+i: Posibilidades de Éxito (Rama Ciencias)
Dr. José Antonio Odriozola Gordón
Fecha de Celebración: 12 de noviembre de 2012
Lugar: Universidad de Sevilla. Pabellón de México. Sevilla
Organismo/Centro Organizador: Instituto de Ciencias de la Educación (ICE), Universidad de Sevilla

ESTANCIAS Y VISITAS DE PERSONAL DEL ICMS EN OTROS CENTROS
PERSONNEL OF THE ICMS IN OTHER LABORATORIES

Universidad de Bucarest		
Bucarest, Rumania	**José Antonio Odriozola Gordón**	3 dias
	Miguel Angel Centeno Gallego	3 dias
Departamento de Ingeniería Química. Facultad de Ingeniería. UDEA		
Medellin, Colombia	**Svetlana Ivanova**	1 semana
Universidad de Utrech		
Utrech, Holanda	**Sandra Palma del Valle**	3 meses
Université Louis Pasteur		
Estrasburgo, Francia	**Andrea Alvarez Moreno**	3 meses

ESTANCIAS Y VISITAS DE INVESTIGADORES EN EL ICMS
PERSONNEL OF THE OTHER LABORATORIES IN THE ICMS

Université Setif 1		
Seti, Argelial	**Fatima Ammari**	2 semanas
Instituto de Investigaciones en Materiales. Universidad Nacional Autónoma de México		
México	**Lorena Martínez De la Cruz**	1 mes
Institute of Catalysis, Bulgarian Academy of Sciences		
Sofia, Bulgaria	**Ivan Bogoev Ivanov**	1 mes
Universidad Nacional		
Bogotá, Colombia	**Hector Mauricio Espitia Sibaja**	1 mes
	Carlos Mario Urruchurto	2 meses
ENSICAEN. L'Ecole Nationale Supérieure d'Ingénieurs de Caen		
Caen, Francia	**Josselin Perrin**	3 meses
	Florent Hugot	3 meses
	Romain Saubion	4 meses
Universidad de Utrech		
Utrech, Holanda	**Caterina Suzanna Wondergen**	4 meses

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

- Dra. María Dolores Alba Carranza

Profesores Titulares

- Dr. Rafael Alvero Reina
- Dr. Alfonso Bravo León
- Dra. Caroline M. Clauss Klamp

Investigadores Contratados

- Dr. Said El Mrabet
- Dra. M. Mar Orta Cuevas
- Dr. Joaquin Ramírez Rico

Becarios Predoctorales

- Ldo. Antonio A. Gutiérrez Pardo
- Ldo. Fredy A. Huaman Mamani
- Lda. M. Carmen Vera García

PROYECTOS DE INVESTIGACIÓN / RESEARCH PROJECTS

Unión Europea
Fondo Europeo de Desarrollo Regional
"Una manera de hacer Europa"
MINISTERIO DE ECONOMÍA Y COMPETITIVIDAD

Inmovilización de cationes en un espacio confinado de alta densidad de carga: gestión de residuos peligrosos

Immobilization of cations in high-density charge confined spaces: management of harmful cations wastes

Código/Code:	CTQ2010-14874/BQU (Plan Nacional)
Periodo/Period:	01-01-2011 / 31-12-2013
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	82.280,00 €
Investigador responsable/Research head:	María Dolores Alba Carranza
Componentes/Research group:	Miguel Angel Castro Arroyo, Maria del Mar Orta Cuevas, Mery Carolina Pazos Zarama, Said ElMrabet, Esperanza Pavón González, Maria Villa Alfageme, Santiago Medina Carrasco, Ana Isabel Becerro Nieto, Alberto José Fernández Carrión

RESUMEN / ABSTRACT

El tema central del Proyecto aborda la exigencia tecnológica 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 crecientes en los planes de I + D + i de los últimos años. Es en este escenario donde debe encuadrarse la presente propuesta, circunscrita en dos hallazgos básicos experimentales: el diseño de silicatos laminares expansibles de alta carga con una distribución controlada de sus centros activos que le confieren especiales propiedades como precursores para el diseño de materiales eficaces en cuanto a la retención de residuos nocivos y radiactivos, y la obtención de fases disilicatos insolubles en condiciones suaves, apropiadas para la inmovilización de dichas especies. Dicho objetivo representa un cambio cualitativo en el trabajo que se viene desarrollando en cuanto a su aplicación a residuos radioactivos y tóxicos reales, de una parte, y en cuanto a la aplicación de la metodología desarrollada en sistemas modelos a silicatos de utilidad, de otra. Los objetivos se adecuan, en general, a las líneas prioritarias de Investigación básica de Química, en concreto en el área de Química Inorgánica (Química del Estado Sólido) y Química Medioambiental, pero a pesar de su carácter básico posee un carácter aplicado que se adecua a diversas líneas de investigación de Química Orientada y ha despertado el interés de diversas empresas observadoras (ENRESA, BEFESA y ALQUIMIA). Estos objetivos requieren el desarrollo de técnicas de análisis sofisticado como la Resonancia magnética Nuclear de Sólido, la Difracción de Rayos-X bajo atmósfera controlada o espectroscopia gamma de bajo recuento, teniendo en cuenta el objetivo particular de dicha área de usar la tecnología instrumental y experimental para el estudio de materiales, satisfaciendo además el objetivo O2.5 (Potenciar la disponibilidad de infraestructuras de uso interdisciplinar y de uso compartido por los distintos agentes del sistema) del plan nacional de I+D+i 2008-2011. La viabilidad de la propuesta se garantiza, en pri-

mer lugar, porque el EI ha publicado recientemente la síntesis de silicatos laminares hidratables de alta carga, donde describe un método a partir del cual se puede ajustar la carga deseada para el material; en segundo lugar, el EI posee una dilatada experiencia en el diseño de mecanismos de síntesis de silicatos, como se demuestra por los numerosos artículos publicados en esta área durante la última década y finalmente, el EI ha desarrollado metodología necesaria para el desarrollo de este experimento en colaboración con otros Grupos con los que mantiene una estrecha colaboración científica.

The central subject of this Project deals with the environmental technological exigency for development of advanced technologies for the elimination of polluting agents. The interest and the effort dedicated to the development of new technologies that allow more effective treatments of retention and new procedures of valorisation is increasing in numerous R&D plans in the last years. It is in this scene where the present proposal must be fitted and circumscribed into two experimental basic findings: designing expansible high layer charge silicates with a controlled distribution of active centres, which can be effective materials for the retention of hazardous and radioactive wastes, and obtaining insoluble disilicate phases in smooth conditions, appropriate for the immobilization of such species. This objective represents a qualitative change in the work that has been developed up to now in relation to the elimination of radioactive and toxic wastes as well as in the application of the methodology to silicate systems. The objectives are adapted, in general, to the high-priority lines of Basic Investigation of Chemistry, in the area of Inorganic Chemistry (Solid State Chemistry) and Environmental Chemistry in particular but, in spite of its basic character, the Project is adapted to diverse lines of investigation of Oriented Chemistry and it is supported by different EPOs (ENRESA, BEFESA and ALQUIMIA). These objectives, of eminent basic character, require the development of techniques of sophisticated analysis like advanced Solid State NMR, X-ray diffraction, under conditions of controlled pressure and temperature or gamma spectroscopy of low counts. This fact fits to the particular objective of the Chemistry Area of “using the instrumental and experimental technology for the study of materials” and with objective O2.5 (Enhance the availability of interdisciplinary infrastructures and sharing use of them) of the R+D+I National Plan 2008-2011. A guarantee of this proposal is that, in a first place, the Research Group (RG) has recently published the synthesis of expansible mica using a method that allows obtaining the desired layer charge in the material; secondly, the RG has a wide experience in the design of synthesis mechanisms of silicates as demonstrated by the number of papers published on this area during the last decade, and, finally, the RG has developed useful methodologies for the present Project in collaboration with other Groups with which it maintains a narrow scientific relationship.

Bioener: Aplicación de tecnologías biomiméticas a sistemas energéticos

Código/Code:	P09-TEP-5152 (Proyecto de Excelencia)
Periodo/Period:	02-02-2009 / 03-02-2014
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	311.167,68 €
Investigador responsable/Research head:	Julián Martínez Fernández
Componentes/Research group:	Antonio R. De Arellano-López, Alfonso Bravo León, Manuel Jiménez Melendo, F.M. Varela Feria, Joaquín Ramírez Rico, Antonio Gutiérrez Pardo

RESUMEN / ABSTRACT

Los avances tecnológicos han permitido diversificar y optimizar la producción de energía, lo que ha conducido al mismo tiempo, a desarrollar nuevas vías de almacenamiento de la misma. En particular, a medida que se diversifican los métodos de producción energética, es necesario desarrollar nuevos materiales para almacenamiento de energía, para su uso en dispositivos de consumo y transporte. Esto es especialmente importante para países que, como España, llevan años aumentando la presencia de las renovables en su carta de tecnologías de producción eléctrica y son actualmente líderes mundiales del sector. El Plan de Fomento de las Energías Renovables 2000-2010 tiene como objetivo para este año generar hasta el 30% de las necesidades de consumo energético nacionales a partir de energías renovables, mientras que en algunas comunidades autónomas la cuota de renovables supone ya más del 75% del consumo anual. La progresiva penetración de las renovables debe ir necesariamente acompañada del desarrollo de nuevas tecnologías de distribución y almacenamiento energético, teniendo en cuenta que la potencia suministrada por las renovables es intermitente y variable, al depender las condiciones meteorológicas.

Otra estrategia a corto plazo para reducir la dependencia de los combustibles fósiles es la mejora de la eficiencia los sistemas de generación de energía convencionales mediante el aumento de las temperaturas de operación, por ejemplo en turbinas de combustión de gas. El desarrollo de nuevos materiales para aplicaciones de alta temperatura ha ido tradicionalmente ligado a la demanda de mayores eficiencias. De entre ellos, los materiales cerámicos se postulan como candidatos para aplicaciones en entornos químicamente agresivos a temperaturas superiores a los 1000ºC. Carburos y nitruros en particular, con su elevado punto de fusión y excelentes propiedades termomecánicas, están siendo extensamente estudiados en la actualidad para este tipo de aplicaciones. Los cerámicos porosos también son de gran interés en aplicaciones energéticas, como intercambiadores de calor o sistemas de filtrado de gas de síntesis, entre otros.

De las líneas de investigación en nuevos materiales para almacenamiento energético, el desarrollo de nuevos sistemas de almacenamiento electroquímico tendrá un gran impacto a corto plazo, especialmente en el consumidor final: el desarrollo de baterías y condensadores electroquímicos de alta capacidad es clave para la viabilidad de tecnologías que, como los vehículos eléctricos, deberán implantarse en un corto plazo para reducir nuestra dependencia de los combustibles fósiles. Es por esta razón que los materiales para almacenamiento electroquímico han recibido gran atención de la comunidad científica y constituyen uno de los grandes retos tecnológicos actuales.

El carburo de silicio biomórfico (bioSiC) es un material cerámico obtenido por infiltración reactiva de preformas de carbono obtenidas mediante pirólisis de precursores vegetales. El precursor, generalmente madera, se mecaniza en bruto y se carboniza mediante un proceso de pirólisis en atmósfera controlada a alta temperatura, obteniéndose un material de carbono macroporoso (bioC) cuya microestructura es similar a la del precursor vegetal. Este precursor se mecaniza hasta una forma cercana al producto final y se infiltra reactivamente con silicio líquido o gaseoso para obtener un material de SiC/Si que presenta unas excelentes propiedades termomecánicas, con una microestructura que mimetiza la microestructura de la madera original. Es posible seleccionar las propiedades del material resultante mediante la elección adecuada del precursor. El silicio residual puede eliminarse para obtener un material de SiC macroporoso. Es posible, por último, reinfiltrar el bioSiC poroso con otros materiales, por ejemplo metales para obtener cermets bioSiC/Al o bioSiC/Cu.

La posibilidad de producir materiales de carbono macroporosos con nanoporosidad controlada es interesante para su uso en aplicaciones electroquímicas, puesto que es posible infiltrar o recubrir los macroporos con una fase secundaria que proporcione funcionalidad adicional, por ejemplo en pilas de litio con arquitectura tridimensional o en supercondensadores oxido/carbono. En este sentido, el desarrollo de nuevos carbonos con porosidad y/o estructura controlada puede abrir una puerta hacia nuevas arquitecturas y diseños de dispositivo capaces de almacenar mayores densidades de energía.

La mayoría de materiales nanoporosos de carbono utilizados en la actualidad se obtienen mediante la activación de carbones obtenidos por pirólisis de precursores orgánicos sintéticos, aunque en los últimos años el carbono obtenido a partir de carburos ha sido objeto de gran interés. Es posible, mediante el tratamiento a alta temperatura de carburos metálicos en una atmósfera clorada, obtener carbonos nanoporosos de alta pureza que resultan idóneos para aplicaciones electroquímicas. En este sentido ya se ha demostrado la posibilidad de obtener carbono nanoporoso a partir de carburos obtenidos por infiltración reactiva de precursores vegetales.

El objetivo de este proyecto es doble: por un lado, se estudiará en detalle el procedimiento de obtención de materiales de carbono a partir de precursores vegetales, prestando atención a la posibilidad de introducir distintos gases (CO2, vapor de agua) durante el proceso de pirólisis para obtener carbones activos con nanoporosidad para su uso en sistemas de almacenamiento electroquímico. Se estudiará además el efecto de la temperatura de pirólisis en el grado de cristalinidad, nanoporosidad, tamaño de cristal y estructura del carbono resultante, y se explorará la posibilidad de promover la cristalización del carbono mediante el uso de distintos catalizadores. Se caracterizará la microestructura de los materiales resultantes, así como sus propiedades físicas y termo-mecánicas.

Technological advances have made possible to diversify and optimize energy production, which in turn has motivated the development of new ways to store energy. In particular, as production methods diversify, it is necessary to develop new materials for energy storage, both large scale and in consumer devices and transportation. This is especially important in the context of higher penetration of renewable energies, which often depend on climatological conditions and require ways to store excess energy at production peaks, so it can be used when production decreases.

In parallel to this strategy and to reduce the share of fossil fuels in the overall energy production, it is necessary to increase the efficiency of conventional power generation systems, for example by increasing material's life and operating temperatures, for example in gas turbine systems, among others. The development of materials for high temperature applications, especially ceramics, has been traditionally linked to the search for increased efficiency of power generation systems. Ceramic materials, due to their high melting point, good creep resistance and resistance to corrosion, are seen as candidates for application in chemically aggressive environments at temperatures over 1000 ºC. Carbides and nitrides in particular are being studied extensively for this kind of applications. Porous ceramics are also of great interest in energy applications, such as heat exchangers or syngas filtration systems, among others.

Among active research lines in the development of new materials for energy storage, electrochemical storage is expected to have the largest impact in the end consumer, as the design of high capacity batteries and electrochemical capacitors is key for the viability of technologies such as plug-in electric cars. For this reason, research into new materials for electrochemical storage has become a strong focal point among the scientific community and constitutes one to the great technological challenges of today.

Biomorphic silicon carbide (bioSiC) is a ceramic material obtained by reactive infiltration of carbon performs derived by pyrolysis of natural precursors. The precursor, usually wood, is rough-machined and then converted to carbon by pyrolysis in a controlled atmosphere at high temperatures. The result is a macroporous carbon material (bioC) with a microstructure that closely resembles that of the original precursor. This carbon template is then machined to near net shape and is melt reacted with silicon either in liquid or vapor phase to obtain a SiC composite with some residual Si that shows excellent thermomechanical properties and a microstructure that closely mimics that of the original wood precursor. Tailoring the material's properties is possible by adequate selection of the precursor, which determines the microstructure and thus the properties of the bioSiC. It is also possible to remove the remaining silicon through chemical etching to obtain a macroporous SiC material which can then be reinfiltrated to create novel composites and cermets, such as bioSiC/Al or bioSiC/Cu.

The prospect of producing macroporous carbon materials with controlled nanoporosity is interesting for electrochemical applications, as it would be possible to infiltrate or coat macropores with a second phase the provides additional function, for instance in three dimensional lithium batteries or carbon/oxide supercapacitors. In this way, the development of new carbon materials with controlled structure and porosity could open the door to novel architectures and designs for devices able to store larger amounts of energy.

Most nanoporous carbon materials used nowadays are obtained through activation of carbons made from pyrolysis of synthetic precursors, although in the last years carbide-derived carbons have been the subject of great interest. It is possible to obtain high-purity nanoporous carbon through high temperature chlorination of metallic carbides, which rank among the best carbon materials for electrochemical applications. In this direction, is has already been shown that carbides obtained from natural precursors, such as bioSiC are viable precursors to carbide-derived carbons.

This proposal's aim is two-fold: on one side, the bioC processing will be studied in detail, paying special attention to precursor selection and to the possibility of introducing different atmospheres during the pyrolysis process, such as CO2 or water vapor, that promote nanoporosity in the material. The effect of processing parameter in the degree of crystallinity, nanoporosity, crystallite size and structure of the resulting carbon material will be assessed. The possibility of promoting carbon graphitization through the use of different catalysis in the pyrolysis process will be studied. The resulting carbon's microstructure and physical properties will be studied and correlated to the processing parameters.

On the other side, the effect of the aforementioned treatments on the bioSiC material will be studied, and the possibility of obtaining novel cermets in-situ, such as bioSiC/Al, bioSiC/Ti, through melt infiltration, will be assessed. In a last step, the possibility of obtained carbon materials with enhanced structure from the ceramic carbides will be explored.

CONVENIOS Y CONTRATOS / CONTRACTS AND AGREEMENTS

Caracterización de mecanismos de retención de actínidos en micas sintética: Aplicación de la retención de Cesio y Yodo

Periodo/Period:	01-10-2011 / 28-02-2013
Organismo Financiador/Financial source:	Empresa Nacional de Residuos Radioactivos ENRESA
importe total/Total amount:	96.250 €
Investigador responsable/Research head:	Miguel Angel Castro Arroyo
Componentes/Research group:	M. Dolores Alba Carranza, M. Mar Orta Cuevas, Said ElMrabet, M. Carolina Pazos Zarama, Maria Villa Alfageme, Santiago Hurtado

Encargo de Realización de tareas en el Proyecto de Investigación y Desarrollo

Periodo/Period:	29-07-2013 / 29-08-2013
Organismo Financiador/Financial source:	ABENGOA Bioenergía Nuevas Tecnologías, S.A.
importe total/Total amount:	6.352,50 €
Investigador responsable/Research head:	M. Mar Orta Cuevas
Componentes/Research group:	M. Dolores Alba Carranza, Miguel Angel Castro Arroyo

Waste to Biofuel: Producción de combustibles a partir de RSU

Periodo/Period:	04-07-2013 / 03-07-2013
Organismo Financiador/Financial source:	BEFESA Gestión de Residuios Industriales
importe total/Total amount:	4.840 €
Investigador responsable/Research head:	M. Mar Orta Cuevas
Componentes/Research group:	M. Dolores Alba Carranza, Miguel Angel Castro Arroyo

Materiales Vítreos Cementantes de Alta Eficiencia y Bajo Impacto Ambiental (MAVIT)

Periodo/Period:	01-01-2012 / 31-12-2014
Organismo Financiador/Financial source:	Refractarios ALFRAN, S.A.
importe total/Total amount:	181.500 €
Investigador responsable/Research head:	Joaquín Ramírez Rico
Componentes/Research group:	Julián Martínez Fernandez, María del Carmen Vera García, M. Dolores Alba Carranza

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

Solution Properties of the System $ZrSiO_4$–$HfSiO_4$: A Computational and Experimental Study
Cota, Agustin; Burton, Benjamin P.; Chain, Pablo; Pavon, Esperanza; Alba, Maria D.
Journal of Physical Chemistry C, **117** (2013) 10013-10019
Mayo, 2013 | **DOI:** 10.1021/jp401539g

$ZrSiO_4$ and $HfSiO_4$ are of considerable interest because of their low thermal expansions, thermal conductivities, and the optical properties of $HfSiO_4$. In addition, silicate phases of both are studied as model radioactive waste disposal materials. Previous first principles calculations reported near ideal mixing in the $Zr_{1-x}Hf_xSiO_4$ system, with a very weak propensity for phase separation. Density functional theory (DFT)/cluster-expansion first principles calculations presented in this work indicate near ideal mixing with a very weak propensity for ordering.
$Zr_{1-x}Hf_xSiO_4$ samples (x = 0, 0.25, 0.5, 0.75, and 1.0) were synthesized from intimate stoichiometric mixtures of constituent-oxides and annealing at 1823 K for 20 days in a platinum crucible. Samples were characterized by X-ray diffraction (XRD; Rietveld analysis) and ^{29}Si MAS NMR. The XRD data exhibited a pronounced negative deviation from Vegard's law in the excess volume of mixing, and the ^{29}Si MAS NMR spectra also suggest nonideal mixing. Given the very weak energetics that favor cation ordering, it is clear that there must be some other cause(s) for the observed deviations from ideal mixing behavior.

Evaluation of rare earth on layered silicates under subcritical conditions: Effect of the framework and interlayer space composition
Chain, P; Cota, A; El Mrabet, S; Pavon, E; Pazos, MC; Alba, MD
Chemical Geology, **347** (2013) 208-216
Junio, 2013 | **DOI:** 10.1016/j.chemgeo.2013.03.006

Clay-based minerals are considered to be an important component in backfill barriers due to both their ability to seal and adsorb radioactive waste and to interact chemically with it under subcritical conditions. Herein, we describe a systematic study of the properties of layered silicates that could affect their hydrothermal reactivity, namely type of layers, octahedral occupancy, origin and total amount of the layer charge, and nature of the interlayer cation. The silicates studied were selected on the basis of their different characteristics associated with these properties and were treated hydrothermally at 300 °C for 48 h in a $7.3 \cdot 10^{-2}$ M $Lu(NO_3)_3 \cdot 3.6H_2O$ solution. The final products were analyzed by X-ray diffraction and solid-state NMR spectroscopy. All the layered silicates studied were found to be able to generate a $Lu_2Si_2O_7$ phase after hydrothermal treatment under subcritical conditions, thereby confirming the participation of a chemical mechanism of the clay barrier generating phases being stables with temperature and pH conditions. However, the extent of this reaction depends to a large extent on the physicochemical properties of the framework and the interlayer space composition, such as the presence or absence of an octahedral sheet, the degree of occupancy of this sheet, and the origin and total layer charge. Therefore, this study allows tuning the clay mineral framework characteristic that favors the rare earth cations (as trivalent actinide simulator) immobilization.

Thermal conductivity of wood-derived graphite and copper–graphite composites produced via electrodeposition

Johnson, MT; Childers, AS; Ramirez-Rico, J; Wang, H; Faber, KT
Composites Part A: Applied Science and Manufaturing, **63** (2013) 182-189
Octubre, 2013 | **DOI:** 10.1016/j.compositesa.2013.06.009

The thermal conductivity of wood-derived graphite and graphite/copper composites was studied both experimentally and using finite element analysis. The unique, naturally-derived, anisotropic porosity inherent to wood-derived carbon makes standard porosity-based approximations for thermal conductivity poor estimators. For this reason, a finite element technique which uses sample microstructure as model input was utilized to determine the conductivity of the carbon phase independent of porosity. Similar modeling techniques were also applied to carbon/copper composite microstructures and predicted conductivities compared well to those determined via experiment.

Modeling Macro-Sized, High Aspect Ratio Through-Hole Filling by Multi-Component Additive-Assisted Copper Electrodeposition

Childers, AS; Johnson, MT; Ramirez-Rico, J; Faber, KT
Journal of the Electrochemical Society, **160** (12) D3093-D3102
Diciembre, 2013 | **DOI:** 10.1149/2.018312jes

A multi-element, time-dependent model is used to examine additive-assisted copper electroplating in macro-channels. This model is an adaptation of the work of Akolkar and Landau [J. Electrochem. Soc., 156, D351 (2009)], used to describe plating in micro-vias for integrated circuits. Using their method for describing species movement in the channel, the model has been expanded to include transport and adsorption limitations of the inhibitor and accelerator, as well as the copper ions in solution. The model is used to investigate copper plating as an infiltration method across many size scales and aspect ratios. Biomorphic graphite scaffolds produced from wood are used as a representative system and the results of a two-additive bath are used to characterize the behavior of the additives and determine the effectiveness of the plating. The results indicate that at macro-scales, channel dimensions play an increasingly important role in dictating the behavior of additive-assisted plating. Because additive systems are designed to establish differential surface coverage within the channel, the success of which is determined by the additive's rates of diffusion and adsorption, certain size scale/aspect ratio combinations preclude such coverage. A guide for sample geometries that may be successfully infiltrated with a two-additive bath is provided.

Joining and interface characterization of in situ reinforced silicon nitride

Asthana, R; Singh, M; Martinez-Fernandez, J
Journal of Alloys and Compounds, **552** (2013) 137-145
Marzo, 2013 | **DOI:** 10.1016/j.jallcom.2012.09.104

Copper-base active metal interlayers were used to bond in situ reinforced silicon nitride (Honeywell AS800) at 1317 K for 5 and 30 min in vacuum. The joints were characterized using scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), electron back scattered diffraction (EBSD), and transmission electron microscopy (TEM). A Ti-rich interaction zone (~3.0–3.5 µm thick) formed at the Si3N4/braze interface. This reaction layer grew toward the inner part of the joint with a featureless microstructure, creating a strong bond. Regions of a Ti-rich phase were frequently found next to the reaction layer but surrounded by the Cu alloy. Extensive Ti and Si enrichments were noted at the interface but there was no evidence of interfacial segregation of Y, La, and Sr (from Y_2O_3, La_2O_3 and SrO, added as sintering aids). The reaction layer thickness and composition did not change when brazing time increased from 5 min to 30 min suggesting rapid growth kinetics in the early stages of reaction. The joints were crack-free and showed features associated with plastic deformation, which indicated that the metal interlayer accommodated strain associated with CTE mismatch. The inner part of the joint consisted of highly textured large grains of the braze alloy.

Effects of thermal and mechanical treatments on montmorillonite homoionized with mono- and polyvalent cations: Insight into the surface and structural changes

Fernandez, M; Alba, MD; Sanchez, RMT

Colloids and Surfaces A: Physicochemical and Engineering Aspects, **423** (2013) 1-10
Abril, 2013 | **DOI:** 10.1016/j.colsurfa.2013.01.040

Smectite is a family of clay minerals that have important applications. In the majority of these clay minerals, the hydrated interlayer cations play a crucial role on the properties of the clay. Moreover, many studies have revealed that both thermal and grinding treatments affect the MMT structure and that interlayer cations play an important role in the degradation of the structure, primarily after mechanical treatment. In this study, the effects of these treatments on MMTs homoionized with mono (Na+, Li+ or K+) or polyvalent (Ca2+ or Al3+) cations were analyzed by the combination of a set of techniques that can reveal the difference of bulk phenomena from those produced on the surface of the particles. The thermal and mechanical (in an oscillating mill) treatments affected the framework composition and structure of the MMT, and the thermal treatment caused less drastic changes that the mechanical one. The effect of the interlayer cations is primarily due to the oxidation state and, to the size of the cations, which also influenced the disappearance of aluminum in the MMT tetrahedral sheet. These treatments caused a decrease in the surface area and an increase in the particle agglomeration and the isoelectric point. Both treatments caused the leaching of the framework aluminum. Furthermore, the mechanical treatment induced structural defects, such as the breakup of the particles, which favored the dehydroxylation and the increase of the isoelectric points of the montmorillonites.

Hydration properties of synthetic high-charge micas saturated with different cations: An experimental approach

Pavon, E; Castro, MA; Naranjo, M; Orta, MM; Pazos, MC; Alba, MD

American Mineralogist, **98** (2013) 394-400
Marzo, 2013 | **DOI:** 10.2138/am.2013.4217

An understanding of the interaction mechanisms between exchangeable cations and layered silicates is of interest from both a basic and an applied point of view. Among 2:1 phyllosilicates, a new family of swelling high-charge synthetic micas has been shown to be potentially useful as decontaminant. However, the location of the interlayer cations, their acidity and the water structure in the interlayer space of these silicates are still unknown. The aim of this paper was therefore to study the hydration state of the interlayer cations in the interlayer space of high-charge expandable micas and to evaluate the effect that this hydration has on the swelling and acidity behavior of these new materials. To achieve these objectives, three synthetic micas with different charge density total layer charges (ranging between 2 and 4 per unit cell) and with five interlayer cations (Na+, Li+, K+, Mg2+, and Al3+) were synthesized and their hydration state, interlayer space, and acidity analyzed by DTA/TG, XRD, and 1H MAS NMR spectroscopy. The results showed that the hydration state depends on both the layer charge and the nature of the interlayer cation. A high participation of the inner-sphere complexes in the highly charged confined space has been inferred and proposed to induce Brønsted acidity in the solid.

Synthesis and characterization of kanemite from fluoride-containing media: Influence of the alkali cation
Corredor, JI; Cota, A; Pavon, E; Alba, MD
American Mineralogist, **98** (2013) 1000-1007
Marzo, 2013 | **DOI:** 10.2138/am.2013.4372

Kanemite belongs to the group of naturally occurring sodium silicate minerals that was first found in Kanem, at the edge of the Lake Chad, and has been synthesized in different ways from NaOH-SiO_2 mixtures and used as precursor for the design of microporous and mesoporous materials. The fluoride route to the synthesis of microporous materials is based on the substitution of OH− anions by fluoride anions, which may subsequently also play a mineralizing role, and gives rise to materials with higher hydrophobicity and thermal and hydrothermal stability. Moreover, F− plays an important role in the incorporation of framework heteroatoms, thereby affecting the activity of the final material. The aim of this study was to synthesize fluorokanemite using different synthetic routes and different F− source. The final product was characterized by a combination of methods that provided information regarding the incorporation of fluorine into the framework and the short- and long-range structural order of the fluorosilicate. Kanemite with water content close to ideal was obtained in all cases. The washing process was found to have no effect in the long- or short-range structural order of the layer framework, although it did affect the structure of the cation in the interlayer space of kanemite. The mineralizing agent therefore appears to be the key to the synthesis. Furthermore, it governs the resulting kanemite structure by controlling the formation of hydrogen bonds in the framework, and therefore the degree of lamellar structure condensation.

Monolayer arrangement of fatty hydroxystearic acids on graphite: Influence of hydroxyl groups
Medina, S; Benitez, JJ; Castro, MA; Cerrillos, C; Millan, C; Alba, MD
Thin Solid Films, **539** (2013) 194-200
Julio, 2013 | **DOI:** 10.1016/j.tsf.2013.05.053

Previous studies have indicated that long-chain linear carboxylic acids form commensurate packed crystalline monolayers on graphite even at temperatures above their melting point. This study examines the effect on the monolayer formation and structure of adding one or more secondary hydroxyl, functional groups to the stearic acid skeleton (namely, 12-

hydroxystearic and 9,10-dihydroxystearic acid). Moreover, a comparative study of the monolayer formation on recompressed and monocrystalline graphite has been performed through X-ray diffraction (XRD) and Scanning Tunneling Microscopy (STM), respectively. The Differential Scanning Calorimetry (DSC) and XRD data were used to confirm the formation of solid monolayers and XRD data have provided a detailed structural analysis of the monolayers in good correspondence with obtained STM images. DSC and XRD have demonstrated that, in stearic acid and 12-hydroxystearic acid adsorbed onto graphite, the monolayer melted at a higher temperature than the bulk form of the carboxylic acid. However, no difference was observed between the melting point of the monolayer and the bulk form for 9,10-dihydroxystearic acid adsorbed onto graphite. STM results indicated that all acids on the surface have a rectangular p2 monolayer structure, whose lattice parameters were uniaxially commensurate on the a-axis. This structure does not correlate with the initial structure of the pure compounds after dissolving, but it is conditioned to favor a) hydrogen bond formation between the carboxylic groups and b) formation of hydrogen bonds between secondary hydroxyl groups, if spatially permissible. Therefore, the presence of hydroxyl functional groups affects the secondary structure and behavior of stearic acid in the monolayer.

Thermopower of Bio-SiC and SiC/Si ecoceramics prepared from sapele tree wood
Smirnov, IA; Smirnov, BI; Orlova, TS; Sulkovski, C; Misiorek, H; Muha, J; Jezowski, A; Ramirez-Rico, J; Martinez-Fernandez, J
Physics of the Solid State, **55** (2013) 54-59
Enero, 2013 | **DOI:** 10.1134/S1063783413010307

The thermopower coefficients of bio-SiC and SiC/Si ecoceramics prepared from sapele tree wood have been measured in the temperature interval 5–300 K. The measurements have been performed both along and perpendicular to empty (bio-SiC), as well as empty and partially silicon-filled (SiC/Si) channels in the samples. In bio-SiC, a contribution to thermopower associated with electron drag by phonons has been shown to exist within the temperature interval 5–200 (250) K. No such effect is realized in SiC/Si. This is assumed to derive from the presence in this material of heavily doped silicon embedded in SiC channels and the dominant part it plays in the behavior of the thermopower of this ceramics. The results obtained for the thermopower are compared with the available data for bio-SiC prepared from white eucalyptus tree wood and heavily doped bismuth.

Structure-mediated transition in the behavior of elastic and inelastic properties of beach tree bio-carbon
Kardashev, BK; Orlova, TS; Smirnov, BI; Gutierrez, A; Ramirez-Rico, J
Physics of the Solid State, **55** (2013) 1884-1891
Septiembre, 2013 | **DOI:** 10.1134/S1063783413090151

Microstructural characteristics and amplitude dependences of the Young modulus E and of internal friction (logarithmic decrement δ) of bio-carbon matrices prepared from beech tree wood at different carbonization temperatures T carb ranging from 600 to 1600°C have been studied. The dependences E(T carb) and δ(T carb) thus obtained revealed two linear regions of increase of the Young modulus and of decrease of the decrement with increasing carbonization temperature, namely, $\Delta E \sim A\Delta T$ carb and $\Delta\delta \sim B\Delta T$ carb, with $A \approx 13.4$ MPa/K and B ≈ −2.2 × 10−6 K−1 for T carb < 1000°C and A ≈ 2.5 MPa/K and B ≈ −3.0 × 10−7 K−1 for T carb > 1000°C. The transition observed in the behavior of E(T carb) and δ(T carb) at T carb = 900–1000°C can be assigned to a change of sample microstructure, more specifically, a change in the ratio of the fractions of the amorphous matrix and of the nanocrystalline phase. For T carb < 1000°C, the elastic properties are governed primarily by the amorphous matrix, whereas for T carb > 1000°C the nanocrystalline phase plays the dominant part. The structurally induced transition in the behavior of the elastic and microplastic characteristics at a temperature close to 1000°C correlates with the variation of the physical properties, such as electrical conductivity, thermal conductivity, and thermopower, reported in the literature.

Heat capacity of Bio-SiC and SiC/Si ecoceramics prepared from white eucalyptus, beech, and sapele tree wood

Smirnov, IA; Smirnov, BI; Orlova, TS; Wlosewicz, D; Hackemer, A; Misiorek, H; Mucha, J; Jezowski, A; Ramirez-Rico, J; Martinez-Fernandez, J

Physics of the Solid State, **55** (2013) 454-460

Febrero, 2013 | **DOI:** 10.1134/S1063783413020285

This paper reports on measurement of the heat capacity at constant pressure C p of silicon biocarbide prepared within the 5–300 K temperature interval from beech tree wood (bio-SiC(BE)), and within 80–300 K, from tree wood of sapele (bio-SiC(SA)), as well as SiC/Si ecoceramics of beech, sapele, and white eucalyptus wood. It has been shown that in bio-SiC(BE) the measured heat capacity contains a significant contribution of surface heat capacity, whose magnitude decreases with increasing temperature. Of the ecoceramics, only SiC/Si(SA) characterized by a high enough porosity has revealed a small contribution to the heat capacity coming from its surface component. The experimental results obtained are discussed.

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

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

European Conference on Advanced Materials and Processes 2013 - EUROMAT

8 – 13 septiembre [Sevilla, España]

Julián Martínez Fernández [Comité Organizador]

COMUNICACIONES EN CONGRESOS Y REUNIONES / COMMUNICATIONS IN CONGRESS AND MEETINGS

37th International Conference and Expo and Advanced Ceramics and Composites

27 enero – 1 febrero [Daytona Beach, Florida, Estados Unidos de América]

Modification of the electric properties in carbonaceous materials by deposition of SnO2 nanowires via CVD
Gutierrez-Pardo, A., R. Fiz, S. Mathur, and J. Ramirez-Rico
Poster

European Conference on Advanced Materials and Processes 2013 - EUROMAT

8 – 13 septiembre [Sevilla, España]

Thermomechanical properties and wear resistance of sintered SiC-fiber bonded ceramics
Vera, M.C., J. Ramirez-Rico, J. Martinez-Fernandez, and M. Singh
Comunicación oral

Microstructure, mechanical properties and sliding wear resistance of biomorphic SiC ceramics
Vera, M.C., J. Ramirez-Rico, J. Martinez-Fernandez, and M. Singh
Comunicación oral

Graphitized carbons obtained through catalysis during the pyrolysis process
Gutierrez-Pardo, A., J. Ramirez-Rico, and J. Martinez-Fernandez
Comunicación oral

Properties modification of C and SiC based materials by CVD of metal oxides
Gutierrez-Pardo, A., R. Fiz, J. Ramirez-Rico, J. Martinez-Fernandez
Comunicación oral
Biomorphic porous SiC obtained from gas infiltration of carbonized wood precursors
Cabezas-Rodriguez, R., J. Ramirez-Rico, and J. Martinez-Fernandez
Poster

Creep of vanadium carbonitride
F.A. Huamán-Mani, M.A. Roldán, C. Real, M. Jiménez-Melendo
Poster

FORMACION / TRAINING

FORMACIÓN DE GRADUADOS / MASTER DEGREE THESIS

Título:	**Materiales Cerámicos basados en el Sistema Ternario Alcalino Térreo-Circonio-Silicato**
Autor:	María del Mar García Mestre
Directores:	M. Dolores Alba Carranza
Grado:	Trabajo Fin de Master
Año Académico:	2012-2013 (20 Febrero 2013)

Título:	**Fijación de Cesio por Micas de Alta Carga**
Autor:	Francisco Javier Osuna Barroso
Directores:	M. Dolores Alba Carranza
Grado:	Trabajo Fin de Master
Año Académico:	2012-2013 (9 Julio 2013)

DOCENCIA / TEACHING

Licenciatura en Ciencias Químicas
Experimentación avanzada en química inorgánica
Dra. M. Dolores Alba Carranza
Lugar: Universidad de Sevilla

Máster en Ciencia, Tecnología y Uso Radional del Medicamento
Análisis y Control de Calidad de Materias Primas
Dra. María del Mar Orta Cuevas
Lugar: Universidad de Sevilla

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

MECANOQUÍMICA Y REACTIVIDAD DE MATERIALES
MECHANO-CHEMISTRY AND REACTIVITY OF MATERIALS

PERSONAL / PERSONNEL

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Dr. José Manuel Criado Luque

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Profesores Titulares

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Lda. Cristina García Garrido

Personal Contratado

Lda. M. Rocío Rodríguez Laguna

PROYECTOS DE INVESTIGACIÓN / RESEARCH PROJECTS

Estudio de los procesos de degradación de los materiales utilizados en la construcción de Órganos Históricos Study of the degradation processes on the materials used in the manufacture of historical organs

Código/Code:	MAT2010-20660 (Plan Nacional)
Periodo/Period:	01-01-2011 / 31-12-2013
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	48.400 €
Investigador responsable/Research head:	Angel Justo Erbez
Componentes/Research group:	Adolfo Iñigo Iñigo, Juan Poyato Ferrera, José Luis Pérez Rodríguez, Liz Karen Herrera Quintero, Angel Justo Estebaranz, Adrián Durán Benito, Carmen Jiménez de Haro, Belinda Sigüenza Carballo

RESUMEN / ABSTRACT

El principal objetivo del proyecto es conocer la composición, microestructura y propiedades mecánicas de las aleaciones estaño-plomo de tubos de órganos históricos españoles. Además se estudiarán los procesos de degradación y corrosión sobre los tubos, así como los productos generados por dichos procesos. Los análisis se realizarán en diferentes Institutos Científicos y Tecnológicos españoles (ICMSE, AIMEN, IRNASA) y grandes instalaciones europeas (ESRF, C2RMF). Se persigue conocer los vectores que provocan los procesos de corrosión, como son los compuestos volátiles desprendidos de las maderas y otros materiales orgánicos utilizados en la construcción de órganos, vapor de agua y/o anhídrido carbónico.

Los trabajos se realizarán en materiales con diferentes grados de corrosión tomados en los propios instrumentos, incluyendo principalmente aleaciones metálicas y maderas. Además, se prepararán aleaciones en el taller de organería Grenzing con distintas proporciones estaño-plomo, y con trazas de otros elementos como As o Bi. Estas aleaciones se someterán a ensayos de corrosión acelerada y se correlacionarán los resultados de la composición y los ensayos de resistencia a la tracción y de fluencia con la velocidad de corrosión.

Se compararán los resultados obtenidos en el laboratorio (aleaciones preparadas) con las muestras procedentes de los órganos y se sacarán conclusiones acerca de las posibles causas de alteración, las composiciones idóneas para las restauraciones y las aleaciones más resistentes a la corrosión para aplicarlas a la construcción de órganos nuevos.

The main objective of the project is to know the composition, microstructure and mechanical properties of tin-lead alloys from Spanish historical pipe organs. Also, we will study the degradation and corrosion processes on the pipe organs and the products of corrosion produced by these processes. Analyses will be performed in Spanish research and technological institutes (ICMSE, AIMEN, IRNASA) and European facilities (ESRF, C2RMF). This objective pursues to know the vectors that produce the corrosion, like the volatile compounds from the wood and other organic materials used in the construction of pipe organs, water vapour and/or carbon dioxide.

The work will be carried out in materials with different grades of corrosion taken in the organs, including alloys and woods. Also, alloys with different tin-lead ratios, and with traces of other elements (As, Bi) will be prepared and will be undergone to corrosion tests. Results from the composition and results of tensile and creep tests will be correlated with the corrosion rate.

The results obtained in the laboratory will be compared with the samples coming from the organs, and conclusions will be reached about the possible alteration causes, the suitable compositions for the restorations, and the most resistant alloys to the corrosion, to apply them to the construction of new organs.

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MINISTERIO DE ECONOMÍA Y COMPETITIVIDAD

Diseño por procedimientos mecanoquímicos de materiales estructurales para aplicaciones tecnológicas de alta temperatura

Mechanochemical design of structural materials for high-temperature technological applications

Código/Code:	MAT2011-22981
Periodo/Period:	01-01-2012 / 31-12-2014
Organismo Financiador/Financial source:	Ministerio de Ciencia e Innovación
Importe total/Total amount:	80.000 €
Investigador responsable/Research head:	Francisco J. Gotor Martínez
Componentes/Research group:	M. Jesús Sayagués de Vega, Concepción Real Pérez, M. Dolores Alcalá González, Pedro José Sánchez Soto, José Manuel Córdoba Gallego, Ernesto Chicardi Augusto

RESUMEN / ABSTRACT

Los carburos, nitruros y boruros de los metales de transición son componentes esenciales de un gran número de materiales compuestos empleados para fines estructurales y de protección a altas temperaturas. Esto es debido a una excelente combinación de propiedades físicas y químicas que les confiere una buena resistencia mecánica, al desgaste, a la oxidación y a la corrosión. Los materiales basados en estos compuestos refractarios se vienen diseñando con un importante carácter multifásico, condicionado por la alta multifuncionalidad que se les exige y por la imposibilidad de alcanzar las propiedades requeridas a partir de un único compuesto.

Durante el procesado de estos materiales es frecuente observar importantes gradientes de composición e interacciones entre las distintas fases constitutivas que dificultan alcanzar las propiedades deseadas. En el presente proyecto, pretendemos abordar un nuevo diseño para este tipo de materiales consistente en incorporar la mayor parte de sus componentes

esenciales como soluciones sólidas complejas. Esto permitirá reducir el número final de fases en el material y obtener con mayor garantía de éxito las propiedades preestablecidas para las diversas aplicaciones tecnológicas. Para ello, planteamos una nueva ruta de síntesis basada en el proceso mecanoquímico denominado reacción de auto-propagación inducida mecánicamente (MSR), ya que nuestro grupo ha demostrado que este método permite obtener de manera sencilla soluciones sólidas pertenecientes a los sistemas MT-B-C-N con un elevado control de la estequiometría. El objetivo principal del presente proyecto consiste en incorporar el método MSR a la metodología empleada para el desarrollo de materiales constituidos por soluciones sólidas que puedan ser utilizados en aplicaciones de alta temperatura. Se pretende caracterizar adecuadamente las propiedades de los materiales obtenidos y compararlas con aquellos fabricados con la metodología hasta ahora empleada.

Carbides, nitrides and borides of transition metals are essential components of a large number of composite materials used for structural and protective applications at high temperature because they show an excellent combination of physical and chemical properties, which confers good mechanical strength, and wear, oxidation and corrosion resistances. The materials based on these refractory compounds are designed by employing multiphasic systems, due to the high multi-functionality that are required and the inability to achieve the intended properties from a single phase material.

During the processing of these materials is common to observe important compositional gradients and interactions between the different constituent phases that hinder achieving the desired properties. In this project, we intend to undertake a new design for this type of material of incorporating most of its key components such as complex solid solutions. This will reduce the final number of phases in the material and obtain greater assurance of success with the preset properties for technological applications. To this end, we propose a new synthesis route based on the mechanochemical process called as mechanically-induced self-sustaining reaction (MSR). Our research group has shown that this method allows obtaining solid solutions belonging to M-B-C-N systems with a high control of the stoichiometry. The main objective of this project is to incorporate the method MSR to the methodology used for the development of materials consisting of solid solutions that can be used in high temperature applications. It is intended to adequately characterize the properties of the developed materials and to compare them with those made using the methods so far employed.

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Procesado de cerámicas avanzadas a partir de precursores poliméricos (PDC) por métodos de control inteligente de la temperatura Processing of advanced ceramics from polymeric precursors by smart temperature methods

Código/Code:	CQT2011-27626
Periodo/Period:	01-01-2012 / 31-12-2014
Organismo Financiador/Financial source:	Ministerio de Economía y Ciencia
Importe total/Total amount:	66.550 €
Investigador responsable/Research head:	Luis A. Pérez Maqueda

Componentes/Research group: Maria Jesús Diánez, José Manuel Criado, Pedro E. Sánchez Jiménez, Antonio Perejón Pazo

RESUMEN / ABSTRACT

Los materiales cerámicos preparados a partir de precursores poliméricos, más conocidos por las siglas PDC (polymer derived ceramics), constituyen un tema de gran interés actual. Estos materiales se preparan a partir de un polímero, que se somete a un proceso de curado previo a su descomposición térmica a temperaturas relativamente suaves si se comparan con las requeridas en un procesado cerámico convencional a partir de precursores en polvo. Este método presenta la ventaja de conducir directamente al producto final consolidado sin requerir el complejo proceso de compactación requerida por los métodos convencionales de procesado cerámico. Es por lo tanto un método "near-net shape". Estos materiales presentan propiedades eléctricas, termomecánicas y de resistencia a la oxidación muy interesantes que le confieren un amplio potencial de aplicaciones que abarcan desde la nanotecnología a la aeronáutica. Sin embargo, una limitación importante en la síntesis de estos materiales radica en la dificultad de controlar la velocidad de descomposición térmica de la pieza precerámica polimérica de modo que no se produzcan defectos, tales como fracturas, que hacen inservible el material. En este proyecto se propone el uso de los métodos de control inteligente de temperatura para el procesado de estos precursores poliméricos precerámicos. En estudios previos hemos puesto de manifiesto las ventajas de esta metodología para controlar la estructura y microestructrura de productos preparados a partir de transformaciones térmicas de precursores y para estudios cinéticos de reacciones en estado sólido. 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, con especial hincapié en el estudio de las cinéticas de los procesos involucrados. Los productos obtenidos se caracterizarán en cuanto a su nanoestructura y propiedades, en particular el coeficiente piezoeléctrico, la capacidad de inserción de litio y la resistencia a la oxidación.

Ceramic materials prepared from polymer precursors, known as polymer-derived ceramics (PDC) are a subject of the most interest. These materials are prepared from a polymer that is first cured and then ceramified, usually by thermal treatment at relatively low temperature if compared with those needed in conventional ceramic processing from ceramic powders. Thus, the final product is directly obtained in a near-net shape process. These materials have very interesting electrical, thermomecanical and oxidation resistance properties. Thus, a number of applications from nanotechnology to aeronautics have been proposed. Nevertheless, a significant limitation of the use of these materials is is related with the ceramification process of the preceramic piece. During this thermal conversion, some defects, such as cracks, appear in the pieces. In this project, we propose the use of smart temperature controlled methods for the processing of the preceramic polymeric precursors. In previous studies, we have shown the advantages of this methodology for controlling the structure and microstructure of the products prepared by thermal transformation of precursors. In addition, this methodology is also useful for kinetic studies of solid state reactions. In the present project, we

expect to obtain defect free PDC materials and to study the influence of the preparation conditions on the nanostructure of the products and get new insights in polymer to ceramic conversion process, paying special attention to the study of the kinetics of the involved processes. The so-obtained products will be characterized in terms of their nanostructure and properties, in particular piezoresistivity, lithium insertion capacity and oxidation resistance.

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 / 15-05-2015
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.

CONVENIOS Y CONTRATOS / CONTRACTS AND AGREEMENTS

Composol-Desarrollo de nuevos componentes para la tecnología solar cilindro parabólico para muy altas temperaturas

Código/Code:	20124462
Periodo/Period:	25-11-2011 / 24-11-2014
Organismo Financiador/Financial source:	Proyectos e Implantación de Tuberias, SA
Importe total/Total amount:	319.440 €
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

Desarrollo de un receptor cerámico de alta temperatura y presión para sistemas solares híbridos ("CERSOL")

Código/Code:	20135212
Periodo/Period:	26-04-2013 / 31-12-2014
Organismo Financiador/Financial source:	Abengoa Solar New Technologies, S.A.
Importe total/Total amount:	278.300 €
Investigador responsable/Research head:	Luis A. Pérez Maqueda
Componentes/Research group:	Pedro E. Sánchez Jiménez, M. Jesús Diánez Millán, José Manuel Criado Luque, Antonio Perejón Pazo, Cristina García Garrido, M. Rocio Rodríguez Laguna

Nuevo concepto de planta de torre con fluidos a muy alta temperatura y mayores rendimientos de ciclo respecto a los actuales

Código/Code:	20135581
Periodo/Period:	09-04-2013 / 31-12-2014
Organismo Financiador/Financial source:	Abengoa Research, S.L.
Importe total/Total amount:	42.350 €
Investigador responsable/Research head:	Luis A. Pérez Maqueda
Componentes/Research group:	Pedro E. Sánchez Jiménez, M. Jesús Diánez Millán, José Manuel Criado Luque, Cristina García Garrido, M. Rocio Rodríguez Laguna

PATENTES / PATENTS

Nanoestructuras Biocidas

Inventores: María Jesús Sayagués de Vega, Rocio Polvillo Hernández y José Luis Royo Sánchez-Palencia
Tipo de Patente **Patente Española con número de solicitud 201330444**
Fecha Solicitud (Fecha de presentación): 26.03.2013
Entidad/es Titular/es: Universidad Pablo de Olavide y CONSEJO SUPERIOR DE INVESTIGACIONES CIENTÍFICAS (CSIC)

Method for obtaining nanostructures actives with visible light and short exposure times of radiation, are capable of inhibiting the growth of algae and bacteria in colloidal suspensions and surfaces. Nanostructures obtained, have applications as a biocide in cooling towers to prevent the spread of Legionella, in health facilities, in cleaning products, as a preservative in food, in plants of water treatment, etc.

Method for producing metallic nanoparticles functionalized with fluorescent organic molecules

Inventores: Ana Paula Zaderenko Partida, Carlos Caro Salazar, José Mejías Romero, Maria Jesús Sayagués De Vega
Tipo de Patente **Patente Española con número de solicitud 14/007546**
Fecha Solicitud (Fecha de presentación): 25.09.2013
Entidad/es Titular/es: Universidad Pablo Olavide y CONSEJO SUPERIOR DE INVESTIGACIONES CIENTÍFICAS (CSIC)

The present invention relates to a method of obtaining metal nanoparticles functionalized with fluorescent organic molecules by means of the treatment of one or more metal salts with a reducing agent in the presence of a fluorescent organic molecule. The invention also relates to the metal nanoparticles obtainable by said method and to their use for detecting molecules and analytes.

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

Hallmarks of mechanochemistry: from nanoparticles to technology
Balaz, P; Achimovicova, M; Balaz, M; Billik, P; Cherkezova-Zheleva, Z; Criado, JM; Delogu, F; Dutkova, E; Gaffet, E; Gotor, FJ; Kumar, R; Mitov, I; Rojac, T; Senna, M; Streletskii, A; Wieczorek-Ciurowa, K
Chemical Society Reviews, **42** (2013) 7571-7637
Septiembre, 2013 | **DOI:** 10.1039/C3CS35468G

The aim of this review article on recent developments of mechanochemistry (nowadays established as a part of chemistry) is to provide a comprehensive overview of advances achieved in the field of atomistic processes, phase transformations, simple and multicomponent nanosystems and peculiarities of mechanochemical reactions. Industrial aspects with successful penetration into fields like materials engineering, heterogeneous catalysis and extractive metallurgy are also reviewed. The hallmarks of mechanochemistry include influencing reactivity of solids by the presence of solid-state defects, interphases and relaxation phenomena, enabling processes to take place under non-equilibrium conditions, creating a well-crystallized core of nanoparticles with disordered near-surface shell regions and performing simple dry time-convenient one-step syntheses. Underlying these hallmarks are technological consequences like preparing new nanomaterials with the desired properties or producing these materials in a reproducible way with high yield and under simple and easy operating conditions. The last but not least hallmark is enabling work under environmentally friendly and essentially waste-free conditions (822 references).

Constant rate thermal analysis for enhancing the long-term CO_2 capture of CaO at Ca-looping conditions
Valverde, JM; Sanchez-Jimenez, PE; Perejon, A; Perez-Maqueda, LA
Applied Energy, **108** (2013) 108-120
Agosto, 2013 | **DOI:** 10.1016/j.apenergy.2013.03.013

Experimental results are reported on the (Ca-looping) multicyclic CO2 capture of CaO and nanosilica/CaO composites derived from Ca(OH)2 and nanosilica/Ca(OH)2 dry mixtures subjected in situ to linear and constant rate thermal analysis (CRTA) preheating programs in either air or air/CO2 atmospheres. By means of CRTA preheating the rates of the reactions taking place during pretreatment are kept at a constant and small value along the entire process. In agreement with a pore skeleton model, previously proposed in the literature for explaining the behavior of natural limestones thermally pretreated, our results suggest that air/CO2-CRTA pretreatment yields a thermally stable hard skeleton of poorly reactive CaO on which a soft skeleton of reactive CaO would be supported. The sorbent subjected to this preheating program exhibits a reactivation in the very first carbonation/calcination cycles, after which CaO conversion decays slowly with the cycle number. In contrast, linearly or air-CRTA preheated sorbents show a significant decrease of CaO conversion within the first cycles. In the latter case, CaO multicyclic conversion fits well to a model where it is assumed that the progressive reduction of surface area as the number of carbonation/calcination cycles is increased obeys to sintering of the preheated sorbent skeleton as it is subjected to repeated calcinations during cycling. In the former case, CaO conversion data conforms to the prediction by a model in which the loss of surface area is mainly due to sintering of a nascent CaO soft skeleton regenerated in the diffusive carbonation phase, which is enhanced by the air/CO2-CRTA pretreatment. As regards the effect of nanosilica, the results indicate that it slows down CaO sintering during pretreatment, which hinders the development of a stable CaO skeleton thus hampering reactivation and stabilization of conversion. On the other hand, as CaO sintering is also lessened during looping calcination, nanosilica is useful to increase the absolute values of CaO conversion.

Arsenic sorption by nanocrystalline magnetite: An example of environmentally promising interface with geosphere

Bujnakova, Z; Balaz, P; Zorkovska, A; Sayagues, MJ; Kovac, J; Timko, M
Journal of Hazardous Materials, **262** (2013) 1204-1212
Noviembre, 2013 | **DOI:** 10.1016/j.jhazmat.2013.03.007

In this paper, the sorption of arsenic onto nanocrystalline magnetite mineral Fe_3O_4 was studied in a model system. Nanocrystalline magnetite was produced by mechanical activation in a planetary ball mill from natural microcrystalline magnetite. As a consequence of milling, the specific surface area increased from 0.1 m^2/g to 11.9 m^2/g and the surface site concentration enhanced from 2.2 sites/nm2 to 8.4 sites/nm2. These changes in surface properties of magnetite lead to the enhancement of arsenic removal from model system. The best sorption ability was achieved with magnetite sample activated for 90 min. In this case the sample was able to absorb around 4 mg/g. The structural changes of magnetite were also observed and the new hematite phase was detected after 120 min of milling. A good correlation between the decreasing particle size, increasing specific surface area and reduction of saturation magnetization was found. In desorption study, KOH and NaOH were found as the best eluents where more than 70% of arsenic was released back into the solution. The principal novelty of the paper is that mineral magnetite, truly one nature's gift can be used after “smart” milling (mechanical activation) as an effective arsenic sorbent.

CO_2 multicyclic capture of pretreated/doped CaO in the Ca-looping process. Theory and experiments

Valverde, JM; Sanchez-Jimenez, PE; Perejon, A; Perez-Maqueda, LA
Physical Chemistry Chemical Physics, **15** (2013) 11775-11793
Julio, 2013 | **DOI:** 10.1039/C3CP50480H

We study in this paper the conversion of CaO-based CO_2 sorbents when subjected to repeated carbonation–calcination cycles with a focus on thermally pretreated/doped sorbents. Analytical equations are derived to describe the evolution of conversion with the cycle number from a unifying model based on the balance between surface area loss due to sintering in the looping-calcination stage and surface area regeneration as a consequence of solid-state diffusion during the looping-carbonation stage. Multicyclic CaO conversion is governed by the evolution of surface area loss/regeneration that strongly depends on the initial state of the pore skeleton. In the case of thermally pretreated sorbents, the initial pore skeleton is highly sintered and regeneration is relevant, whereas for nonpretreated sorbents the initial pore skeleton is soft and regeneration is negligible. Experimental results are obtained for sorbents subjected to a preheating controlled rate thermal analysis (CRTA) program. By applying this preheating program in a CO_2 enriched atmosphere, CaO can be subjected to a rapid carbonation followed by a slow rate controlled decarbonation, which yields a highly sintered skeleton displaying a small conversion in the first cycle and self-reactivation in the next ones. Conversely, carbonation of the sorbent at a slow controlled rate enhances CO_2 solid-state diffusion, which gives rise, after a quick decarbonation, to a highly porous skeleton. In this case, CaO conversion in the first cycle is very large but it decays abruptly in subsequent cycles. Data for CaO conversion retrieved from the literature and from further experimental measurements performed in our work are analyzed as influenced by a variety of experimental variables such as preheating temperature program, preheating exposition time, atmosphere composition, presence of additives, and carbonation–calcination conditions. Conversion data are well fitted by the proposed model equations, which are of help for a quantitative interpretation of the effect of experimental conditions on the multicyclic sorbent performance as a function of sintering/regeneration parameters inferred from the fittings and allow foreseeing the critical conditions to promote reactivation. The peculiar behavior of some pretreated sorbents, showing a maximum conversion in a small number of cycles, is explained in light of the model.

Spark plasma sintering of TixTa$_{1-x}$C$_{0.5}$N$_{0.5}$-based cermets: Effects of processing conditions on chemistry, microstructure and mechanical properties

Cordoba, Jose M.; Chicardi, Ernesto; Poyato, Rosalia; Gotor, Francisco J.; Medri, Valentina; Guicciardi, Stefano; Melandri, Cesare

Chemical Engineering Journal, **230** (2013) 558-566

Agosto, 2013 | **DOI:** 10.1016/j.cej.2013.06.104

Nanometric powdered $Ti_xTa_{1-x}C_{0.5}N_{0.5}$-based cermets were fabricated using a mechanically induced self-sustaining reaction and consolidated by spark plasma sintering. Highly dense cermets were obtained, and their chemistry, microstructure and mechanical properties were characterised by X-ray diffraction, scanning electron microscopy, image analysis, microindentation and nanoindentation. The microhardness was found to depend directly on the contiguity and size of the ceramic hard particles. The samples synthesised at the lowest temperature (1150 °C) exhibited more homogeneous microstructures and smaller ceramic particles and the best combination of microhardness and fracture toughness.

Studies of isothermal crystallisation kinetics of sunflower hard stearin-based confectionery fats

Bootello, MA; Hartel, RW; Levin, M; Martinez-Blanes, JM; Real, C; Garces, R; Martinez-Force, E; Salas, JJ

Food Chemistry, **139** (2013) 184-195

Agosto, 2013 | **DOI:** 10.1016/j.cej.2013.06.104

The crystallisation and polymorphic properties of three sunflower hard stearins (SHSs) and cocoa butter equivalents (CBEs) formulated by blending SHSs and palm mid fraction (PMF) were studied and compared with those from cocoa butter (CB), to explore their possibilities as confectionery fats. The isothermal crystallisation kinetics of these fats were examined by pNMR and DSC at three different temperatures. All samples studied displayed a two-step crystallisation profile that could be fitted to an exponential-Gompertz equation. Stop-and-return DSC studies showed that SHSs and CBEs exhibited different crystallisation mechanisms according to their triacylglycerol composition, with a quick formation of metastable crystals, followed by a polymorphic transition to the more stable β or β′ forms. X-ray diffraction (XRD) was used to investigate the polymorphic forms of tempered SHSs and CBEs in the long term. In all cases the resulting fats displayed short spacing patterns associated with β polymorphism. These formulations based on SHSs and PMF met all the requirements to be considered as CBEs; therefore they could be used as an alternative to traditional confectionery fats.

Reversible reactions of Ni and Pd hydroxo pincer complexes [(iPrPCP)M-OH] with CO_2: Solid-state study of the decarboxylation of the monomeric bicarbonate complexes [(i PrPCP)M-OCOOH] (M = Ni, Pd)

Martinez-Prieto, LM; Real, C; Avila, E; Alvarez, E; Palma, P; Campora, J
European Journal of Inorganic Chemistry, **32** (2013) 5555-5566
Noviembre, 2013 | **DOI:** 10.1002/ejic.201300995

Monomeric Ni and Pd hydroxides stabilized by the iPrPCP pincer ligand react with CO_2 to give labile terminal bicarbonate complexes that readily lose CO_2 and water to give binuclear carbonate complexes. Differential scanning calorimetry (DSC) has been used to monitor the decomposition of both bicarbonates in the solid state. When the carbonate complexes are heated under reflux in thf in the presence of water, full decarboxylation takes place, restoring the starting hydroxides and demonstrating that CO_2 insertion is a fully reversible process. The decarboxylation of the nickel carbonate complex is completed more readily, suggesting that the reaction of the Pd hydroxide with CO_2 is more favourable than that of its nickel counterpart. This is supported by DFT calculations, which also shows that CO_2 insertion takes place through a concerted Lipscomb-type mechanism. Monomeric Ni and Pd hydroxides stabilized by the iPrPCP pincer ligand react with CO_2 to give labile terminal hydrogen carbonate complexes that readily lose CO_2 and water to give binuclear carbonate complexes.

Role of Looping-Calcination Conditions on Self-Reactivation of Thermally Pretreated CO_2 Sorbents Based on CaO

Valverde, JM; Sanchez-Jimenez, PE; Perejon, A; Perez-Maqueda, LA
Energy & Fuels, **27** (2013) 3373-3384
Junio, 2013 | **DOI:** 10.1021/ef400480j

The conversion of thermally pretreated CaO along successive carbonation/calcination cycles has been investigated, as affected by looping-calcination conditions, by means of Thermogravimetric Analysis (TGA). Sorbent samples have been subjected in situ to a thermal preheating program based on Constant Rate Thermal Analysis (CRTA) by virtue of which decarbonation is carried out at a low controlled rate, which is able to promote self-reactivation in the first carbonation/calcination cycles. Our observations support a pore-skeleton model according to which solid-state diffusion in the first carbonation stages, which is enhanced by thermal pretreatment, gives rise to a soft skeleton with increased surface area. Yet, the results show that self-reactivation is hindered as looping-calcination conditions are harshened. Increasing the looping-calcination temperature and/or the looping calcination time period favors sintering of the soft skeleton and eventually self-reactivation is precluded. A model is developed that retrieves the main features of multicyclic conversion of thermally pretreated sorbents in the first cycles based on the balance between surface area gain due to promoted solid-state diffusion carbonation and surface area loss due to sintering of the soft skeleton in the looping-calcination stage, which can be useful to investigate the critical looping-calcination conditions that nullify self-reactivation. The proposed model allows envisaging the behavior of the sorbent performance as a function of the pretreatment conditions.

Liquid-phase sintering of Ti(C,N)-based cermets. The effects of binder nature and content on the solubility and wettability of hard ceramic phases

Cordoba, JM; Chicardi, E; Gotor, FJ
Journal of Alloys and Compounds, **559** (2013) 34-38
Mayo, 2013 | **DOI:** 10.1016/j.jallcom.2013.01.046

Different commercial TiC–TiN/Co/Ni mixtures were used as raw materials for Ti(C,N) cermets, and the effects of the sintering parameters (binder content, binder nature, sintering time and additives) on the final hard ceramic phase were studied at the sintering temperature of 1400 °C. When Co is used as the binder medium, it is possible to completely convert the starting commercial TiC–TiN mixture into TiC_xN_{1-x}. When Ni is used, which exhibits lower solubilising capacity than Co, the total conversion can never be reached and the metallurgical reactions between TiC and TiN during the liquid-phase sintering are more dependent on the sintering time than on the binder content. However, the use of Co–Ni mixtures, showing a synergic effect between the wettability capacity of Ni and the solubilising capacity of Co, enhances the metallurgical reactions at short sintering times.

Mechanochemical synthesis of ZrB_2–SiC–ZrC nanocomposite powder by metallothermic reduction of zircon

Jalaly, M; Tamizifar, M; Bafghi, MS; Gotor, FJ
Journal of Alloys and Compounds, **581** (2013) 782-787
Diciembre, 2013 | **DOI:** 10.1016/j.jallcom.2013.07.142

Aluminium and magnesium were used in the $M/ZrSiO_4/B_2O_3/C$ (M = Al, Mg) system to induce a mechanically induced self-sustaining reaction (MSR). Aluminium was not able to reduce the system to the desired products, and the system became amorphous after 10 h milling. However, nanocomposite powder of ZrB_2–SiC–ZrC was in situ synthesized by the magnesiothermic reduction with an ignition time of approximately 6 min. The mechanism for the formation of the product in this system was determined by studying the relevant sub-reactions.

Limitations of model-fitting methods for kinetic analysis: Polystyrene thermal degradation

Sanchez-Jimenez, PE; Perez-Maqueda, LA; Perejon, A; Criado, JM
Resources, Conservation and Recycling, **74** (2013) 75-81

Mayo, 2013 | **DOI:** 10.1016/j.resconrec.2013.02.014

In this paper, some clarifications regarding the use of model-fitting methods of kinetic analysis are provided in response to the lack of plot linearity and dispersion in the activation energy values for the thermal degradation of polystyrene found in the literature and some results proposing an nth order model as the most suitable one. In the present work, two model-fitting methods based on the differential and integral forms of the general kinetic equation are evaluated using both simulated and experimental data, showing that the differential method is recommended due to its higher discrimination power. Moreover, the intrinsic limitations of model-fitting methods are highlighted: the use of a limited set of kinetic models to fit experimental data and the ideal nature of such models. Finally, it is concluded that a chain scission model is more appropriate than first order.

Historic preservation, GIS, & rural development: The case of Almería province, Spain
Cano, M; Garzon, E; Sanchez-Soto, PJ
Applied Geograhpy, **42** (2013) 34-47
Agosto, 2013 | **DOI:** 10.1016/j.apgeog.2013.04.014

A computerized database was created, based on a Geographic Information System (GIS), with hyperlinks to the website for a Rural Development Association (Almería province, Andalusia, Spain). Thus, a catalogue of traditional rural buildings in this particular area was compiled, identifying and characterizing each one, establishing criteria for a dynamic and rational selection. The purpose to select this example was to facilitate their management by public organizations or private individuals, for their reuse, restoration or both. The cataloguing and promotion of rural architecture will contribute to creating jobs by stimulating new economic activity, such as the promotion of cultural tourism, while preserving a valuable source of information on rural culture, recovering local construction techniques, encouraging a sense of community, and making villages and rural areas more attractive to visitors. The assessment of the rehabilitation potential of rural buildings in this region has helped to establish a priority order for their reuse, and so an intervention map has been devised in terms of a "Decision Index" corresponding to each considered building.

Comments on "Thermal decomposition of pyridoxine: an evolved gas analysis-ion attachment mass spectrometry study". About the application of model-fitting methods of kinetic analysis to single non-isothermal curves
Sanchez-Jimenez, PE; Perez-Maqueda, LA; Perejon, A; Criado, JM
Rapid Communications in Mass Spectrometry, **27** (2013) 500-502
Febrero, 2013 | **DOI:** 10.1002/rcm.6472

No abstract is available for this article.

Pyrolysis kinetics of ethylene–propylene (EPM) and ethylene–propylene–diene (EPDM)
Perejon, A; Sanchez-Jimenez, PE; Gil-Gonzalez, E; Perez-Maqueda, LA; Criado, JM
Polymer Degradation and Stability, **98** (2013) 1571-1577

Septiembre, 2013 | **DOI:** 10.1016/j.polymdegradstab.2013.06.029

The thermal degradation kinetics of several ethylene–propylene copolymers (EPM) and ethylene–propylene–diene terpolymers (EPDM), with different chemical compositions, have been studied by means of the combined kinetic analysis. Until now, attempts to establish the kinetic model for the process have been unsuccessful and previous reports suggest that a model other than a conventional nth order might be responsible. Here, a random scission kinetic model, based on the breakage and evaporation of cleavaged fragments, is found to describe the degradation of all compositions studied. The suitability of the kinetic parameters resulting from the analysis has been asserted by successfully reconstructing the experimental curves. Additionally, it has been shown that the activation energy for the pyrolysis of the EPM copolymers decreases by increasing the propylene content. An explanation for this behavior is given. A low dependence of the EPDM chemical composition on the activation energy for the pyrolysis has been reported, although the thermal stability is influenced by the composition of the diene used.

Mechanochemical synthesis of Sb_2S_3 and Bi_2S_3 nanoparticles
Dutkova, E; Takacs, L; Sayagues, MJ; Balaz, P; Kovac, J; Satka, A
Chemical Engineering Science, **85** (2013) 25-29
Enero, 2013 | **DOI:** 10.1016/j.ces.2012.02.028

The mechanochemical synthesis of Sb_2S_3 and Bi_2S_3 nanoparticles has been studied, starting from the corresponding metals and sulfur and using high-energy mechanochemical processing in a planetary laboratory mill. XRD, specific surface area measurement, SEM and TEM (HRTEM) with ED were used for the characterization of the nanoparticles. The XRD patterns confirmed the production of Sb_2S_3 (JCPDS 42–1393, orthorhombic) and Bi_2S_3 (JCPDS 17–320, orthorhombic) nanopowders. The transformation is about three times faster in the Bi–S than in the Sb–S system. The kinetics of the reaction has been determined from XRD line intensities. The grain size is about 30 nm for Sb_2S_3 and 24 nm for Bi_2S_3. The particles are highly agglomerated due to their nanometer size consequent large specific surface area. Unlike more conventional methods, mechanochemical synthesis is a simple and fast alternative for the preparation of these nanopowders that can be carried out at ambient temperature and atmospheric pressure.

Electrical Properties of Stoichiometric BiFeO3 Prepared by Mechanosynthesis with Either Conventional or Spark Plasma Sintering
Perejon, A; Maso, N; West, AR; Sanchez-Jimenez, PE; Poyato, R; Criado, JM; Perez-Maqueda, LA
Journal of the American Ceramic Society, **96** (2013) 1220-1227
Abril, 2013 | **DOI:** 10.1111/jace.12186

Phase-pure powders of stoichiometric $BiFeO_3$ have been prepared by mechanosynthesis. Ceramics sintered by either conventional heating in air or spark plasma sintering (SPS) followed by oxidative anneal in air are highly insulating at room temperature with resistivity, extrapolated from the Arrhenius plots, of ~1016 Ωcm and activation energy 1.15(2) eV, comparable with those of a good-quality $BiFeO_3$ single crystal. By contrast, the as-prepared SPS sample without the postsinter anneal shows lower resistivity, e.g., ~1010 Ωcm at 25°C and activation energy 0.67(3) eV, indicating some reduction in the sample by the SPS process. The reason for the high conductivity reported for some ceramic samples in the literature remains unclear at present.

Cystine-capped CdSe@ZnS nanocomposites: mechanochemical synthesis, properties, and the role of capping agent
Balaz, M; Balaz, P; Tjuliev, G; Zubrik, A; Sayagues, MJ; Zorkovska, A; Kostova, N
Journal of Materials Science, **48** (2013) 2424-2432
Marzo, 2013 | **DOI:** 10.1007/s10853-012-7029-3

Cystine-capped CdSe@ZnS nanocomposites were synthesized mechanochemically with the aim to prepare a material which could be used in medicine for biosensing applications. Although synthesized CdSe@ZnS nanocomposites were capped with l-cysteine, cystine was formed from l-cysteine during the milling process. It was proven that water plays the key role in this oxidative transformation. The novel material was characterized by the complex of physico-chemical methods (FTIR, XPS, SEM, EDX, surface area measurements) and CHNS analysis. The leakage of Cd^{2+} and Zn2+ ions into physiological solution was also studied.

Formation mechanism of ZrB2–Al2O3 nanocomposite powder by mechanically induced self-sustaining reaction
Jalaly, M; Bafghi, MS; Tamizifar, M; Gotor, FJ
Journal of Materials Science, **48** (2013) 7557-7567
Noviembre, 2013 | **DOI:** 10.1007/s10853-013-7571-7

ZrB_2–Al_2O_3 nanocomposite powder was produced by aluminothermic reduction in $Al/ZrO_2/B_2O_3$ system. In this research, high energy ball milling was used to produce the necessary conditions to induce a mechanically induced self-sustaining reaction. The ignition time of the composite formation was found to be about 13 min. The synthesis mechanism in this system was investigated by examining the corresponding sub-reactions as well as changing the stoichiometry of reactants. Thermal behavior of the system was also studied.

Influence of the milling parameters on the mechanical work intensity in planetary mills
Gotor, FJ; Achimovicova, M; Real, C; Balaz, P
Powder Technology, **233** (2013) 1-7
Enero, 2013 | **DOI:** 10.1016/j.powtec.2012.08.031

The formation of ZnSe via a mechanically-induced self-sustaining reaction (MSR) from a Zn/Se mixture showed that only size reduction and mixing of the reactants without product formation occurred during the induction period prior to ignition. Therefore, all mechanical energy supplied by the planetary mill during this time, called the ignition time (tig), was used exclusively in the activation of the reactants. This system was chosen to study the dependence of tig on the main parameters characterising the milling intensity of planetary mills. The variation of the ignition time with the process conditions reflected changes in the mechanical dose rate of the planetary mill. A direct relationship between the inverse of the ignition time and the power of the planetary mill was established, which allows the validation of theoretical models proposed in the literature for the energy transfer in milling devices and the comparison of milling equipment efficiencies.

Characterization and repair measures of the medieval building materials of a Hispanic–Islamic construction
Pineda, P; Robador, MD; Perez-Rodriguez, JL
Construction and Building Materials, **41** (2013) 612-633
Abril, 2013 | **DOI:** 10.1016/j.conbuildmat.2012.12.034

This paper focuses on the physical–chemical and mechanical characterization of the building materials of a precious damaged structure, the Salares tower, a Hispanic–Islamic medieval construction located in the South of Spain, an active seismic area. An exhaustive description of the materials has been obtained, enriching the knowledge on historical construction materials. Sophisticated laboratory tests (XRD, SEM, EDX, FTIR, Micro-Raman spectroscopy, DTA and TG) have provided crucial complementary data to use together with traditional laboratory procedures, especially when the damage processes are the main concern. The experimental investigation has allowed a better understanding of the remote origin and of the failure mechanisms of this damaged structure. On the basis of these results, the most adequate repair materials are selected.

Kinetic approach to partially overlapped thermal decomposition processes
Koga, N; Goshi, Y; Yamada, S; Perez-Maqueda, LA
Journal of Thermal Analysis and Calorimetry, **111** (2013) 1463-1474
Febrero, 2013 | **DOI:** 10.1007/s10973-012-2500-6

Practical usefulness of the kinetic deconvolution for partially overlapped thermal decomposition processes of solids was examined by applying to the co-precipitated basic zinc carbonate and zinc carbonate. Comparing with the experimental deconvolutions by thermoanalytical techniques and mathematical deconvolutions using different statistical fitting functions, performance of the kinetic deconvolution based on an accumulative kinetic equation for the independent processes overlapped partially was evaluated in views of the peak deconvolution and kinetic evaluation. Two-independent kinetic processes of thermal decompositions of basic zinc carbonate and zinc carbonate were successfully deconvoluted by means of the thermoanalytical measurements in flowing CO2 and by applying sample controlled thermal analysis (SCTA). The deconvolutions by the mathematical curve fittings using different fitting functions and subsequent formal kinetic analysis provide acceptable values of the mass-loss fractions and apparent activation energies of the respective reaction processes, but the estimated kinetic model function changes depending on the fitting functions employed for the peak deconvolution. The mass-loss fractions and apparent kinetic parameters of the respective reaction processes can be optimized simultaneously by the kinetic deconvolution based on the kinetic equation through nonlinear least square analysis, where all the parameters indicated acceptable correspondences to those estimated through the experimental and mathematical deconvolutions. As long as the reaction processes overlapped are independent kinetically, the simple and rapid procedure of kinetic deconvolution is useful as a tool for characterizing the partially overlapped kinetic processes of the thermal decomposition of solids.

Kinetic studies in solid state reactions by sample-controlled methods and advanced analysis procedures

Perez-Maqueda, LA; Criado, JM; Sanchez-Jimenez, PE; Perejon, A
Journal of Thermal Analysis and Calorimetry, **113** (2013) 1447-1453
Septiembre, 2013 | **DOI:** 10.1007/s10973-013-3114-3

A comparative study of both conventional rising temperature and sample-controlled methods, like constant rate thermal analysis (CRTA), is carried out after analyzing a set of solid state reactions using both methods. It is shown that CRTA avoids the influence of heat and mass transfer phenomena for a wide range of sample sizes leading to reliable kinetic parameters. On the other hand, conventional rising temperature methods yield α–T plots dependent on experimental conditions, even when using samples sizes smaller than 2 mg. Moreover, it is shown that the discrimination of overlapping processes is dramatically improved using sample-controlled methods instead of conventional heating procedures. An advanced method for performing the kinetic analysis of complex processes from a single CRTA experiment is proposed.

The Ozawa's generalized time concept and YZ-master plots as a convenient tool for kinetic analysis of complex processes

Malek, J; Koga, N; Perez-Maqueda, LA; Criado, JM
Journal of Thermal Analysis and Calorimetry, **113** (2013) 1437-1446
Septiembre, 2013 | **DOI:** 10.1007/s10973-013-2939-0

The concept of generalized time θ=∫exp(−Ea/RT)dt θ = ∫ exp (− E a / RT) d t in non-isothermal kinetics was introduced by Ozawa in 1965, together with the well-known isoconversional plot, i.e., Ozawa plot. The generalized time is the key concept to tie the kinetic data under varying temperature to the kinetic relationship at a constant temperature. It is well known that many processes studied by thermal analysis and calorimetry reveal a complex nature. Therefore, the generalized time concept seems to be very useful for the description of the change in the rate behavior depending on the fractional conversion. Using the concept of θ, three kinds of experimental master plots can be formalized in differential, integral, and multiplied forms. Among others, combination of the differential and multiplied master plots, y(α) = (dα/dθ) and z(α) = (dα/dθ)θ, show a high performance to discriminate the kinetic model based on the maxima condition of y(α y*) and z(α z*). The α y* − α y* kinetic plot is a useful tool to visualize the complexity of the kinetic process and to determine the most suitable kinetic model. The usefulness of α y* − α y* kinetic plot and the YZ-master plots is illustrated as exemplified by the kinetic analyses of complex crystallization processes of the as-prepared, thermally and mechanically treated amorphous zirconia.

Allochthonous red pigments used in burial practices at the Copper Age site of Valencina de la Concepción (Sevilla, Spain): characterisation and social dimension

Rogerio-Candelera, MA; Herrera, LK; Miller, AZ; Sanjuan, LG; Molina, CM; Wheatley, DW; Justo, A; Saiz-Jimenez, C
Journal of Archaeological Science, **40** (2013) 279-290
Enero, 2013 | **DOI:** 10.1016/j.jas.2012.08.004

The use of red pigments linked to burial practices is widely documented in the Iberian prehistoric record and very often it has been traditionally interpreted as a ritual practice entailing the utilisation of local raw materials (iron oxides). Some research works, nevertheless, have also detected the use of red pigments which can only be interpreted as allochthonous. The red pigments spread over a single inhumation in a monumental Megalithic tomb surrounding Valencina de la Concepción Copper Age settlement was studied by means of X-ray diffraction, field emission scanning electron microscopy with energy dispersive X-ray spectroscopy, X-ray microfluorescence, micro-Raman and Fourier transform infrared spectroscopies. This approach allowed characterising the red pigments as cinnabar, mixed with tiny amounts of iron oxides. The presence of cinnabar, a product that was necessarily imported, in a context of an exceptional set of grave goods, suggests that the use of cinnabar was linked not only to ritual but also to practices related to the display of social status.

Generalized master plots as a straightforward approach for determining the kinetic model: The case of cellulose pyrolysis

Sanchez-Jimenez, PE; Perez-Maqueda, LA; Perejon, A; Criado, JM
Thermochimica Acta, **552** (2013) 54-59
Enero, 2013 | **DOI:** 10.1016/j.tca.2012.11.003

The thermal degradation of cellulose is a complex reaction and, despite the large amount of work by many investigators during the last decades, the actual understanding of the thermal decomposition kinetics is still very limited. Thus, while several mechanisms have been proposed to describe the process, the real model has not yet been clearly identified. In this paper, a set of experimental curves recorded under different heating schedules, i.e., linear heating rate, isothermal and constant rate thermal analysis (CRTA), has been analyzed using isoconversional and master plots methodology to discriminate the kinetic model followed by the reaction.

Enhanced general analytical equation for the kinetics of the thermal degradation of poly(lactic acid) driven by random scission

Carrasco, F; Perez-Maqueda, LA; Sanchez-Jimenez, PE; Perejon, A; Santana, OO; Maspoch, ML
Polymer Testing, **32** (2013) 937-945
Agosto, 2013 | **DOI:** 10.1016/j.polymertesting.2013.04.013

An enhanced general analytical equation has been developed in order to evaluate the kinetic parameters of the thermal degradation of poly(lactic acid) (PLA) at various linear heating rates and at constant rate conditions. This improvement consisted of replacing the n-order conversion function by a modified form of the Sestak-Berggren equation f(α) = c(1−α)nαm, which led to better adjustment of experimental data, and also adequately represented the conventional mechanisms for solid-state processes. The kinetic parameters so obtained have been compared to those determined by conventional differential and isoconversional methods. Given that the thermal degradation of PLA has been argued to be caused by random chain scission reactions of ester groups, the conversion function (α) = 2(α1/2−α), corresponding to a random scission mechanism, has been tested.

Effect of sintering time on the microstructure and mechanical properties of (Ti,Ta)(C,N)-based cermets
Chicardi, E; Torres, Y; Cordoba, JM; Sayagues, MJ; Rodriguez, JA; Gotor, FJ
International Journal of Refractory Metals and Hard Materials, **38** (2013) 73-80
Mayo, 2013 | **DOI:** 10.1016/j.ijrmhm.2013.01.001

Complete solid-solution cermets based on titanium–tantalum carbonitride using a starting nominal composition with 80 wt.% of (Ti0.8Ta0.2)(C0.5N0.5) and 20 wt.% of Co were performed by pressure-less sintering at 1550 °C for different times (from 0 to 180 min) in an inert atmosphere. Chemical and phase analyses were conducted using X-ray diffraction (XRD), elemental analysis and energy dispersive X-ray spectrometry (EDX). The binder mean free path and the contiguity of the carbonitride particles were used to rationalise the microstructural effects of the mechanical behaviour. Mechanical characterisation included determining the Vickers hardness, the fracture toughness (conventional indentation microfractures, IM), the dynamic Young's modulus (ultrasonic technique), the biaxial strength (ball on three ball) and a detailed fractographic examination. Finally, the experimental findings were combined with a theoretical fracture mechanics analysis to estimate the critical processing flaw sizes. Binder-less carbonitride clusters, pores and coarse carbonitride grains were the main defects observed and were responsible for the fractures.

Bio-inspired mechanochemical synthesis of semiconductor nanomaterial using eggshell membrane
Balaz, M; Balaz, P; Sayagues, MJ; Zorkovska, A
Materials Science in Semiconductor Processing, **16** (2013) 1899-1903
Diciembre, 2013 | **DOI:** 10.1016/j.mssp.2013.06.024

Eggshell membrane and lead acetate were successfully used as precursors for the mechanochemical synthesis of lead sulphide nanocrystals with crystallite sizes ∼8 nm. XRD, specific surface area measurements, SEM and EDX were used to characterise the synthesised material. The mechanochemical synthesis follows three-step mechanism. The “fish-like” grains with sizes around 30 µm were obtained.

Clarifications regarding the use of model-fitting methods of kinetic analysis for determining the activation energy from a single non-isothermal curve
Sanchez-Jimenez, PE; Perez-Maqueda, LA; Perejon, A; Criado, JM
Chemistry Central Journal, **7** (2013) 25
Febrero, 2013 | **DOI:** 10.1186/1752-153X-7-25

Background
This paper provides some clarifications regarding the use of model-fitting methods of kinetic analysis for estimating the activation energy of a process, in response to some results recently published in Chemistry Central journal.
Findings
The model fitting methods of Arrhenius and Savata are used to determine the activation energy of a single simulated curve. It is shown that most kinetic models correctly fit the data, each providing a different value for the activation energy. Therefore it is not really possible to determine the correct activation energy from a single non-isothermal curve. On the other hand, when a set of curves are recorded under different heating schedules are used, the correct kinetic parameters can be clearly discerned.
Conclusions
Here, it is shown that the activation energy and the kinetic model cannot be unambiguously determined from a single experimental curve recorded under non isothermal conditions. Thus, the use of a set of curves recorded under different heating schedules is mandatory if model-fitting methods are employed.

Colour and ultrasound propagation speed changes by different ageing of freezing/thawing and cooling/heating in granitic materials

Inigo, AC; Garcia-Talegon, J; Vicente-Tavera, S; Martin-Gonzalez, S; Casado-Marin, S; Vargas-Munoz, M; Perez-Rodriguez, JL

Cold Regions Science and Technology, **85** (2013) 71-78
Enero, 2013 | **DOI:** 10.1016/j.coldregions.2012.08.004

In the present work we determined the chromatic coordinates (L*, a*, b*) and ultrasound propagation speeds on the three spatial planes (Vx, Vy, Vz) of three ornamental granites (Aqueduct of Segovia, Spain) before, during, and after being subjected to 70 cycles of two types of accelerated ageing (typical of cold regions): a) freezing/thawing and cooling/heating (T1), and b) freezing/thawing and cooling/heating + salt crystallization (T2). A multivariate technique (Canonical Biplot) was applied to the data obtained, with the observation of significant variations between the two types of accelerated artificial ageing as compared with those obtained in quarry rock in the three chromatic coordinates (L*, a*, b*). With regard to the ultrasound propagation speed, we only detected differences in the results of the T2 artificial ageing treatment with respect to those of quarry rock. This fact is confirmed by the estimated data of resistance to compression.

Mechanosynthesis of nanocrystalline ZrB2-based powders by mechanically induced self-sustaining reaction method

Jalaly, M; Bafghi, MS; Tamizifar, M; Gotor, FJ

Advances in Applied Ceramics, **112** (2013) 383-388
Octubre, 2013 | **DOI:** 10.1179/1743676113Y.0000000091

Preparation of nanocrystalline ZrB2-based powder by aluminothermic and magnesiothermic reductions in M/ZrO2/B2O3 (M=Al or Mg) systems was investigated. In this research, high energy ball milling was employed to persuade necessary conditions for the occurrence of a mechanically induced self-sustaining reaction (MSR). The course of MSR reactions were recorded by a noticeable pressure rise in the system during milling. Ignition times for ZrB2 formation by aluminothermic and magnesiothermic reductions were found to be 13 and 6 min, respectively. Zirconium diboride formation mechanism in both systems was explained through the analysis of the relevant sub-reactions.

Preservation and Conservation of Rural Buildings as a Subject of Cultural Tourism: A Review Concerning the Application of New Technologies and Methodologies

Cano, M.; Garzón, E.; Sánchez Soto, P.J.
Journal of Tourism & Hospitality, **2:2** (2013) 1000115
DOI: 10.4172/2167-0269.1000115

The cataloguing and promotion of rural architecture contribute to creating jobs by stimulating new economic activity, such as the promotion of cultural tourism, while preserving a valuable source of information on rural culture, recovering local construction techniques, encouraging a sense of community, and making villages and rural areas more attractive to visitors.The general aim of the present Review is to analyse agricultural and rural buildings and their properties in order to identify key issues concerning sustainable reutilisation in tourism.
It is intended to Almeria province (Andalucía, Spain) a more complete sustainable tourism development framework to satisfy all the demands, from sun and beach at the coast as well as alternative tourism at the interior, based on the protection and the valuable re-utilization of popular rural architectural heritage with potential interest for tourism purposes. Thus, a catalogue of traditional rural buildings in a particular area was carried out, identifying and characterizing each one, establishing criteria for a dynamic and rational selection. The modelling of the lifecycle for the architectural project carried out, for the reuse of rural heritage, as a way of stimulating the tourism sector, is based on methods and risk analysis (statistical methods) and multicriteria analysis that allow the reduction in the subjectivity of the deterioration models carried out. Through the application of the Analytic Hierarchy Process (AHP), as a weighing method, a linear, weighting and additive model that combines all of the factors in one unique global assessment can be obtained, for which values have been assigned to the coefficients of the linear expression that reflect the relative importance of each factor.

Planning collection and solid waste flow (construction and demolition, concrete, ceramics and others) by utilizing a computerized tool for sustainable management

Garzon, E; Sanchez-Soto, PJ
Boletin de la Sociedad Española de Cerámica y Vidrio, **5** (2013) V-XIV (Notas Técnicas)
Septiembre, 2013 | **DOI:** 10.3989/cyv.2013.v52.i5

Se presenta un procedimiento para la planificación de recogida y flujo de los residuos sólidos (de construcción y demolición, hormigón, cerámica, vidrio y otros) basado en la utilización de una herramienta informatizada, para conseguir una optimización de su gestión. Dicho procedimiento parte de normativa establecida según un Plan Director Territorial de la Gestión de Residuos Sólidos Urbanos (RSU) aprobado en una Comunidad Autónoma, en este caso se

particulariza a la de Andalucía, tomando como ejemplo el volumen de residuos que se producen en una colectividad de tamaño medio (provincia de Almería), siendo extensible a otras mayores en población y territorio, disponiendo de datos actualizados.
El procedimiento utiliza una herramienta informática de gran difusión en el mundo, como es Google Earth y, de este modo, genera un número de "Centros deTransferencia" con objeto de minimizar el gasto de transporte, partiendo de una premisa previa en cuanto a distancia entre núcleos poblacionales y centros de tratamiento. Los Centros generados con la aplicación del procedimiento se pueden visualizar en un mapa topográfico, con áreas de influencia y vías de acceso a los mismos y se le pueden asociar una serie de datos tabulados con información adicional de utilidad. El procedimiento propuesto se va retroalimentando de manera constante con datos reales e información de campo, permitiendo a las empresas que producen residuos de distinta tipología como son los residuos de construcción y demolición principalmente, pero también hormigón, cerámica, vidrio, mezclas de todos ellos, residuos clasificados como peligrosos e incluso de otros materiales, a la propia administración y a la sociedad, en general, conocer las tasas de cada planta de tratamiento y qué se hace con los residuos entregados para contribuir a la reducción del impacto medioambiental de los mismos y a su gestión sostenible.

Porous Aluminas: The biotemplate method for the synthesis of stable high surface area aluminas

Guerrero, MB; Maqueda, LP; Castro, PP; Cosp, JP
Boletin de la Sociedad Española de Cerámica y Vidrio, **52** (2013) 251-267
Noviembre, 2013 | **DOI:** 10.3989/cyv.322013

Development of porous alumina has been the objective of numerous studies in recent decades, due to the intrinsic properties of aluminium oxide, such as high melting point, low thermal conductivity, chemical inertness and corrosion resistance which, in addition to a high surface area and permeability, make aluminas being used for many different industrial and technical applications. The crystallographic and textural stability of alumina acquires significant importance in those processes involving high temperatures; however, most of the synthesis methods yield metastable oxides of little interest in high-temperature processes due to the transformation to alpha phase, with the consequent reduction in surface area. The present article reviews diverse procedures for obtaining porous alumina with high specific surface area, including methods and strategies for preparing high surface alpha-alumina. Within this framework, the paper analyzes the results obtained through bioreplica of lignocellulosic materials. This technology allows preparing aluminas with the complex structural hierarchy of the lignocellulosic templates.

Estudio in-situ de la transformación térmica de limonita utilizada como pigmento procedente de Perú

Romero-Gomez, P; Gonzalez, JC; Bustamante, A; Ruiz-Conde, A; Sanchez-Soto, PJ
Boletin de la Sociedad Española de Cerámica y Vidrio, **52** (2013) 127-131
Mayo, 2013 | **DOI:** 10.3989/cyv.162013

Se ha realizado un estudio cinético de la transformación térmica de limonita [$FeO(OH).nH_2O$] mediante análisis térmico gravimétrico (TGA), termodifracción de rayos X (DRX) y espectroscopía µ-Raman. La muestra estudiada fue extraída de un yacimiento en el distrito de Taraco, provincia de Huancané, Región de Puno (Perú). La técnica DRX en polvo identificó la fase goetita como el principal componente mineralógico, además de cuarzo. La muestra se sometió a un tratamiento térmico in-situ en un intervalo de temperaturas de 100 a 500 °C en atmósfera de aire e inerte (nitrógeno) y se estudió por DRX. Los resultados han mostrado que la fase goetita permanece estable desde la temperatura ambiente hasta 200 °C. A partir de los 250 °C se produce una transformación de fase $\alpha\text{-}FeO(OH) \rightarrow \alpha\text{-}Fe_2O_3$ con un cambio cromático, es decir, el paso de la fase hidroxilada goetita (amarillo) a la fase oxidada hematites (rojo) con una pérdida de peso de un 8 %, teniendo como evidencia la evolución de los perfiles de difracción y los resultados de ATG. Los espectros µ-Raman del tratamiento térmico in-situ corroboran que se produce también una transición de fase a la temperatura de 290 °C a través de la transformación de las bandas Raman características de la fase goetita hacia la fase hematites en el rango de frecuencias de 200 a 1800 cm^{-1}.

Direct mechanosynthesis of pure $BiFeO_3$ perovskite nanoparticles: reaction mechanism
Perejon, A; Murafa, N; Sanchez-Jimenez, PE; Criado, JM; Subrt, J; Dianez, MJ; Perez-Maqueda, LA
Journal of Materials Chemistry C, **1** (2013) 3551-3562
Junio, 2013 | **DOI:** 10.1039/C3TC30446A

In this work, a mechanochemical procedure is proposed as a simple and fast method to synthesize the pure $BiFeO_3$ perovskite phase as a nanostructured material without the need for purification treatments, while the mechanochemical reaction mechanism has been investigated and correlated with that of the conventional solid-state reaction. Thus, different milling conditions have been used as a tool for tailoring the crystallite size of the resulting $BiFeO_3$ nanoparticles. The materials prepared by the mechanochemical reaction could be annealed or sintered without the formation of undesirable phases. Both the ferroelectric and ferromagnetic transitions were observed by DSC. Finally, the dielectric constants of the prepared material at different frequencies as a function of the temperature have been measured, showing that the material is clearly an isolator below 200 °C, characteristic of a high quality $BiFeO_3$ material.

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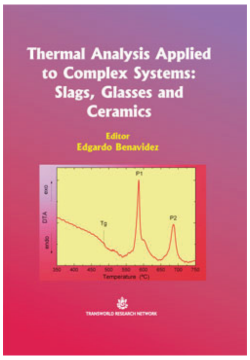

Sample controlled thermal analysis (SCTA): Applications to the kinetic analysis of solid state reactions and the synthesis of materials
J.M. Criado; L.A. Pérez-Maqueda; P.E. Sánchez-Jiménez; M.J. Diánez
En: Thermal Analysis Applied to Complex Systems: Slags, Glasses and Ceramics, (2013) 109-142 (Editor Edgardo Benavidez) Editorial Transworld Research Network
ISBN: 978-81-7895-588-9

Las patentes de invención como medio para la promoción y registro de los resultados científico-tecnológicos de investigación
Díaz-Hernández, J.L.; Miranda-Hernández, J.M.; Sánchez-Soto, P.J.
Acta Científica y Tecnológica, **1** (2013) 17-27

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

COMUNICACIONES EN CONGRESOS Y REUNIONES / COMMUNICATIONS IN CONGRESS AND MEETINGS

Electronic Materials and Applications 2013

23 - 25 enero [Orlando, Florida, Estados Unidos de América]

Electrical properties of stoichiometric $BiFeO_3$ prepared by mechanosynthesis with either conventional or spark plasma sintering
A. Perejon; L.A. Perez-Maqueda; N. Maso; A. R. West
Comunicación oral

International Congress on Particle Technology - PARTEC 2013

23 - 25 abril [Nuremberg, Alemania]

A CaO/nano-silica composite powder with enhanced CO2 capture capacity and mechanical stability
J. M. Valverde; M.A.S. Quintanilla; A. Perejón; L. A. Perez-Maqueda; J. M. P. Ebri; J. Perez-Vaquero
Comunicación oral

18th PLANSEE SEMINAR

3 - 7 junio [Reutte, Austria]

Hot press sintering of hard material master alloys synthesized in situ by mechanically induced self-sustaining reaction
J.M. Córdoba, E. Chicardi, Z. Lences and F.J. Gotor

Design, processing and mechanical behaviour of Cermet/WC-Co laminates
F. J. Gotor, J. M. Córdoba, E. Chicardi, Y. Torres, S. Guicciardi and V. Medri

Mechanical strength of WC-Co: Influence of microstructure and testing configuration
Y. Torres, R. Bermejo, F. J. Gotor, L. M. Llanes and E. Chicardi

8th International Conference on Chemical Kinetics

8 – 12 julio [Sevilla, España]

Intelligent control of the reaction temperature: Applications to the kinetic analysis of solid state reactions and the synthesis of materials
Pérez-Maqueda, L.A.; Criado, J.M.; Sánchez-Jiménez, E.; Perejón, A.; Diánez, M.J.
Poster

17th International Congress on Project Management and Engineering

17 – 19 julio [La Rioja, España]

Modelo para la evaluación del potencial de rehabilitación de la arquitectura
Garzón Garzón, E.; Cano García, M.; Sánchez Soto, P.J.
Conferencia invitada

Efectos del cemento y de la cal en muestras de materiales que contienen filitas procedentes del sur de España
Garzón Garzón, E.; Cano García, M.; Morales Hernández, L.; Sánchez Soto, P.J.
Comunicación oral

European Congress and Exhibition on Advanced Materials and Processes – EUROMAT 2013

8 – 13 septiembre [Sevilla, España]

Fabrication of mullite ceramics and mullite-based composite materials from kaolinite and aluminium metal wastes
Pascual Cosp, J.; Benítez-Guerrero, M.; Ruiz-Conde, A.; Garzón, E.; Cano, M.; Moreno-Megías, V.; Sánchez-Soto, P.J.
Comunicación oral

Ceramics from clays and galvanic sludges: processing, properties and microstructural characterization
Péres-Villarejo, L.; Martínez-Martínez, S.; Eliche-Quesada, D.; Corpas-Iglesias, F.A.; Garzón-Garzón, E.; Ruiz-Conde, A.; Sánchez-Soto, P.J.
Poster

Characterization by XRD, XRF, OM, SEM-EDX, Thermal Analysis, FTIR and Raman Spectroscopies of ancient ceramics (IV-II Centuries b.c.) from selected archaeological sites at the Guadalquivir Valley
Moreno-Megias, V.; García-Fernández, F.J.; Ferrer, E.; Ruiz-Conde, A.; Martín del Río, J.J.; Sánchez-Soto, P.J.
Poster

Creep of vanadium carbonitride
Huaman-Mamani, F.A.; Roldán, M.A.; Real, A.; Jiménez-Melendo, M.
Poster

Multilayered hard metal composites conformed by conventional powder metallurgical methods
J.M. Córdoba, E. Chicardi, M.J. Sayagués, Y. Torres and F J. Gotor

Effects of carbon addition on the mechanical properties of (TixTa1-x)(CyN1-y)-Co cermets
E. Chicardi, J.M. Córdoba, Y. Torres, M.J. Sayagués and F.J. Gotor

19th International Vacuum Congress – IVC 19
9 – 13 septiembre [Paris, Francia]

Mechanochemical treatment of layer silicates: preparation of nanostructured materials with enhanced surface properties and reactivity
P.J. Sanchez-Soto, M.A. Avilés, A. Ruiz-Conde, F.J. Gotor, E. Garzón, J. Pascual
Poster

CONGRESOS Y REUNIONES NACIONALES / NATIONAL CONGRESS AND MEETINGS

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LIII Congreso de la Sociedad Española de Cerámica y Vidrio y XII Congreso Nacional de la Asociación de Técnicos Cerámicos (ATC)
23 - 25 octubre [L'Alcora, Castellón de la Playa, España]

Materiales cerámicos a partir de arcillas y residuos de las industrias eléctricas: procesado, propiedades y caracterización microestructural
Pérez-Villarejo, L.; Carrasco-Hurtado, B.; Eliche-Quesada, D.; Ruiz-Conde, A.; Sánchez-Soto, P.J.
Poster

Nuevas aportaciones a los estudios arqueométricos de materiales cerámicos vidriados (siglos XI-XII) procedentes de intervenciones arqueológicas en los Reales Alcázares de Sevilla
Sánchez-Soto, P.J.; Moreno-Megías, V.; Pérez-Villarejo, L.; Ruiz-Conde, A.
Poster

Caracterización física, química y microstructural de nuevos materiales compuestos a partir de filitas
Garzón, E.; Cano, M.; Morales, L.; Pérez-Villarejo, L.; Ruiz-Conde, A.; Sánchez-Soto, P.J.
Poster

Caracterización tridimensional de objetos patrimoniales realizados en vidrio: colección de vidrios procedentes de la Real Fábrica de la Granja
Durán Domínguez, G.; Ruiz-Conde, A.; Sánchez-Soto, P.J.
Poster

DOCENCIA / TEACHING

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Curso de Nanotecnología
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- 1 molino planetario PM100 (Retsch)
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- 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

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

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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

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Dr. Mauricio E. Calvo Roggiani
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Becarios Predoctorales

Lda. María Alcaire Martín
Lda. Sara Borrego González
Ldo. Gabriel Castillo Dali
Ldo. Pedro Castillero Durán
Ldo. José Raúl Castro Smirnov
Ldo. Alberto J. Fernández Carrión
Ldo. Alejandro N. Filippin Emilio
Lda. Lola González García
Ldo. Alberto Jiménez Solano
Ldo. Manuel Macías Montero
Ldo. Manuel Oliva Ramírez
Ldo. Noe Orozco Corrales
Ldo. Julián Parra Barranco
Lda. Sonia Rodríguez Liviano
Lda. Liliam Beatriz Romero Sánchez
Lda. Antonia Terriza Fernández

Personal Contratado

Ldo. Miguel Anaya Martín
Lda. Silvia Colodrero Pérez
Dr. Juan F. Galisteo López
Dra. Ana García Navarro
Ldo. Jorge Gil Rostra
Lda. M. Carmen López López
Dra. Elisa Martínez de Castro
Ldo. Antonio Méndez Montoro de Damas

PROYECTOS DE INVESTIGACIÓN / RESEARCH PROJECTS

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. 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.

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 / 31-08-2015
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.

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Nuevos materiales para envasado, etiquetado inteligente, control de fraudes y monitorización visual del estado de los productos New materials for advanced packaging, intelligent labeling, anti-counterfeiting and monitoring of agricultural and livestock products

Código/Code:	RECUPERA2020 - 1.4.2
Periodo/Period:	02-12-2013 / 31-12-2015
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	454.239,23 €
Investigador responsable/Research head:	Angel Barranco Quero
Componentes/Research group:	Ana Isabel Borrás, Francisco Yubero, José Cotrino, Juan Pedro Espinós, Juan Ramón Sánchez Valencia, Francisco Javier Aparicio Rebollo

En esta actividad se pretende desarrollar una serie de nuevos materiales y procesos basados en marcado por láser para el desarrollo de un nuevo sistema de marcaje y etiquetado "inteligente" capaz de lograr una mejora en los procesos de control y de la trazabilidad de productos agropecuarios.

This Project intends the development of novel materials and processes for intelligent labeling of agricultural and livestock products to improve their traceability. The project is based on the development of active optical structures, laser processing strategies and the fabrication of practical testing prototypes.

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Purificación del aire en invernaderos y centros de tratamiento de alimentos Purification of air in greenhouses and food processing centers

Código/Code:	RECUPERA2020 - 2.2.3
Periodo/Period:	02-12-2013 / 31-12-2015
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	454.239,23 €
Investigador responsable/Research head:	José Cotrino Bautista
Componentes/Research group:	Ana María Gómez Ramírez, Antonio Méndez Montoro de Damas

RESUMEN / ABSTRACT

Este proyecto está relacionada con una tecnología para generar un plasma frío a presión atmosférica con aire que fluye a través de un reactor. El objetivo específico de esta actividad es el desarrollo de un sistema prototipo de purificación de aire para invernaderos, centros de procesamiento de alimentos, recintos para el ganado u otros tipos similares en mercados o recintos donde la concentración de gases nocivos para la salud de los trabajadores puede ser muy significativa por el uso de insecticidas, fungicidas, desinfectantes y otros compuestos. El sistema desarrollado debe ser capaz de purificar el aire en instalaciones cerradas y donde un gran número de productos químicos, compuestos orgánicos volátiles, principalmente, se acumulan en el aire del establecimiento. El diseño de la tecnología del reactor de plasma frío sigue las características de descarga de barrera dieléctrica con dieléctrico en forma de lecho empaquetado utilizando materiales dieléctricos de carácter ferroeléctrico con elevada constante dieléctrica.

This project is related with a technology to generate a cold plasma at atmospheric pressure with air flowing through a reactor. The specific objective of this activity is the development of a prototype air purification system for greenhouses, food processing centers, livestock enclosures, or other similar types of markets or enclosures where the concentration of gases harmful to the health of the workers can be very significant by the use of insecticides, fungicides, disinfectants or other compounds. The developed system should be able to purify the air in closed installations and where a large number of chemicals, mainly volatile organic compounds, accumulate in the air that is handled. The cold plasma reactor technology design

follows the characteristics of packed-bed dielectric barrier discharge by using ferroelectric dielectric.

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Sensores micro-fluídicos integrados para el control de la fermentación Microfluidic integrated sensors for the control of fermentation

Código/Code:	RECUPERA2020 – 1.4.1
Periodo/Period:	02-12-2013 / 31-12-2015
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	454.539,23 €
Investigador responsable/Research head:	Agustín R. González-Elipe
Componentes/Research group:	Juan Pedro Espinós Manzorro, José Cotrnio Bautista, Francisco Yubero Valencia, Alberto Palmero Acebedo, Angel Barranco Quero, Ana I. Borrás Martos, Victor J. Rico Gavira, Rafael Alvarez Molina, Pedro Angel Salazar Carballo

RESUMEN / ABSTRACT

El objetivo de este proyecto es el desarrollo de nuevos sistemas micro/nano fluídicos integrados y robustos que permitan la incorporación fiable de tests de control, sensorización y/o análisis rápido de productos agroalimentarios, fundamentalmente líquido o productos solubles. La tecnología a desarrollar se intenta aplicar tanto para el control de productos finales como durante las etapas de elaboración de los mismos. En concreto, un nicho de aplicación que directamente que se abordará dentro del proyecto es el control de procesos de fermentación, con el desarrollo de nuevos transductores fluídicos integrados que permitan la detección cuantitativa de glucosa y/o otros azúcares mediante desarrollos electroquímicos y fotónicos en dispositivos microfluídicos y similares.

The objective of this Project is the development of new integrated and robust micro/nano- fluidic systems that enable the reliable incorporation of control tests, sensorization and rapid analysis of agrofood products, mainly liquids or soluble. The technology to be developed should be applied to final products, as well as during their different elaboration steps. IN particular, a niche of application that will be directly addressed in the project is the control of fermentation process with the development of new integrated fluidic transductors that permit the quantitative detection of glucose and/or other sugars by means of electrochemical and photonic developments integrated in microfluidic and similar devices.

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Control de la Absorcion y la Emisión Ópticas de Nanomateriales integrados en Estructuras Fotónicas Porosas Multifuncionales

Control of the Optical Emission and Absorption properties of Nanomaterials Integrated in Multifunctional Porous Photonic Structures

Código/Code:	MAT2011-23593
Periodo/Period:	01-01-2012 / 31-12-2014
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	210.000 €
Investigador responsable/Research head:	Hernán R. Míguez García
Componentes/Research group:	Nuria Nuñez Alvarez, Mauricio Calvo Roggiani, Carmen López López, Sonia Rodríguez Liviano, Manuel Ocaña Jurado, Silvia Colodrero Pérez, José Raúl Castro Smirnov

RESUMEN / ABSTRACT

En este proyecto se estudiarán las modificaciones que tienen lugar tanto en la absorción como en la emisión ópticas de nanomateriales de diverso tipo (nanopartículas dopadas con tierras raras, nanopartículas semiconductoras, nanopartículas metálicas, films de colorantes orgánicos de grosor nanométrico) por el hecho de encontrarse éstos formando parte de una estructura fotónica en la que tienen lugar fenómenos ópticos complejos. El estudio se realizará tanto desde el punto de vista fundamental como aplicado, centrándose en materiales que tengan interés en distintos campos de la tecnología actual tales como células solares, sensores o dispositivos emisores de luz. La motivación principal de este proyecto desde el punto de vista aplicado radica en la posibilidad de modificar controladamente estos procesos de absorción y emisión, de tal modo que puedan inhibirse o amplificarse según convenga a un fin determinado. En concreto, se pretende poner en práctica estos nuevos fenómenos para el diseño de células solares más eficientes, capaces de recolectar una mayor cantidad de la radiación incidente, y en el desarrollo de films para sensores sensibles a modificaciones de distinto tipo en su entorno, tales como presencia de analitos de distinto tipo, variaciones en la presión del vapor ambiente, etc.... Del mismo modo, y basándonos en los exitosos resultados del proyecto MAT que ahora termina, pretendemos preparar materiales en los que tengan lugar mecanismos más eficientes de extracción de luminiscencia y que puedan emplearse en dispositivos emisores de luz. Nuestro proyecto incluye en esta ocasión como objetivo el desarrollo de pequeños dispositivos prototipo en los que se pongan en práctica los conceptos que están siendo investigados.

En su aspecto más fundamental, nuestra propuesta ahonda en el análisis de la interacción entre luz y materia en sistemas en los que existe una fuerte dispersión y anisotropía de la constante dieléctrica, y en los que es posible obtener bajas velocidades de propagación de fotones. Para este análisis se emplearán distintos tipos de estructuras fotónicas porosas, tales como cristales fotónicos con orden uni- y tri-dimensional o materiales formados por partículas

desordenadas, como matrices en los que se infiltrarán nanomateriales tanto orgánicos como inorgánicos en diferentes configuraciones y cuya absorción y emisión serán estudiadas.

Aunque este proyecto tiene un carácter fundamental tanto por la naturaleza de las técnicas de preparación como de los complejos fenómenos ópticos que pretenden explorarse, es nuestro objetivo estratégico continuar generando y transfiriendo propiedad intelectual basada en los nuevos conceptos, propiedades y diseños objeto de nuestra investigación.

In this project the modifications of both optical emission and absorption of nanomaterials of different sort (rare earth doped nanoparticles, semiconductor quantum dots, metallic nanoparticles, and films of organic dyes of nanometer dimensions) that occur when they are embedded in different types of photonic structures will be investigated. Both fundamental and applied aspects of the subject will be analysed. Efforts will be mainly focused on materials of current technological interest for solar cells, sensors and light emitting devices. From the applied point of view, this project finds its motivation in the possibility that photonic structures offer of modifying absorption and emission processes in a controlled manner so that they can be inhibited or amplified depending on the specific goal pursued. Particularly, we seek to put into practice these concepts to generate new designs of more efficient solar cells, capable of harvesting a larger amount of the incident radiation, and in the development of films for sensing devices responsive to modifications of different kind, such as presence of targeted molecules, variations of ambient gas pressure, etc... Also, more efficient or controlled light extraction from light emitting devices is sought after. The development of small prototype devices to prove the novel concepts under research is also an objective of this grant proposal.

In its more fundamental aspect, our project aims at deepening our knowledge of the interaction between light and matter in systems in which there exists a strong dispersion and anisotropy of the dielectric constant, and in which it is possible to attain very low photon propagation speeds. For this analysis, we will employ different types of porous photonic structures, such as one-dimensional and three-dimensional photonic crystals, as well as disordered assemblies of particles, as hosts in which a wide range of organic and inorganic nanomaterials will be integrated in different configurations and whose absorption and emission will be experimentally and theoretically studied.

Although this project has a fundamental character due to the nature of the preparation techniques and complex optical properties we seek to analyze, it is our aim to continue generating and transferring intellectual property based on the novel concepts, properties and designs which are the subject of our research.

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Láminas finas porosas funcionales y nanoestructuras 1D soportadas de óxidos para el desarrollo de microfluídica en lámina delgada, válvulas fotónicas y micro plasmas (POROUSFILMS) Functional porous thin films and 1D supported oxide nanostructures for the development of thin film microfluidics, photonic, valves, and microplasmas (POROUSFILMS)

Código/Code:	MAT2010-18447
Periodo/Period:	01-01-2011 / 31-12-2013
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	278.300 €
Investigador responsable/Research head:	Francisco Yubero Valencia
Componentes/Research group:	Agustín R. González-Elipe, Juan Pedro Espinós Manzorro, Alberto Palmero Acebedo, Rafael Alvarez Molina, Juan Carlos González González, Victor J. Rico Gavira, Jorge Gil Rostra, Ana Isabel Borrás Martos, Lola González García, José Cotrino Bautista, Manuel Oliva Ramírez, Antonia Terriza, Manuel Macías Montero, Francisco J. García García

RESUMEN / ABSTRACT

En este proyecto se prepararán láminas delgadas de TiO_2, ZnO, SiO_2 y SnO_2 dopado, así como nanostructruras de fibras soportadas de estos óxidos mediante deposition por plasma y por evaporación a ángulo rasante (GLAD). Se controlará la nano- y micro-estructura de las capas y fibras mediante la selección de las condiciones adecuadas de deposición usando GLAD y, en el caso de la deposición mediante plasma, tras ajustar y modelizar el plasma y sus principales parámetros de operación, así como los fenómenos ligados a la vaina del plasma y su influencia en el desarrollo de la nanoestructura de las capas y fibras. El primer objetivo del proyecto es conseguir controlar la porosidad y otras propiedades críticas (ópticas, conductividad eléctrica, comportameinto frente al mojado, etc.) de los materiales sintetizados para propiciar el desarrollo de nuevos métodos de manejo de fluidos (líquidos y gases) a escalas micro y, posiblemente, nanométricas de manera de inventar y desarrollar aplicaciones en los campos de microfuídica y microplasmas.

Un objetivo adicional es el procesado de estas estructuras tanto en una aproximación 2D (procesado litográfico) como 3D mediante el uso de técnicas basadas en láser, el apilamiento en multicpas de diferentes estrucuras de capas finas porosas y/o la deposición selectiva de zonas hidrofóbicas de otros materiales tales como polímeros, siliconas, etc. A partir de estas estructuras se desarrollarán dispositivos microfluídicos basados en láminas delgadas porosas de TiO2 y ZnO activadas con luz (válvulas fotónicas). Estos materiales se vuelven superhidrofílicos cuando se iluminan con luz de $\lambda<390$ nm que, usada para iluminar ciertas áreas pequeñas del material (canales, circuitos micrométricos, etc.) usando lámparas adecuadas o un láser, permitirá activar selectivamente esas zonas. El desarrollo de microfiltración controlada por luz es otra nueva aplicación en este campo que debe permitir la difusión/filtración preferente de líquidos polares a través de las zonas iluminadas (válvula abierta). Conseguir una rápida reversión de este proceso (válvula cerrada) is otro reto que se abordará dentro del proyecto.

Un objetivo final de carácter exploratorio es el modelado, diseño y desarrollo de microplasmas basados en las estrucutras porosas en forma de capa delgada desarrolladas durante las etapas iniciales del trabajo. Estos prototipos de microplasmas estarán formados por electrodos de capas porosas de SnO_2 dopadas permeables a gases y capas aislantes porosas de SiO2 que actuarán como barreras de separación. La evaluación de las caracterísitcas del plasma en estos dispositivos prototipo será una acción addicional que se abordará en el proyecto.

Functional TiO_2, ZnO, SiO_2 and doped SnO_2 in the form of porous thin films and other supported fiber-like nanostructures will be prepared by plasma deposition and evaporation at glancing angles (GLAD). Precise control of the nano and microstructure of the films and fibers will be attained by selecting appropriate GLAD deposition conditions and, in the case of plasma deposition, by adjusting the principal plasma parameters after modelling the plasma processes and sheath-related phenomena that control the development of the film/fibers nanostructure. The primary objective of the project is to successfully tailor the porosity and other key properties (optical, electrical conductivity, wetting behaviour etc.) of the synthetized materials to enable novel methods of fluid handling (liquids and gases) at the micro and, possibly, nanoscales so as to invent and develop applications in the fields of microfluidic and microplasmas.

A further objective is the processing of these structures in both 2D (i.e., lithographic processsing) and 3D by use of laser-based techniques, multilayer stacking of different porous thin film structures and/or selected plasma deposition of hydrophobic patches of other materials such as polymers, silicones, etc. Microfluidic thin film-based devices controlled by light (i.e., photonic valves) will then be developed by employing appropriately designed TiO_2 and ZnO porous structures. These materials become superhydrophilic when illuminated with light of $\lambda<390$ nm which will be used to selectively illuminate very small areas (channels, micrometer circuits, etc.) by either a suitable lamp or a laser. Light-controlled microfiltration is envisaged as another new application in this field, whereby preferential diffusion/filtration of polar liquids through the illuminated zones may be induced (i.e. valve open). Achieving prompt reversal of this process (i.e. valve closed) is another challenge that will be addressed by the project.

A final, exploratory objective is the modelling, design and development of microplasmas based on the most promising thin film porous structures developed during the earlier phases of the work. These prototype microplasma devices will consist of porous doped SnO_2 thin film electrodes permeable to gases with porous insulator layers (SiO_2) acting as separation barriers. Evaluation of the plasma characteristics of these prototype devices will be another distinct task undertaken by the project.

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Funcionalización superficial de materiales para aplicaciones de alto valor añadido (FUNCOAT) Surface functionalisation of materials for high added value applications (FUNCOAT)

Código/Code:	CSD2008-00023 (Consolider)
Periodo/Period:	15-12-2008 / 15-12- 2013
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	420.000 €
Investigador responsable/Research head:	González-Elipe, A.R.
Componentes/Research group:	Fernández Camacho, A., Espinós, J.P., Yubero, F., Cotrino, J., Sánchez López, J.C., Barranco, A., Palmero, A., Rojas, C., A. Borrás

RESUMEN / ABSTRACT

FUNCOAT es un proyecto integrado dentro de la convocatoria CONSOLIDER-INGENIO 2010 que persigue explotar las sinergias existentes entre la comunidad científica española en materiales, teniendo como objetivo general el desarrollo de nuevos métodos y la optimización de procedimientos de funcionalización superficial de materiales para la mejora de sus propiedades. El proyecto integra a 14 centros de investigación diferentes, que cubren desde aspectos básicos y teóricos hasta diversas aplicaciones finales. Esta integración de esfuerzos es crítica para lograr avances sustanciales en este campo, más allá que la mera acumulación de resultados. Los grupos de investigación pertenecen a diversas Universidades, CSIC (organismo que actúa como gestor del proyecto) y centros tecnológicos, y presentan relaciones científicas que, en algunos casos, se remontan hasta hace 15 años. Objetivos científico-técnicos parciales son: la comprensión de los fenómenos fundamentales que rigen la modificación de superficies y crecimiento de capas, el control micro y nanoestructural de superficies y capas delgadas, la optimización de los procesos de crecimiento de las capas, la obtención de superficies modificadas con características multifuncionales con aplicaciones en diversos campos (protección mecánica, óptica, magnetismo, biomateriales, energía, etc) y, finalmente, el desarrollo de nuevos dispositivos y aplicaciones de los materiales funcionales en sectores tecnológicos clave. Otros objetivos horizontales, relacionados con los anteriores, son la formación de investigadores en técnicas y métodos de funcionalización y el desarrollo de una transferencia tecnológica eficaz a los sectores productivos. Sectores estratégicos clave en los que las actividades de FUNCOAT inciden con mayor impacto son la metalurgia, vidrio, plásticos, sensores, salud y energía. El proyecto se estructura en torno a los ejes de actividad siguientes que deben permitir la mejor coordinación de los esfuerzos y la integración de las distintas actividades de los grupos. A) Fenómenos básicos de superficies, intercaras y láminas delgadas, B) Nuevos procesos para el control de la micro- y nano- estructura superficiales, C) Recubrimientos mecánicos y metalúrgicos para protección superficial, D) Funcionalización química de superficies y aplicaciones biomédicas, E) Recubrimientos para aplicaciones ópticas y control eficiente de captación energía solar, F) Nuevos fenómenos magnéticos en superficies e interfases.

FUNCOAT is an integrated project within the application call CONSOLIDER-INGENIO 2010 aiming at the exploitation of synergies existing in the Spanish scientific community, with the general objective of developing principles, processes and devices related to the surface functionalisation of materials. The project integrates 14 well-accredited research centres covering from fundamental and theoretical aspects to final applications. This large effort of integration is critical to achieve substantial advances in this broad field, which go beyond the mere accumulation of results. The research teams belong to different institutions: University, CSIC (responsible for the management of the project) and Technological centres. They maintain scientific relationships among them that extend over the last 15 years. Specific scientific and technological objectives are: understanding of fundamental phenomena driving the modification of surfaces and interfaces, control of the micro- and nano- structure of surfaces and thin films, optimization of thin film deposition methods, process development of multifunctional surfaces for novel applications (mechanical and metallurgical, optical, magnetic, energy, biomaterials, etc) and, finally, the production of new devices based on functionalised surfaces. Other important objectives include the technological transfer of the scientific results to the productive sectors as well as the education and training of scientists, young researchers and engineers. Strategic sectors of our modern society where the activities of FUNCOAT find a di-

rect impact are material processing, energy, environment, health care, agriculture, etc. In order to accomplish an efficient coordination of efforts and the integration of the activities of all the groups, the project is structured around six workpackages: A) Fundamental phenomena in surfaces, interfaces and thin films, B) New processes for the control of the micro- and nanostructure of films and surfaces, C) Mechanical and metallurgical coatings for surface protection, D) Chemical functionalisation and biomedical applications, E) Coatings for optical control, photonic applications and solar energy collection and F) Novel magnetic phenomena in surfaces/interfaces.

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Síntesis mediante plasma CVD de nuevos materiales orgánicos nanoestructurados integrados en dispositivos planares para aplicaciones como sensores fotónicos y de marcaje de seguridad NANOPLASMA

Plasma CVD synthesis of novel organic nanostructured materials integrated in planar devices for photonic sensing and security applications NANOPLASMA

Código/Code:	MAT2010-21228 (Plan Nacional)
Periodo/Period:	01-01-2011 / 31-12-2013
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad. Programa FEDER Unión Europea.
Importe total/Total amount:	169.4000 €
Investigador responsable/Research head:	Angel Barranco Quero
Componentes/Research group:	Ana Borrás Martos, Agustín R. González-Elipe, Juan Pedro Espinós, José Cotrino, Carmen Ruiz, M. Carmen López-Santos, Ricardo Molina

RESUMEN / ABSTRACT

En NANOPLASMA se propone el desarrollo de nuevas técnicas basadas en plasmas para la síntesis y procesado de nuevos materiales funcionales orgánicos. La tecnología de plasma para síntesis de materiales actualmente en uso, como el CVD activado por plasma (PECVD) o los procesos de polimerización por plasma, implica siempre la fragmentación completa de un precursor volátil. En contraste NANOPLASMA persigue la síntesis de una nueva familia de materiales luminiscentes en forma de película delgada o nanocables 1D soportados, mediante el control químico y del proceso de fragmentación en la zona remota de una descarga de plasma. La investigación se centrará en la síntesis de matrices orgánicas con microestructura nanométrica controlada que incorporan moléculas orgánicas luminiscentes (por ejemplo perilenos, rodaminas, ftalocianinas y porfirinas) y nanocables orgánicos 1D a partir del mismo tipo de moléculas.

El proyecto también contempla el desarrollo de metodologías basadas en procesos de etching por plasma y ablación láser, para la fabricación de patrones litográficos 2D a partir de las láminas delgadas y las nanoestructuras soportadas. La investigación se contempla con es-

tudios básicos encaminados al desarrollo de procesos de "patterning químico" basados en procesos de funcionalización superficial mediante plasmas y de derivatización química de monocapas autoensambladas. Tanto la metodología sintética mediante plasmas remotos como los procesos de microstructuración son totalmente compatibles con la tecnología optoelectrónica actual y la tecnología microelectrónica. Ambos procesos, por tanto, pueden llevarse a cabo a escala de oblea de silicio (wafer scale) y escalarse a procesos de fabricación industrial efectivos.

Estos materiales y procesos se emplearán en la fabricación de dos tipos de dispositivos a escala de prototipo como son: los sensores de gases fotónicos y las microestructuras luminiscentes para aplicaciones de marcado inteligente. Los sensores de gases consistirán en una lámina o estructura luminiscente integrada en un cristal fotónico 1D diseñados de acuerdo a las propiedades ópticas y la longitud de onda de la emisión luminiscente de la capa sensora correspondiente. Los dispositivos de marcado inteligente consistirán en patrones litográficos complejos construidos a partir de láminas o multicapas luminiscentes con funcionalidades específicas e, incluso, respuesta ambiental o sensora no desarrollada por ninguna otra tecnología hasta la fecha.

NANOPLASMA proposes the development of novel techniques based on plasma for the synthesis and processing of new organic functional materials. In contrast with the established plasma technology used in plasma enhanced CVD and plasma polymerization that implies the complete fragmentation of volatile precursor molecules, NANOPLASMA processes achieve the synthesis of new families of fluorescent thin films and supported 1D nanomaterials by controlling the chemistry and fragmentation degree at the boundaries of plasma discharge. The research focuses in the synthesis of organic matrices with a well controlled nanometric microstructure incorporating luminescent dye molecules (i.e. perylenes, rhodamines, phtalocyanines y porphirins) and 1D luminescent organic nanowires formed by similar molecules.

The project also contemplates the development of methodologies based on the plasma etching and laser ablation for the production of 2D lithographic patterns of the luminescent thin films and nanostructures. The research in this line will be completed with basic studies aiming the development of a "chemical patterning" process based on the plasma surface functionalization and chemical derivatization of self-assembled monolayers. Both the synthetic methodologies and the patterning strategies of NANOPLASMA are fully compatible with the present optoelectronic and silicon technologies and can be adapted to wafer scale integration for mass scale production.

These materials and processes will be used for the fabrication of two types of prototype devices: photonic gas sensors and luminescent microstructures for intelligent labelling applications. The gas sensing devices consist of a luminescence film and/or structure integrated onto a 1D photonic crystal with a stacking defect designed and constructed to couple the luminescent signal of the sensor layer. The intelligent labelling devices are patterned lithographic structures made on single or multilayer structures of luminescence films with specific functionalities and environmental responses not achieved by any available technology.

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Materiales mesoporosos (HA-SBA-15) funcionalizados con una proteína rhBMP-2 con afinidad por colágeno y sus estructuras híbridas con colágeno para ingeniería del tejido óseo

Mesoporous materials (HA-SBA-15) functionalized with a collagen-targeted rhBMP-2 and their related collagen hybrid composites for bone tissue engineering

Código/Code:	BIO2009-13903-C02-02 (Plan Nacional)
Periodo/Period:	01-01-2010 / 30-04- 2013
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	77.440 €
Investigador responsable/Research head:	M. Aránzazu Díaz Cuenca
Componentes/Research group:	M. Lourdes Ramiro Gutiérrez

RESUMEN / ABSTRACT

Los materiales soporte denominados biomateriales, matrices, armazones, andamiajes tisulares o scaffold son uno de los elementos básicos para la fabricación de nuevos tejidos artificiales. El biomaterial sirve de soporte para las interacciones celulares y para la formación de matriz extracelular (ECM). Entre los distintos tipos de materiales que se emplean naturales o sintéticos, las cerámicas bioactivas con base de sílice en el sistema ternario SiO2-CaO-P2O5 han mostrado excelente propiedades para su aplicación en cirugía ortopédica reparadora y regeneradora. Por otra parte el desarrollo de nuevas rutas de síntesis que combinan la química sol-gel y el autoensamblado de moléculas (polímeros) directoras o formadoras de estructura ofrece grandes posibilidades para la síntesis de de nuevos materiales bioactivos organizados a escala nanométrica con alta superficie y reactividades mejoradas. Trabajo previo de la IP de esta solicitud ha resultado en la síntesis de un nuevo biomaterial nanocomposite (HA-SBA-15) biocompatible constituido por nanopartículas de hidroxiapatito de calcio en asociación con la matriz de sílice mesoestructurada. Además de la biocompatibilidad, la alta superficie y la distribución de poros con un tamaño homogéneo hacen que este material sea un candidato muy interesante para su utilización como matriz para la adsorción de moléculas terapéuticas, medicamentos o factores de crecimiento que requieran su liberación de forma controlada. Las proteínas morfogenéticas de hueso (BMPs) han sido utilizadas ampliamente debido a sus propiedades osteoinductoras. Las proteínas recombinantes BMP-2 y BMP-7 han sido aprobadas por la FDA para su uso en cirugía ortopédica, sin embargo, el uso de estos factores de crecimiento no está muy extendido debido al alto costo de estos tratamientos y al miedo a posibles efectos secundarios como consecuencia de su utilización en altas concentraciones sin un sistema de liberación adecuado. Por otra parte resultados recientes del equipo coordinador de esta solicitud (subproyecto 2) ha producido y patentado una proteína recombinante BMP (rh-BMP-2) con un dominio de unión al colágeno tipo I (CBD). Esta proteína de fusión ha mostrado ventajas respecto a la proteína nativa BMP-2 y su uso combinado con colágeno puede representar

una alternativa terapéutica mejor y más segura en la reparación del tejido óseo. En este subproyecto se propone el estudio de nuevas rutas de síntesis para producir un material nano-organizado (HA-SBA-15) con variaciones en los parámetros texturales y las nanopartículas de HA con el objetivo de optimizar las propiedades de adsorción y posterior liberación de la proteína rhBMP-2 con afinidad por colágeno. Un objetivo paralelo será determinar las variables y condiciones experimentales adecuadas para incorporar la proteína rhBMP-2-CBD al material nano-organizado. El estudio analizará la cantidad de biomolécula, la cinética de desorción y su integridad. Una segunda tarea de esta propuesta es la consolidación del material particulado nano-organizado en piezas tridimensionales híbridas de material cerámico-colágeno biocompatibles, macroporosas y con una estabilidad mecánica mínima que permita su utilización en los experimentos in vivo que se proponen llevar a cabo como parte de las tareas del Subproyecto 2. Se ensayaran métodos para obtener piezas tridimensionales que conserven sus características nanoestructuradas. Se abordará la integración del material sin funcionalizar y funcionalizado con rhBMP-2-CBD.

A key component in tissue engineered materials for bone repair and regeneration is the scaffold that serves as a template for cell interactions and the formation on bone-extracellular matrix. This scaffold material also provides structural support to the newly formed tissue. Materials in the ternary system SiO2-CaO-P2O5 have demostrated excellent bioactivity for their use in orthopaedic repair and regeneration. The development of new synthesis routes which combine sol-gel chemistry and Block Copolymer (BCPs) self-assembly procedures have potential to be used as interesting methods to produce mesoporous organised SiO2-CaO-P2O5 materials with improved surface area and reactivity. Previous work carried out by the PI of this application has resulted in the synthesis of a biocompatible material (HA-SBA-15) consisting of calcium phosphate hydroxyapatite (HA) nanoparticles growth within a mesoporous (nano-sized-pore-organised) silica SBA-15 structure. Among their biocompatibility, the high surface area and the ordered distribution of pores with very homogeneous size confers to this material very interesting properties for their application as a matrix material for the adsorption of therapeutic agents, drugs or growth factors with requires their application in a controlled and prolonged release. The bone morphogenetic proteins (BMPs) have been widely used because their potent osteinductive properties and certain recombinant proteins BMP-2 and BMP-7 have been approved by the FDA for their use in orthopaedic surgery. Nevertheless, the use of these growth factors is not very extended due to the very high costs of these treatments and the fear to possible undesired side effects due to the use of high concentrations without any controlled delivery system. On the other hand, recent achievements of the team coordinator of this project application (Subproject 2) has produced and patented a recombinant BMP (rhBMP-2) with an additional decapeptidic collagen type I binding domain (CBD) which has shown that this fusion protein has advantages over native BMP-2, and that its combination with collagen may be better and safer alternative for bone repair. In this SubProject application we propose to work in new synthesis routes to produce a nanostructured composite material (HA-SBA-15) with variations in the textural and HA nanoparticle parameters to optimise improved collagen targeted BMP-2 protein adsorption capacities and delivery properties capacities and kinetics. A related objective will be to find and asses the experimental conditions and variables to incorporate successfully a collagen targeted BMP-2 protein to the nano-organised material. The study will cover the analysis of the biomolecule loading, desorption kinetics and final integrity. A second task of the proposed project will be the consolidation of the nano organised powder precursors in 3D ceramic-collagen hybrids composite scaffolds structures which fulfil requirements of biocompatibility, macroporosity and minimal mechanical stability for be using in the in the vivo experimental models which will be carried out as part of the working plan of the other SubProject (Subproyect 2). Work will be carried out to develop fabrication methods of the nanostructured materials into 3D scaffolds while retaining their nanostructural features. The integration of both the protein free HA-SBA-15 and also the fuctionalised collagen targeted BMP-2 material will be considered.

Sol y Visión par la energía térmica actual
Sun and vision for the present thermal energy. SOLVENTA

Código/Code:	IPT-2011-1425-920000
Periodo/Period:	04-05-2011 / 31-12-2014
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	246.992,80 €
Investigador responsable/Research head:	Agustín R. González-Elipe
Componentes/Research group:	Francisco Yubero Valencia, Juan Pedro Espinós Manzorro, Angel Barranco Quero, Victor J. Rico, Ana Borrás Martos, José Cotrino, Jorge Gil, Pedro Castillero, Fran J. García

RESUMEN / ABSTRACT

Este proyecto persigue el desarrollo de una serie de equipos y dispositivos para la monitorización de las condiciones de funcionamiento de plantas termosolares de concentración con espejos cilíndrico-parabólicos. La participación del ICMSE se centra en la aplicación tecnologías de plasma y el desarrollo de sistemas en lámina delgada capaces de diagnosticar las condiciones de funcionamiento de dichas instalaciones.

This Project aims at the development of a series of equipment and devices to monitor the working conditions of solar thermal plants based on light concentration with cylindrical parabolic mirrors. The role of ICMSE in this project focusses on the application of plasma technology systems and the development of thin films able to determine the working conditions of these facilities.

Desarrollo de membranas de regeneración ósea guiada modificadas a escala nanométrica (OSTEOMEM)
Development of bones regeneration membranes modified at nanometric scale (OSTEOMEM)

Código/Code:	P09-CTS-5189 (Proyecto de Excelencia)
Periodo/Period:	03-02-2010 / 02-02-2013

Organismo Financiador/Financial source: Junta de Andalucía
Importe total/Total amount: 221.673,68 €
Investigador responsable/Research head: R. González-Elipe, Agustín
Componentes/Research group: Cotrino, J., Alvarez Molina, R., López Santos, C., Gil Rostra, J., Terriza Fernández, A.

RESUMEN / ABSTRACT

OSTEOMEM persigue el desarrollo de membranas de regeneración ósea reabsorbibles y biodegradables de base polimérica para su uso en cirugía oral y maxilofacial para el tratamiento de defectos óseos. Durante la curación de estos, las membranas deben preservar la aparición de tejidos blandos en los mismos propiciando que la regeneración del hueso ocupe el defecto, dejando tras la reabsorción de la membrana una estructura de tejidos análoga a la que existía antes de la intervención quirúrgica. Este efecto se basa fundamentalmente en un mecanismo de barrera con membranas que se degradan en el interior del cuerpo en un periodo de cuatro a seis meses, evitando la segunda cirugía que generalmente se requiere para retirar membranas no biodegradables. Se espera que las membranas desarrolladas en el proyecto permitan alcanzar los resultados clínicos de las membranas de regeneración animal evitando los problemas asociados a su origen.

OSTEOMEM aims at developing disposable and biodegradable membranes for bone regeneration to be use in chirurgic oral and maxillofacial implants for the treatment of defects. During the healing of the bone defects, membranes must simultaneously preserve the formation of soft tissues and promote the filling of the hole by the new bone, so that, after the reabsorption of the membrane, the structure of tissues would be similar to that prior to the chirurgical intervention. To achieve that, the membranes should degrade within the body in a period of four-six months, thus avoiding the need of a second intervention required to remove non-biodegradable membranes. It is expected that the membranes developed in the project are comparable to that of animal membranes and avoid the problems associated with the use of these latter.

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 / 05-06-2015
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 SiO2-CaO-P2O5 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 SiO2-CaO-P2O5 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 SiO2-CaO-P2O5 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 SiO2-CaO-P2O5 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.

Control de procesos en plasmas para la síntesis de materiales nanoestructurados en forma de láminas delgadas (PLASMATER)
Process-control in plasmas for the synthesis of nanostructured thin films (PLASMATER)

Código/Code:	P09-FQM-6900 (Proyecto de Excelencia)
Periodo/Period:	15-03-2011 / 14-03-2014
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	167.209,71 €
Investigador responsable/Research head:	Alberto Palmero Acebedo
Componentes/Research group:	José Cotrino Bautista, Ana Borrás Martos, Francisco Yubero Valencia, Rafael Alvarez Molina, Juan Carlos González González, Carmen López Santos

RESUMEN / ABSTRACT

En el proyecto PLASMATER abordamos el desarrollo de nuevos procesos basados en plasmas para controlar la nanoestructura, porosidad y morfología superficial de películas delgadas, con el objetivo de mejorar sus funcionalidades para aplicaciones finales. En las técnicas de deposición de películas delgadas asistida por plasma, las cantidades que definen la deposición, tales como la potencia electromagnética empleada, presión de los gases, etc., definen las propiedades del plasma, que a su vez, y a través de procesos no-lineales y fuertemente acoplados entre sí, producen el crecimiento de la película delgada en un porta-substrato. La complejidad de todos estos procesos ha dado lugar a que existan multitud de relaciones de carácter empírico entre cantidades controlables experimentales y las nanoestructuras crecidas, sin que haya una explicación clara sobre los mecanismos que controlan dicho crecimiento, y la conexión entre ambas. Este conocimiento es esencial para proponer modificaciones en las condiciones de la deposición que permitiesen un mayor control y versatilidad a la hora de sintetizar películas delgadas nanoestructuradas.

En PLASMATER nos proponemos desarrollar herramientas para controlar procesos en plasmas y obtener capas nanoestructuradas y sistemas 1D de TiO2 y ZnO soportados en substratos, para mejorar propiedades funcionales tales como foto-actividad o propiedades de mojado, entre otras. Se explorarán tres aspectos relacionados entre sí: i) diagnosis completa de la fase gaseosa (plasma y vaina) y caracterización de la nanoestructura de los materiales depositados, ii) estudio de las funcionalidades de dichos materiales, y iii) el desarrollo de códigos numéricos predictivos, a partir de los cuales se desarrollen nuevos protocolos que permitan un mayor control sobre la nanoestructura del material y, por lo tanto, sobre las funcionalidades. El uso de modelos predictivos tiene una gran relevancia debido a que, hasta nuestro conocimiento, i) será la primera vez en la literatura que se dé una visión completa del proceso de deposición y nanoestructuración de estas películas delgadas basada en fenómenos fundamentales, a partir del valor de las cantidades experimentales de control (potencia electromagnética empleada, presión de los gases, etc.), y ii) se utilizará el modelo para proponer modificaciones en el proceso de deposición que ayuden a controlar los procesos de nanoestructuración de la película y proveer de más flexibilidad y versatilidad a los materiales depositados con vista a mejorar sus funcionalidades.

Para desarrollar el proyecto PLASMATER nos proponemos seguir una estrategia mixta simulación/diagnosis experimental del proceso de deposición para desarrollar interactivamente los modelos de crecimiento en múltiples condiciones. Se considerará el estudio de las diferentes escalas espaciales en el problema, desde el propio plasma (escala típica de decenas de centímetros), la vaina del plasma (por debajo de 1 milímetro), y la superficie del material (decenas de nanometros), y se utilizarán herramientas de diagnostico avanzado del plasma y de la película delgada que ayudarán al desarrollo del proyecto. Por último, la investigación también se centrará en encontrar las condiciones experimentales que permitan obtener la mejores propiedades de las capas con vista a optimizar sus aplicaciones tecnológicas e industriales.

Project PLASMATER aims at developing new plasma-based procedures to control the nanostructure, porosity and morphology of deposited thin films, and optimize the material functionalities and applications. From an experimental point of view, plasma-assisted thin film deposition techniques make use of various quantities to define the deposition conditions, such as the electromagnetic power, pressure in the reactor, etc. These quantities controls the plasma properties, which at the same time conditions the growth mechanism of the films. The complexity of the relation between experimentally controllable quantities and growth processes has produced the existence of empirical relations between experimental conditions and final film structure and composition, whose justification from a fundamental point of view is unclear.

In PLASMATER we propose to analyze three related aspects of the deposition of TiO2 and ZnO thin films assisted by plasmas: i) complete diagnosis of the plasma bulk and sheath in connection with the material microstructure, ii) functionality of the material, and iii) the development of predictive numerical codes that calculate the final film microstructure as a function of experimentally controllable quantities. This last part is of relevance because to our knowledge, i) it is the first time in the literature the deposition is fully characterized from a fundamental point of view, ii) this knowledge can be applied to suggest modifications in the deposition reactor in order to enhance different structural properties of the films.

In order to carry out the PLASMATER project, we aim at following at mixed theoretical and experimental strategy in order to interactively develop numerical codes of the thin film growth in multiple conditions. All the spatial scales involved in the description must be studied, from the plasma bulk itself (typically of few tens cm), the plasma sheath (below 1 mm), and the surface of the material (tens nm). Advanced diagnosis techniques will be employed to understand the plasma behavior and the film growth. Finally, PLASMATER will focus on the experimental conditions that lead to an optimized performance of the studied materials for advance applications in technology and industry.

Síntesis y propiedades de nanopartículas luminiscentes basadas en tierras raras para aplicaciones biomédicas Synthesis and properties of luminiscent nanoparticles based on rare earth for biological applications

Código/Code:	FQM6090 (Proyecto de Excelencia)
Periodo/Period:	15-03-2011 / 14-03-2014
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	100.000 €
Investigador responsable/Research head:	Manuel Ocaña Jurado
Componentes/Research group:	Angel Justo Erbez, Nuria O. Núñez Alvarez, Fernando Cussó, Jesús Martínez de la Fuente, Vanesa Sanz Beltrán, Sonia Rodríguez Liviano

RESUMEN / ABSTRACT

El trabajo de investigación que se propone en esta memoria está dirigido al desarrollo de nuevos métodos de síntesis, poco agresivos para el medioambiente, de materiales nanoestructurados luminiscentes constituidos por partículas uniformes con forma y tamaño de partícula controlados de fluoruros, vanadatos y fosfatos de Gd, Y o La dopados con diversos cationes lantánidos, así como al análisis de sus propiedades ópticas y magnéticas (en compuestos de Gd) y de sus posibles aplicaciones biomédicas. En concreto, se abordará el dopado con diversos cationes con luminiscencia convencional y emisión en el visible (Eu, Tb, etc.) o infrarrojo (Nd, Er, Pr) y el codopado con pares Er/Yb, Tm/Yb o Ho/Yb con luminiscencia "up-conversion". Las principales ventajas que presentan estos materiales basados en tierras raras frente a otros materiales fluorescentes propuestos para las aplicaciones mencionadas son su menor toxicidad, su mayor estabilidad química y sobre todo, en el caso de los materiales con fluorescencia "up-conversion", su excitación con radiación infrarroja evita que se dañen los tejidos biológicos. Las muestras preparadas se caracterizarán en términos de sus propiedades estructurales, morfológicas y composicionales y se evaluarán sus propiedades ópticas (absorción y luminiscencia) y magnéticas en relación con sus características morfológicas y estructurales. Posteriormente, se desarrollarán procedimientos de funcionalización de las nanopartículas obtenidas para obtener materiales estables en condiciones fisiológicas. Por último, se analizará la biocompatibilidad de las nanopartículas obtenidas y su eficacia para la detección de tumores.

This project is devoted to the development of new methods, environmentally friendly, for the synthesis of nanostructured luminescent materials consisting of uniform particles with controlled size and shape, based on a Gd, Y or La matrix (fluoride, vanadate or phosphate) doped with different lanthanide (Ln) cations, as well as to the analysis of theirs optical and magnetic (Gd compounds) properties and the evaluation of theirs biomedical applications. Several Ln cations with down-conversion and emissions in the visible (Eu, Tb) or near-infrared (NIR) (Nd, Er, Pr) and up-coversion (Er/Yb, Tm/Yb, Ho/Yb) luminescence have been selected. The main advantages that these Ln based nanomaterials present over other luminescent systems with potential bioapplications are their lower toxicity and higher chemical stability. In addition, in the case of up-converting nanophosphors, they are excited with NIR radiation which, in contrast with ultraviolet excitation, does not cause damage to biological tissues. Several procedures for the functionalization of the obtained nanoparticles will be essayed in order to obtain stable dispersions in physiological media. Finally, the nanophosphors biocompatibility and their efficiency for the detection of tumors will be also evaluated.

Nanoestructuras híbridas flexibles para aplicaciones con filtros de radiación ultravioleta, visible e infrarroja

Flexible hybrid nanostructures for applications as ultraviolet, visible and near infrared filters

Código/Code:	FQM6090 (Proyecto de Excelencia)
Periodo/Period:	03-02-2010 / 03-02-2013
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	193.431,68 €
Investigador responsable/Research head:	Hernán Míguez García
Componentes/Research group:	Mauricio Calvo Roggiani, Agustín Mihi Cervelló, Silvia Colodrero Pérez, Nuria Hidalgo Serrano, Gabriel Lozano Barbero, Olalla Sánchez Sobrado

RESUMEN / ABSTRACT

El objetivo principal de esta propuesta es el desarrollo de filtros interferenciales flexibles que reflejen rangos seleccionados de radiación ultravioleta (UV), visible (Vis) o infrarroja (IR). Se persigue, además, que sean transferibles y adhesivos, de forma que puedan adaptarse a distintos tipos de superficies. Se explorarán aplicaciones inmediatas de estos nuevos espejos dieléctricos como protectores adhesivos y transparentes de la piel contra radiaciones solares de alta energía (principalmente, el rangos conocido como UVA, de 315 nm a 400 nm), por lo que se pondrá especial énfasis en la integración de polímeros que sean compatibles con la piel humana. El interés y la originalidad de la aproximación aquí propuesta radica en la ausencia en el mercado de filtros de este tipo no absorbentes, es decir, no basados en procesos de absorción de luz que implican necesariamente un calentamiento, si no que funcionen como un espejo dieléctrico que refleje el rango de frecuencias deseado sin que causen un aumento local de la temperatura. En general, estos nuevos materiales permitirían proteger de la radiación no deseada cualquier tipo de superficie, de rugosidad y composición arbitraria. Del mismo modo, y en referencia a otro ámbito de aplicación, se buscará que los filtros propuestos proporcionen color a superficies arbitrarias, a las que se adaptarán y adherirán, reflejando un rango de frecuencias cuya posición central y anchura puede determinarse con precisión en todo el espectro visible. Por último, se explorarán aplicaciones en el IR como protectores contra calentamiento o, por el contrario, como recubrimientos transparentes en el rango visible pero que eviten las perdidas de calor, según la configuración empleada.

This project aims at developing radiation filters and screens in the shape of films and capable of blocking or selecting ultraviolet (UV), visible (Vis) or near infrared (NIR) radiation within well-defined spectral ranges. Biocompatibility, flexibility and specific adhesive properties will be sought after in order to make these films usable to protect all types of ill, wounded or burnt skin. The aim is to fill a currently existing hole in the field of skin phototherapy based on the healing properties of UV-Vis-NIR light, which is the absence of biocompatible patches in which selected ranges of the electromagnetic spectrum wavelengths can be sharply blocked or allowed to pass depending on the needs of the patient. For clinical cases that so required, an integral approach to skin photo-healing will be taken, devising materials that allow therapeutic wavelengths to reach the skin while blocking harmful ones and providing the controlled topical release of substances that have a beneficial effect on the skin. This project is based on a new series of novel prototype materials that have recently been developed in the group headed by the applicant in the Institute of Materials Science of Seville.

Materiales poliméricos y nanocomposites hibridos en forma de lámina delgada para aplicaciones fotónicas y fotovoltaicas (NANOPHOTON) Polymeric and hybrid nanocomposite thin films for photonic and photovoltaic applications

Código/Code:	P09-TEP-5283 (Proyecto de Excelencia)
Periodo/Period:	01-01-2010 / 02-02-2013
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	257.923,68 €
Investigador responsable/Research head:	Angel Barranco
Componentes/Research group:	Ana Borrás, Fabián Frutos, Lola González-García, Said Hamad, S. Lago, Alberto Palmero, Carmen Ruiz-Herrero, Juan R. Sánchez-Valencia, Johan Toudert

RESUMEN / ABSTRACT

El proyecto NANOPHOTON persigue el desarrollo de una nueva familia de materiales, estructuras y prototipos de dispositivos con aplicación en campos tales como el aprovechamiento de energía solar, el análisis medioambiental y la tecnología espacial. El punto de partida del proyecto es la investigación en nuevos materiales ópticamente activos en forma de película delgada nanométrica de carácter polimérico e híbrido. Estas láminas delgadas activas se procesarán e integrarán usando diferentes técnicas para formar estructuras ópticas de uso como sensores fotónicos ambientales, filtros fotónicos insensibles al ángulo de incidencia para

comunicaciones en satélites y celdas fotovoltaicas. Un aspecto muy interesante del proyecto es su completa compatibilidad con los métodos sintéticos de uso común en tecnología microelectrónica y optoelectrónica siendo fácilmente transferibles a la industria. NANOPHOTON integra desde estudios fundamentales de simulación molecular a procesos de fabricación de estructuras nanométricas con actividad fotónica pasando por la fabricación de estructuras y prototipos para llegar a test de validación de prototipos en condiciones reales.

The Nanophoton project aims the development of a novel family of materials, structures and device prototypes for application in solar energy, environmental sensing and space communication technology. The starting point of the project is the research in the photonic properties of hybrid nanometric films. These functional thin films will be processed and integrated in optical structures. The project encompasses fundamental molecular simulation studies, the development of novel nanometric functional structures, the study of suitable processing/integration procedures and the validation of prototype devices. These devices will be of three kinds: photonic gas sensors, detectors insensitive to the angle of detection for diffuse optical communications and photovoltaic cells. An interesting characteristic of the Nanophoton technology will be its fully compatibility with the current optoelectronic and microelectronic industrial manufacturing processes.

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.

PROYECTOS EN LOS QUE PARTICIPAN INTEGRANTES DE LA UNIDAD

Nuevas técnicas de procesado de cerámica y vidrio respuestas con el medio ambiente (CERAMGLASS)   Environmentally friendly processing of ceramics and glass (CERAMGLASS)

Código/Code:	LIFE11 ENV/ES/560
Periodo/Period:	01-01-2011 / 31-12-2014
Organismo Financiador/Financial source:	Unión Europea
Importe total/Total amount:	1.456.870 €
Investigador responsable/Research head:	Germán de la Fuente Leis
Componentes/Research group:	ICMS: Agustín R. González-Elipe, Victor J. Rico, Angel Barranco Quero, Juan Pedro Espinós Manzorro, Jorge Gil, Francisco Yubero Valencia

RESUMEN / ABSTRACT

El objetivo general del proyecto CERAMGLASS es disminuir el impacto al medio ambiente durante los tratamientos térmicos en la industria cerámica mediante la aplicación de una tecnología innovadora de horno láser a cerámica plana y vridrio. El proyecto persigue construir una planta piloto basada en la combinación innovadora de un horno continuo y un láser que barra la superficie. El propósito es probar que esta combinación permite una reducción significativa en el consumo energético y la escalabilidad industrial del proceso.

En primer lugar, el proyecto persigue demostrar que es posible producir placas cerámicas robustas de sólo 4 mm de espesor. Esto significaría una reducción del 50% en el espesor, con la consiguiente reducción en el consumo de materias primas. El proyecto persigue también adaptar las composiciones decorativas incorporando el uso de materiales menos agresivos al uso del procesado láser. En concreto, se tratará de adaptar los procesos de decorado “screen printing” a productos de tercer fuego con lustre y efectos metálicos, así como el de tintas decorativas al vidrio plano. La sustitución de productos iniciales tóxicos permitirá una disminución en la generación de CO2 y otras emisiones gaseosas, residuos tóxicos y la reducción de los requerimientos energéticos del proceso.

The general objective of the 'CERAMGLASS' project is to reduce the environment impact of thermal treatment of ceramics by the successful application of an innovative laser-furnace technology on planar ceramics and glass. The project plans to construct a pilot plant based on the innovative combination of a continuous furnace and a scanning laser. It aims at demonstrating a considerable reduction in energy consumption and the industrial scalability of the process.

The project primarily aims at showing that it is feasible to produce robust ceramic tile of only 4 mm thick. This would represent a 50% reduction in tile thickness, with consequent reduction in consumption of raw source materials. The project will adapt decoration compositions with more environmentally friendly materials by using the laser processing. Specifically it will adapt screen printing decorations to third-fire products with lustre and metallic effects and decoration inks for planar glass. The replacement of toxic starting materials will allow a minimisation of CO2 and other gas emissions, toxic residues and a reduction of the energy consumption of the process.

CONVENIOS Y CONTRATOS / CONTRACTS AND AGREEMENTS

Evaluación de la aplicabilidad de diversas estructuras inorgánicas como matriz huésped en el desarrollo de nuevos pigmentos inorgánicos

Periodo/Period:	01-01-2013 / 30-09-2014
Organismo Financiador/Financial source:	AL-FARBEN, S.A.
Investigador responsable/Research head:	Manuel Ocaña Jurado

Optimización de la recolección de luz en un módulo fotovoltáico formado por una lámina concentradora luminiscente integrada con células solares CIGS

Periodo/Period: 03-05-2012 / 02-05-2013
Organismo Financiador/Financial source: Abengoa Solar New Technologies, S.A.
Investigador responsable/Research head: Hernán Míguez García

Validación de la tecnología "Thin Film" fotovoltáica a escala piloto

Periodo/Period: 1-01-2010 / 31-12-2013
Organismo Financiador/Financial source: Abengoa Solar
Investigador responsable/Research head: Agustín R. González-Elipe

Fabricación de láminas de subóxidos de Silicio como sustratos para descomposición espinodal por procesado láser

Periodo/Period: 01-01-2011 / 31-12-2014
Organismo Financiador/Financial source: ABENGOA RESEARCH, S.L.
Investigador responsable/Research head: Ana Borrás Martos

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.
Investigador responsable/Research head: Ana Borrás Martos

Desarrollo de Added Value New CPVs enhanced developtments (ADVANCED)

Periodo/Period: 23-07-2013 / 31-12-2014
Organismo Financiador/Financial source: ABENGOA SOLAR NEW TECHNOLOGIES, S.A.
Investigador responsable/Research head: Agustín R. González-Elipe

Construcción y puesta en funcionamiento de un reactor de plasma DBD para la producción de hidrógeno

Periodo/Period: 26-04-2012 / 26-04-2013
Organismo Financiador/Financial source: ABENGOA HIDRÓGENO, S.A.
Investigador responsable/Research head: José Cotrino Bautista

Denominación del proyecto: Acuerdo de Colaboración para producir espejos de alta reflectividad a los fines de reducir costes en la tecnología Cilindro Parabólica

Periodo/Period: 4-11-2013 / 4-08-2014
Organismo Financiador/Financial source: ABENGOA Solar, S.A.
Investigador responsable/Research head: Hernán Míguez García

COOPERACIÓN INTERNACIONAL Y OTROS INTERNATIONAL COOPERATION AND OTHERS

Propiedades Químicas y Opticas del Nitruro de Cobre

Periodo/Period:	21-10-2013 / 04-11-2013
Código/Code:	E2013MX009
Entidad Financiadora/Financial source:	Estancias de investigadores de la UNAM en Centros del CSIC. Programa: PAISES CAD
Participantes/Participants:	Francisco Yubero Valencia; Wencel J. De la Cruz Hernández

PATENTES / PATENTS

Procedimiento de fabricación de un sensor de detección de hidrógeno y sensor así fabricado
Inventores: G. E. Rueda, N. M. Sanz, A. R. González-Elipe, A. B. Quero, F. Yubero, J. Cotrino y F. J. Garcia-Garcia
Solicitud número: P201300896
Fecha Solicitud: 30 Septiembre 2013
Entidad/es Titular/es: Abengoa Solar New Technologies

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

Angular response of photonic crystal based dye sensitized solar cells
López López, C.; Colodrero, S.; Calvo, M.E. and Míguez, H.
Energy & Environmental Science, **6** (2013) 1260-1266
Abril, 2013 | **DOI:** 1010.1039/C3EE23609A

Herein we report an experimental analysis of the performance of photonic crystal based dye solar cells (PC-DSCs) as the incident light angle moves away from the normal with respect to the cell surface. Nanoparticle multilayers operating at different wavelength ranges were coupled to the working electrode of a dye solar cell for this study. The interplay between optical and photovoltaic properties with the incident light angle is discussed. We demonstrate that an efficiency enhancement is attained for PC-DSCs at all angles measured, and that rational design of the photonic crystal back mirror leads to a reduction of the photocurrent losses related to the tilt angle of the cell, usually labeled as cosine losses. Angular variations of the cell transparency are also reported and discussed. These angular properties are relevant to the application of these solar devices in building integrated photovoltaics as potential window modules.

Tuning of Cell–Biomaterial Anchorage for Tissue Regeneration
Leal-Egana, Aldo; Diaz-Cuenca, Aranzazu; Boccaccini, Aldo R
Advanced Materials, **25** (2013) 4049-4057
Agosto, 2013 | **DOI:** 10.1002/adma.201301227

Which mechanisms mediate cell attachment to biomaterials? What role does the surface charge or wettability play on cell–material anchorage? What are the currently investigated strategies to modify cell–matrix adherence spatiotemporally? Considering the development of scaffolds made of biocompatible materials to temporarily replace the structure and/or function of the extracellular matrix, focus is given to the analysis of the specific (i.e., cell adhesive peptide sequences) and unspecific (i.e., surface charge, wettability) mechanisms mediating cell-matrix interactions. Furthermore, because natural tissue regeneration is characterized by the dynamic attachment/detachment of different cell populations, the design of advanced scaffolds for tissue engineering, based in the spatiotemporal tuning of cell–matrix anchorage is discussed.

Resonant Photocurrent Generation in Dye-Sensitized Periodically Nanostructured Photoconductors by Optical Field Confinement Effects
Anaya, M; Calvo, ME; Luque-Raigon, JM; Miguez, H
Journal of the American Chemical Society, **135** (2013) 7803-7806
Mayo, 2013 | **DOI:** 10.1021/ja401096k

Herein we show experimental evidence of resonant photocurrent generation in dye-sensitized periodically nanostructured photoconductors, which is achieved by spectral matching of the sensitizer absorption band to different types of localized photon modes present in either periodic or broken symmetry structures. Results are explained in terms of the calculated spatial distribution of the electric field intensity within the configurations under analysis.

Selective UV Reflecting Mirrors Based on Nanoparticle Multilayers
Smirnov, JRC; Calvo, ME; Miguez, H
Advanced Functional Materials, **23** (2013) 2805-2811
Junio, 2013 | **DOI:** 10.1002/adfm.201202587

A new type of nanostructured selective ultraviolet (UV) reflecting mirror is presented. Periodic porous multilayers with photonic crystal properties are built by spin-coating-assisted layer-by-layer deposition of colloidal suspensions of nanoparticles of ZrO2 and SiO2 (electronic band gap at $\lambda < 220$ nm). These optical filters are designed to block well-defined wavelength ranges of the UVA, UVB, and UVC regions of the electromagnetic spectrum while preserving transparency in the visible. The shielding against those spectral regions arises exclusively from optical interference phenomena and depends only on the number of stacked layers and the refractive index contrast between them. In addition, it is shown that the accessible pore network of the as-deposited multilayer allows preparing thin, flexible, self-standing, transferable, and adaptable selective UV filters by polymer infiltration, without significantly losing reflectance intensity, i.e., preserving the dielectric contrast. These films offer a degree of protection comparable to that of traditional ones, without any foreseeable unwanted secondary effects, such as photodegradation, increase of local temperature or, as is the case for organic absorbers, generation of free radicals, all of which are caused by light absorption.

Tuning Dichroic Plasmon Resonance Modes of Gold Nanoparticles in Optical Thin Films
Gonzalez-Garcia, L; Parra-Barranco, J; Sanchez-Valencia, JR; Ferrer, J; Garcia-Gutierrez, MC; Barranco, A; Gonzalez-Elipe, AR
Advanced Functional Materials, **23** (2013) 1655-1663 (Cover Front)
Abril, 2013 | **DOI:** 10.1002/adfm.201201900

A simple method is presented to tune the gold surface plasmon resonance (SPR) modes by growing anisotropic nanoparticles into transparent SiO2 thin films prepared by glancing angle deposition. In this type of composite film, the anisotropy of the gold nanoparticles, proved by gracing incidence small angle X-ray scattering, is determined by the tilted nanocolumnar structure of the SiO2 host and yields a strong film dichroism evidenced by a change from an intense colored to a nearly transparent aspect depending on light polarization and/or sample orientation. The formation in these films of lithographic non-dichroic SPR patterns by nanosecond laser writing demonstrates the potentialities of this procedure to develop novel optical encryption or anti-counterfeiting structures either at micrometer- or macroscales.

Vertically Aligned Hybrid Core/Shell Semiconductor Nanowires for Photonics Applications
Macias-Montero, M; Filippin, AN; Saghi, Z; Aparicio, FJ; Barranco, A; Espinos, JP; Frutos, F; Gonzalez-Elipe, AR; Borras, A
Advanced Functional Materials, **23** (2013) 5981-5989 (Cover Front)
Diciembre, 2013 | **DOI:** 10.1002/adfm.201301120

A family of 1D organic/inorganic core/shell materials formed by an inner organic nanowire (ONW) conformally covered with an inorganic wide band gap semiconductor (ZnO or TiO2) layer is presented. The developed procedure is a two-steps vacuum methodology involving the formation of supported single crystal small-molecule nanowires by physical vapor deposition and plasma enhance chemical vapor deposition (PECVD) of the inorganic shell. Critical characteristics of the last technique are the possibilities of low temperature and remote configuration deposition. Additionally, an initial step has to be included in order to create nucleation centers for the growth of the ONWs. The procedure and its general character in terms of the variability in organic core and inorganic shells composition and the applicability of the technique to different substrates are presented. The formation of the inorganic shell with no damage of the organic core single-crystalline structure is demonstrated by high resolution transmission electron microscopy. The vertical alignment of the hybrid nanostructure is achieved thanks to the interaction of the 1D organic nanostructured surfaces and the glow discharge during the deposition of the inorganic shell by PECVD. The optical properties of these core/shell NWs are studied by fluorescence spectroscopy and microscopy, and their application as nanoscale waveguides in the 550–750 nm range addressed.

Perfectly Transparent $Sr_3Al_2O_6$ Polycrystalline Ceramic Elaborated from Glass Crystallization

Alahrache, S; Al Saghir, K; Chenu, S; Veron, E; Meneses, DD; Becerro, AI; Ocana, M; Moretti, F; Patton, G; Dujardin, C; Cusso, F; Guin, JP; Nivard, M; Sangleboeuf, JC; Matzen, G; Allix, M
Chemistry of Materials, **25** (2013) 4017-4024
Octubre, 2013 | **DOI:** 10.1021/cm401953d

The highly visible and infrared (up to 6 mu m) transparent $Sr_3Al_2O_6$ polycrystalline ceramic was obtained by full crystallization of the corresponding glass composition. The glass synthesis and the direct congruent crystallization processes are described, and the material transparency is discussed in light of its microstructure. This new transparent ceramic exhibits a high density (i.e., complete absence of porosity) and micrometer-scale crystallites with very thin grain boundaries. These microstructural characteristics, inherent to the preparation method, minimize light scattering and demonstrate the advantages of this synthesis route compared to the high-pressure process used for the few reported transparent polycrystalline materials. This $Sr_3Al_2O_6$ ceramic shows a H = 10.5 GPa hardness, a E-r = 150 GPa reduced elasticity modulus, and a 9.6 x 10(-6) K-1 thermal expansion coefficient. Such a transparent strontium aluminate ceramic opens the way to a wide range of applications, especially photonics when doped by various doping agents. As examples, the luminescence of $Sr_3Al_2O_6{:}Eu^{3+}$ and $Sr_3Al_2O_6{:}Er^{3+}$, which show strong emissions in the visible and infrared ranges, respectively, is presented. Moreover, the $Sr_3Al_2O_6{:}Ce^{3+}$ material was found to exhibit scintillation properties under X-ray excitation. Interestingly, the analogous $Sr_3Ga_2O_6$ transparent polycrystalline ceramic material could equally be prepared using the same elaboration method, although its hygroscopicity prevents the preservation of its high transparency under normal conditions. The establishment of the key factors for the transparency of this economical and innovative synthesis method should enable the prediction of new classes of technologically relevant transparent ceramics.

Competing Misfit Relaxation Mechanisms in Epitaxial Correlated Oxides
Sandiumenge, F; Santiso, J; Balcells, L; Konstantinovic, Z; Roqueta, J; Pomar, A; Espinos, JP; Martinez, B
Physical Review Letters, **110** (2013) 107206
Marzo, 2013 | **DOI:** 10.1103/PhysRevLett.110.107206

Strain engineering of functional properties in epitaxial thin films of strongly correlated oxides exhibiting octahedral-framework structures is hindered by the lack of adequate misfit relaxation models. Here we present unreported experimental evidence of a four-stage hierarchical development of octahedral-framework perturbations resulting from a progressive imbalance between electronic, elastic, and octahedral tilting energies in La0.7Sr0.3MnO3 epitaxial thin films grown on SrTiO3 substrates. Electronic softening of the Mn-O bonds near the substrate leads to the formation of an interfacial layer clamped to the substrate with strongly degraded magnetotransport properties, i.e., the so-called dead layer, while rigid octahedral tilts become relevant at advanced growth stages without significant effects on charge transport and magnetic ordering.

Colored and Transparent Oxide Thin Films Prepared by Magnetron Sputtering: The Glass Blower Approach
Gil-Rostra, J; Chaboy, J; Yubero, F; Vilajoana, A; Gonzalez-Elipe, AR

ACS Applied Materials & Interfaces, **5** (2013) 1967-1976
Marzo, 2013 | **DOI:** 10.1021/am302778h

This work describes the reactive magnetron sputtering processing at room temperature of several mixed oxide MxSiyOz thin films (M: Fe, Ni, Co, Mo, W, Cu) intended for optical, coloring, and aesthetic applications. Specific colors can be selected by adjusting the plasma gas composition and the Si–M ratio in the magnetron target. The microstructure and chemistry of the films are characterized by a large variety of techniques including X-ray photoemission spectroscopy, X-ray absorption spectroscopy (XAS), and infrared spectroscopy, while their optical properties are characterized by UV–vis transmission and reflection analysis. Particularly, XAS analysis of the M cations in the amorphous thin films has provided valuable information about their chemical state and local structure. It is concluded that the M cations are randomly distributed within the SiO_2 matrix and that both the M concentration and its chemical state are the key parameters to control the final color of the films.

Liquids Analysis with Optofluidic Bragg Microcavities
Oliva-Ramirez, M; Gonzalez-Garcia, L; Parra-Barranco, J; Yubero, F; Barranco, A; Gonzalez-Elipe, AR
ACS Applied Materials & Interfaces, **5** (2013) 6743-650
Julio, 2013 | **DOI:** 10.1021/am401685r

Porous Bragg microcavities formed by stacking a series of porous nanocolumnar layers with alternate low (SiO_2) and high (TiO_2) refractive index materials have been prepared by physical vapor deposition at glancing angles (GLAD). By strictly controlling the porosity and refractive index of the individual films, as well as the relative orientation of the nanocolumns from one layer to the next, very porous and nondispersive high optical quality microcavities have been manufactured. These photonic structures have been implemented into responsive devices to characterize liquids, mixtures of liquids, or solutions flowing through them. The large displacements observed in the optical spectral features (Bragg reflector gap and resonant peak) of the photonic structures have been quantitatively correlated by optical modeling with the refractive index of the circulating liquids. Experiments carried out with different glucose and NaCl solutions and mixtures of water plus glycerol illustrate the potentialities of these materials to serve as optofluidic devices to determine the concentration of solutions or the proportion of two phases in a liquid mixture.

Crystal Structure and Luminescent Properties of Eu^{3+}-Doped A-$La_2Si_2O_7$ Tetragonal Phase Stabilized by Spray Pyrolysis Synthesis
Fernandez-Carrion, Alberto J.; Ocana, Manuel; Florian, Pierre; Garcia-Sevillano, Jorge; Cantelar, Eugenio; Fitch, Andrew N.; Suchomel, Matthew R.; Becerro, Ana I.
Journal of Physical Chemistry C, **117** (2013) 20876-20886
Octubre, 2013 | **DOI:** 10.1021/jp407172z

Pure A-$La_2Si_2O_7$ powder has been synthesized through a spray pyrolysis method followed by calcination at 1100 degrees C for 15 h. The crystallographic structure, refined from the synchrotron powder diffraction pattern of the sample, showed tetragonal symmetry with space group P4(1), a = 6.83565(1) angstrom, and c = 24.84133(1) angstrom. The Si-29 and La-139 NMR spectra have been described here for the first time in the literature and could be simulated with four Si and four La resonances, respectively, in good agreement with the presence of four Si and four La crystallographic sites in the unit cell. The same synthesis method was 2 successful for the synthesis of Eu3+-doped A-$La_2Si_2O_7$ (%Eu = 3-40). The analysis of the unit cell volumes indicated that Eu3+ replaces La3+ in the unit cell for all Eu3+ substitution levels investigated. However, anomalous diffraction data indicated that the La/Eu substitution mechanism was not homogeneous, but Eu much prefers to occupy the RE3 sites. The Eu-doped A-$La_2Si_2O_7$ phosphors thus synthesized exhibited a strong orange-red luminescence after excitation at 393 nm. Lifetime measurements indicated that the optimum phosphor was that with an Eu3+ content of 20%, which showed a lifetime of 2.3 ms. The quantum yield of the latter was found to be 12% at 393 nm excitation. These experimental observations together with the high purity of the phase obtained by the proposed spray pyrolysis method make this material an excellent phosphor for optoelectronic applications.

Crystal Structures and Photoluminescence across the $La_2Si_2O_7$–$Ho_2Si_2O_7$ System
Fernandez-Carrion, AJ; Allix, M; Ocana, M; Garcia-Sevillano, J; Cusso, F; Fitch, AN; Suard, E; Becerro, AI
Inorganic Chemistry, **52** (2013) 13469-13479
Noviembre, 2013 | **DOI:** 10.1021/ic401867c

The $La_2Si_2O_7$–$Ho_2Si_2O_7$ system displays a solid solubility region of G-$(La,Ho)_2Si_2O_7$ which extends to the La0.6Ho1.4Si_2O_7 composition. Compositions richer in Ho3+ show a two-phase domain (G+δ), while δ-$(La,Ho)_2Si_2O_7$ is the stable phase for Ho3+ contents higher than La0.2Ho1.8Si_2O_7. A preferential occupation of Ho for the RE2 site of the G-unit cell is observed. Luminescence measurements have shown that the lifetimes remain unchanged in the range 0.5% < [Ho3+] < 10%, and only above this value does concentration quenching become operative.

Synthesis and Properties of Multifunctional Tetragonal Eu:$GdPO_4$ Nanocubes for Optical and Magnetic Resonance Imaging Applications
Rodriguez-Liviano, S; Becerro, AI; Alcantara, D; Grazu, V; de la Fuente, JM; Ocana, M
Inorganic Chemistry, **52** (2013) 647-654
Enero, 2013 | **DOI:** 10.1021/ic3016996

A simple and fast (7 min) procedure for synthesis of gadolinium phosphate nanocubes (edge = 75 nm) based on the microwave-assisted heating at 120 °C of gadolinium acetylacetonate and phosphoric acid solutions in buthylene glycol is reported. These nanocubes were highly crystalline and crystallized into a tetragonal structure, which has not been ever reported for pure gadolinium phosphate. Determination of such crystal structure has been carried out here for the first time in the literature by means of powder X-ray diffraction. The developed synthesis procedure was also successful for preparation of multifunctional europium(III)-doped the gadolinium phosphate nanocubes, which were nontoxic for cells and exhibited strong red luminescence under UV illumination and high transverse relaxivity (r2) values. These properties confer them potential applications as biolabels for in vitro optical imaging and as negative contrast agent for magnetic resonance imaging.

A Novel 3D Architecture of $GdPO_4$ Nanophosphors: Multicolored and White Light Emission
Becerro, AI; Rodriguez-Liviano, S; Fernandez-Carrion, AJ; Ocaña, M
Crystal Growth & Design, **55** (2013) 454-460
Febrero, 2013 | **DOI:** 10.1021/cg301023k

Homogeneous monoclinic $GdPO_4$ particles composed of three intersecting lance-shaped crystals forming a penetration twin have been synthesized following a very restrictive, simple, and fast (10 min) method consisting of the hydrothermal reaction of gadolinium acetylacetonate with H_3PO_4 in a mixture of ethylene glycol and water at 180 °C. Slightly increasing the amount of water in the solvent mixture leads to hexagonal rodlike $GdPO_4{\cdot}0.5H_2O$ nanoparticles, whereas the variation of the Gd source, PO_4 source, aging temperature, and polyol type gave rise to heterogeneous particles. The synthesis procedure is also suitable for the preparation of Eu3+-, Tb3+-, and Dy3+-doped $GdPO_4$ particles with the same morphology and crystalline structure as the undoped materials. The effect of the doping level on the luminescent properties of the twinlike nanophosphors was evaluated, finding optimum doping levels of 5, 5, and 1% for the Eu3+-, Tb3+-, and Dy3+-doped materials, respectively. The twinlike $GdPO_4$ nanophosphors were found to be more efficient than the rodlike $GdPO_4$ ones in terms of emission intensity. Finally, a solid-state single-phase white-light-emitting nanophosphor has been fabricated for the first time in this system by triply doping the $GdPO_4$ twined particles with appropriate concentrations of Eu3+, Tb3+, and Dy3+ and exciting through the Gd–Ln energy-transfer band at 273 nm. In addition to this energy transfer band, other energy charge transfer processes among the three dopants (Eu3+, Tb3+, and Dy3+) have been observed in the triply doped material.

Ionic Liquid Mediated Synthesis and Surface Modification of Multifunctional Mesoporous Eu:GdF3 Nanoparticles for Biomedical Applications
Rodriguez-Liviano, S; Nunez, NO; Rivera-Fernandez, S; de la Fuente, JM; Ocana, M
Langmuir, **29** (2013) 3411-3418
Marzo, 2013 | **DOI:** 10.1021/la4001076

A procedure for the synthesis of multifunctional europium(III)-doped gadolinium(III) fluoride (Eu:GdF3) nanoparticles (85 nm) with quasispherical shape by precipitation at 120 °C from diethylene glycol solutions containing lanthanide chlorides and an ionic liquid (1-Butyl, 2-methylimidazolium tetrafluoroborate) as fluoride source has been developed. These nanoparticles were polycrystalline and crystallized into a hexagonal structure, which is unusual for GdF3. They were also mesoporous (pore size = 3.5 Å), having a rather high BET surface area (75 m2 g–1). The luminescent and magnetic (relaxivity) properties of the Eu:GdF3 nanoparticles have been also evaluated in order to assess their potentiality as “in vitro” optical biolabels and contrast agent for magnetic resonance imaging. Finally, a procedure for their functionalization with aspartic-dextran polymers is also reported. The functionalized Eu:GdF3 nanoparticles presented negligible toxicity for Vero cells, which make them suitable for biotecnological applications.

Microwave-Assisted Synthesis of Biocompatible Europium-Doped Calcium Hydroxyapatite and Fluoroapatite Luminescent Nanospindles Functionalized with Poly(acrylic acid)

Escudero, A; Calvo, ME; Rivera-Fernandez, S; de la Fuente, JM; Ocana, M
Langmuir, **29** (2013)
Febrero, 2013 | **DOI:** 10.1021/la304534f

Europium-doped calcium hydroxyapatite and fluoroapatite nanophosphors functionalized with poly(acrylic acid) (PAA) have been synthesized through a one-pot microwave-assisted hydrothermal method from aqueous basic solutions containing calcium nitrate, sodium phosphate monobasic, and PAA, as well as sodium fluoride in the case of the fluoroapatite particles. In both cases a spindlelike morphology was obtained, resulting from an aggregation process of smaller subunits which also gave rise to high specific surface area. The size of the nanospindles was 191 (32) × 40 (5) nm for calcium hydroxyapatite and 152 (24) × 38 (6) nm for calcium fluoroapatite. The luminescent nanoparticles showed the typical red luminescence of Eu^{3+}, which was more efficient for the fluoroapatite particles than for the hydroxyapatite. This is attributed to the presence of OH– quenchers in the latter. The nanophosphors showed negligible toxicity for Vero cells. Both PAA-functionalized nanophosphors showed a very high (up to at least 1 week) colloidal stability in 2-(N-morpholino)ethanesulfonic acid (MES) at pH 6.5, which is a commonly used buffer for physiological pH. All these features make both kinds of apatite-based nanoparticles promising tools for biomedical applications, such as luminescent biolabels and tracking devices in drug delivery systems.

Surface modified Eu:$GdVO_4$ nanocrystals for optical and MRI imaging
Nunez, Nuria O.; Rivera, Sara; Alcantara, David; de la Fuente, Jesus M.; Garcia-Sevillano, Jorge; Ocana, Manuel
Dalton Transactions, **42** (2013) 10725-10734
Agosto, 2013 | **DOI:** 10.1039/C3DT50676B

A facile solvothermal route has been developed for the preparation of europium doped gadolinium orthovanadate nanoparticles ([similar]70 nm) with tetragonal structure, based on a homogenous precipitation reaction at 120 °C from rare earth precursors (yttrium nitrate and europium nitrate) and sodium orthovanadate solutions using an ethylene glycol–water mixture as the solvent. The effects of the doping level on the luminescence properties were evaluated in order to find the optimum nanophosphors. These nanocrystals were successfully functionalized with amino (two step process) and carboxylate (one-pot process) groups provided by amino-dextran polymers (AMD) and polyacrylic acid (PAA), respectively. It was found that while the luminescent properties of both kinds of functionalized systems were similar, the colloidal stability of the PAA-modified sample was higher, because of which, it was selected to study their cytotoxicity and magnetic properties (relaxivity and phantom analyses) to assess their potentiality as multifunctional probes for both “in vitro” optical biolabels and negative contrast agents for magnetic resonance imaging.

Synthesis of metallic silver nanoparticles and silver organometallic nanodisks mediated by extracts of Capsicum annuum var. aviculare (piquin) fruits
Mendoza-Resendez, R; Nunez, NO; Barriga-Castro, ED; Luna, C
RSC Advances, **43** (2013) 20765-20771
Septiembre, 2013 | **DOI:** 10.1039/C3RA43524E

Silver-based nanostructures were prepared through reduction/oxidation reactions of aqueous silver nitrate solutions mediated by extracts of red fruits of the piquin pepper (Capsicum annuum var. aviculare) at room temperature. Detailed morphological and microstructural studies using X-ray diffraction, conventional and high-resolution transmission electron microscopy and selected area electron diffraction revealed that the product was constituted by three kinds of nanoparticles. One of them was composed of twinned metallic silver nanoparticles with a size of few nanometers. Other kind of particles was ultrafine disk-like single crystals of silver 4,4'-dimethyldiazoaminobenzene, being in our best knowledge the first time that this compound is reported in the form of nanoparticles. Both kinds of nanoparticles experienced processes of self-assembly and subsequent grain growth to form the third kind of nanoparticles. Such resulting nanostructures are monocrystalline and flattened metallic silver nanoparticles that have diameters around tens of nanometers, the [112] direction perpendicular to the particle plane, and are coated by a surface organometallic layer and residues of biomolecules. The ultraviolet-visible spectrum of the biosynthesized product showed a surface plasmon resonance (SPR) extinction band with an absorbance maximum at around 400 nm, thereby confirming the presence of fine Ag particles. Studies carried out by Fourier transform infrared spectroscopy indicated that the principal active compounds responsible of the reduction of the Ag ions are proteins and capsaicin (through the amino groups) and phenolic compounds (through hydroxyl groups).

Enhanced reactivity and related optical changes of Ag nanoparticles on amorphous Al_2O_3 supports

Pelaez, RJ; Castelo, A; Afonso, CN; Borras, A; Espinos, JP; Riedel, S; Leiderer, P; Boneberg, J
Nanotechnology, **24** (2013) 365702
Septiembre, 2013 | **DOI:** 10.1088/0957-4484/24/36/365702

Pairs of samples containing Ag nanoparticles (NPs) of different dimensions have been produced under the same conditions but on different substrates, namely standard glass slides and a thin layer of amorphous aluminum oxide (a-Al_2O_3) on-glass. Upon storage in ambient conditions (air and room temperature) the color of samples changed and a blue-shift and damping of the surface plasmon resonance was observed. The changes are weaker for the samples on-glass and tend to saturate after 12 months. In contrast, the changes for the samples on a-Al_2O_3 appear to be still progressing after 25 months. While x-ray photoelectron spectroscopy shows a slight sulfurization and negligible oxidation of the Ag for the on-glass samples upon 25 months aging, it shows that Ag is strongly oxidized for the on a-Al_2O_3 samples and sulfurization is negligible. Both optical and chemical results are consistent with the production of a shell at the expense of a reduction of the metal core dimensions, the latter being responsible for the blue-shift and related to the small (<10 nm initial diameter) of the NPs. The enhanced reactivity of the Ag NPs on the a-Al_2O_3 supports goes along with specific morphological changes of the Ag NPs and the observation of nitrogen.

Growth regimes of porous gold thin films deposited by magnetron sputtering at oblique incidence: from compact to columnar microstructures

Alvarez, R; Garcia-Martin, JM; Macias-Montero, M; Gonzalez-Garcia, L; Gonzalez, JC; Rico, V; Perlich, J; Cotrino, J; Gonzalez-Elipe, AR; Palmero, A
Nanotechnology, **24** (2013) 045604
Febrero, 2013 | **DOI:** 10.1088/0957-4484/24/4/045604

Growth regimes of gold thin films deposited by magnetron sputtering at oblique angles and low temperatures are studied from both theoretical and experimental points of view. Thin films were deposited in a broad range of experimental conditions by varying the substrate tilt angle and background pressure, and were analyzed by field emission scanning electron microscopy and grazing-incidence small-angle x-ray scattering techniques. Results indicate that the morphological features of the films strongly depend on the experimental conditions, but can be categorized within four generic microstructures, each of them defined by a different bulk geometrical pattern, pore percolation depth and connectivity. With the help of a growth model, a microstructure phase diagram has been constructed where the main features of the films are depicted as a function of experimentally controllable quantities, finding a good agreement with the experimental results in all the studied cases.

Laser induced enhancement of dichroism in supported silver nanoparticles deposited by evaporation at glancing angles
Filippin, AN; Borras, A; Rico, VJ; Frutos, F; Gonzalez-Elipe, AR
Nanotechnology, **24** (2013) 045301
Febrero, 2013 | **DOI:** 10.1088/0957-4484/24/4/045301

Silver nanoparticles (NPs) depicting well defined surface plasmon resonance (SPR) absorption were deposited on flat substrates by physical vapor deposition in a glancing angle configuration. The particles were characterized by scanning electron microscopy and atomic force microscopy and their optical properties examined by UV–vis absorption spectroscopy using linearly polarized light. It was found that, depending on the amount of deposited silver and the evaporation angle, part of the 'as-prepared' samples present NPs characterized by an anisotropic shape and a polarization dependent SPR absorption and different colors when using polarized white light at 0° and 90°. Low-power irradiation of these materials with an infrared Nd-YAG nanosecond laser in ambient conditions produced an enhancement in such dichroism. At higher powers, the dichroism was lost and the SPR bands shifted to lower wavelengths as a result of the reshaping of the silver NPs in the form of spheres. The possible factors contributing to the observed changes in dichroism are discussed.

Solvent-Controlled Synthesis and Luminescence Properties of Uniform Eu:YVO4 Nanophosphors with Different Morphologies
Nunez, N; Sabek, J; Garcia-Sevillano, J; Cantelar, E; Escudero, A; Ocana, M
European Journal of Inorganic Chemistry, **8** (2013) 1301-1309
Marzo, 2013 | **DOI:** 10.1002/ejic.201201016

A facile solvothermal route has been developed for the preparation of tetragonal europium-doped yttrium orthovanadate nanoparticles (Eu:YVO4) and is based on a homogeneous precipitation reaction at 120 °C from solutions of rare earth precursors (yttrium acetylacetonate and europium nitrate) and sodium orthovanadate in ethylene glycol or ethylene glycol/water mixtures. The nature of the solvent has a dramatic effect on the morphology and crystallinity of the resulting nanoparticles. Polycrystalline nanoellipsoids (130 × 60 nm) were obtained in pure ethylene glycol, whereas quasispherical nanoparticles (100 nm) with monocrystalline character precipitated in ethylene glycol/water (7:3 by volume) mixtures. To explain these different morphological and structural features, the mechanism of particles formation was investigated. The effects of the doping level on the luminescence properties (emission spectra and luminescence lifetime) were also evaluated to find the optimum nanophosphors. Finally, it is shown that the luminescent efficiency of the quasispherical nanoparticles was higher than that of the nanoellipsoids; this can be related to differences in crystallinity and in impurity content.

Small Particle-Size Talc Is Associated with Poor Outcome and Increased Inflammation in Thoracoscopic Pleurodesis
Arellano-Orden, E; Romero-Falcon, A; Juan, JM; Jurado, MO; Rodriguez-Panadero, F; Montes-Worboys, A
Respiration, **86** (2013) 201-209
Septiembre, 2013 | **DOI:** 10.1159/000342042

Rationale: Talc is very effective for pleurodesis, but there is concern about complications, especially acute respiratory distress syndrome. Objectives: It was the aim of this study to investigate if talc with a high concentration of small particles induces greater production of cytokines, and if pleural tumor burden has any influence on the local production and spillover of cytokines to the systemic circulation and eventual complications. Methods: We investigated 227 consecutive patients with malignant effusion submitted to talc pleurodesis. One hundred and three patients received ‘small-particle talc' (ST; containing about 50% particles <10 µm) and 124 received ‘large-particle talc' (with <20% particles <10 µm). Serial samples of both pleural fluid and blood were taken before and 3, 24, 48 and 72 h after thoracoscopy. Also, mesothelial cells were stimulated with both types of talc in vitro. Measurements and Results: Interleukin-8, tumor necrosis factor-α, vascular endothelial growth factor, basic fibroblast growth factor and thrombin-antithrombin complex were measured in all samples. Early death (<7 days after talc) occurred in 8 of 103 patients in the ST and in 1 of 124 in the ‘large-particle talc' group (p = 0.007). Patients who received ST had significantly higher proinflammatory cytokines in pleural fluid and serum after talc application, and also in supernatants of the in vitro study. Pleural tumor burden correlated positively with proinflammatory cytokines in serum, suggesting that advanced tumor states induce stronger systemic reactions after talc application. Conclusions: ST provokes a strong inflammatory reaction in both pleural space and serum, which is associated with a higher rate of early deaths observed in patients receiving it.

Light induced hydrophilicity and osteoblast adhesion promotion on amorphous TiO_2
Terriza, A; Diaz-Cuenca, A; Yubero, F; Barranco, A; Gonzalez-Elipe, AR; Caballero, JLG; Vilches, J; Salido, M
Journal of Biomedical Materials Research A, **101A** (2013) 1026-1035
Abril, 2013 | **DOI:** 10.1002/jbm.a.34405

We have studied the effect of the UV induced superhydrophilic wetting of TiO_2 thin films on the osteoblasts cell adhesion and cytoskeletal organization on its surface. To assess any effect of the photo-catalytic removal of adventitious carbon as a factor for the enhancement of the osteoblast development, 100 nm amorphous TiO_2 thin layers were deposited on polyethylene terephthalate (PET), a substrate well known for its poor adhesion and limited wettability and biocompatibility. The TiO_2/PET materials were characterized by X-ray photoelectron spectroscopy, and atomic force microscopy and their wetting behavior under light illumination studied by the sessile drop method. The amorphous TiO_2 thin films showed a very poor photo-catalytic activity even if becoming superhydrophilic after illumination. The illuminated samples recovered partially its initial hydrophobic state only after their storage in the dark for more than 20 days. Osteoblasts (HOB) were seeded both on bare PET and on TiO_2/PET samples immediately after illumination and also after four weeks storage in darkness. Cell attachment was much more efficient on the immediately illuminated TiO_2/PET samples, with development of focal adhesions and cell traction forces. Although we cannot completely discard some photo-catalytic carbon removal as a factor contributing to this cell enhanced attachment, our photodegradation experiments on amorphous TiO_2 are conclusive to dismiss this effect as the major cause for this behavior.

Effects of plasma surface treatments of diamond-like carbon and polymeric substrata on the cellular behavior of human fibroblasts

Lopez-Santos, C; Fernandez-Gutierrez, M; Yubero, F; Vazquez-Lasa, B; Cotrino, J; Gonzalez-Elipe, A; San Roman, J

Journal of Biomaterials Applications, **27** (2013) 669-683

Febrero, 2013 | **DOI:** 10.1177/0885328211422832

Surface properties play an important role in the functioning of a biomaterial in the biological environment. This work describes the influence of the changes that occurred on diamond-like carbon (DLC) and polymeric substrata by different nitrogen and ammonia plasmas treatments and its effects on the cell proliferation on these materials. All substrata were additionally subjected to the effect of neutral beams of nitrogen atoms and NH species for comparison purposes. Results about the proliferation, viability, and morphology of fibroblasts were correlated with surface chemical composition, surface tension, and topography. It was found that the presence of amine groups on the surface and the surface tension are beneficial factors for the cell growth. Surface roughness in DLC also plays a positive role in favoring cell adhesion and proliferation, but it can be detrimental for some of the treated polymers because of the accumulation of low molecular weight fragments formed as a result of the plasma treatments. Analysis of the overall results for each type of material allowed to define a unique parameter called ‘factor of merit’ accounting for the influence of the different surface characteristics on the cell deployment, which can be used to predict qualitatively the efficiency for cell growth.

Growth of silver on ZnO and SnO_2 thin films intended for low emissivity applications

Alvarez, R; Gonzalez, JC; Espinos, JP; Gonzalez-Elipe, AR; Cueva, A; Villuendas, F

Applied Surface Science, **268** (2013) 507-515

Marzo, 2013 | **DOI:** 10.1016/j.apsusc.2012.12.156

In the present work we have investigated the relationships existing between the optical properties and the growth mechanism, microstructure and surface roughness of SnO_2 and ZnO oxide films prepared by magnetron sputtering under conditions resembling those utilized in industry. Thin films of these oxides with different thicknesses were characterized by atomic force microscopy, glancing incidence X-ray diffraction (GIXRD), X-ray reflectometry and spectroscopic ellipsometry. The roughness evolution of the film properties (density, surface roughness and refraction index) as a function of their thickness has been evaluated within the concepts of the Dynamic Scaling Theory of thin film growth. Zinc oxide films were rougher than tin oxide films of similar thickness, indicating a different growing mechanism for the two materials. Silver was evaporated onto the surface of the two oxide thin films and its earlier stages of nucleation studied by background analysis of the X-ray photoemission spectra. A different nucleation mechanism was found depending on the nature of the oxide acting as substrate. The superior performance of the zinc oxide based low emissive coatings is related with a better wetting of silver on the surface of this oxide despite the comparatively lower roughness of the tin oxide layers.

Atomistic model of ultra-smooth amorphous thin film growth by low-energy ion-assisted physical vapour deposition

Alvarez, R; Vazquez, L; Gago, R; Redondo-Cubero, A; Cotrino, J; Palmero, A
Journal of Physics D: Applied Physics, **46** (2013) 395303
Septiembre, 2013 | **DOI:** 10.1088/0022-3727/46/39/395303

The growth of ultra-smooth amorphous thin films induced by low-energy (below 1 keV) ion-assistance processes is studied. The relative contribution of ion-induced smoothening effects is analysed by means of a Monte Carlo model and experimental data. In general, highly rough granular or ultra-smooth (with roughness below one monolayer) films are produced depending on the competition between surface shadowing and ion-induced adatom mobility and sputtering. The ultra-smooth growth regime is experimentally and theoretically consistent with the Edwards–Wilkinson growth mode, which is related to the ion-induced enhancement of surface mobility. Overall, the framework and the fundamentals to analyse this type of growth are developed and discussed.

Thermal Expansion of Rare-Earth Pyrosilicates

Fernandez-Carrion, AJ; Allix, M; Becerro, AI
Journal of the American Ceramic Society, **96** (2013) 2298-2305
Julio, 2013 | **DOI:** 10.1111/jace.12388

The use of $RE_2Si_2O_7$ materials as environmental barrier coatings (EBCs) and in the sintering process of advanced ceramics demands a precise knowledge of the coefficient of thermal expansion of the $RE_2Si_2O_7$. High-temperature X-ray diffraction (HTXRD) patterns were collected on different $RE_2Si_2O_7$ polymorphs, namely A, G, α, β, γ, and δ, to determine the changes in unit cell dimensions. $RE_2Si_2O_7$ compounds belonging to the same polymorph showed, qualitatively, very similar unit cell parameters behavior with temperature, whereas the different polymorphs of a given $RE_2Si_2O_7$ compound exhibited markedly different thermal expansion evolution. The isotropy of thermal expansion was demonstrated for the A-$RE_2Si_2O_7$ polymorph while the rest of polymorphs exhibited an anisotropic unit cell expansion with the biggest expansion directed along the REOx polyhedral chains. The apparent bulk thermal expansion coeficcients (ABCTE) were calculated from the unit cell volume expansion for each $RE_2Si_2O_7$ compound. All compounds belonging to the same polymorph exhibited similar ABCTE values. However, the ABCTE values differ significantly from one polymorph to the other. The highest ABCTE values correspond to A-$RE_2Si_2O_7$ compounds, with an average of 12.1 × 10−6 K−1, whereas the lowest values are those of β- and γ-$RE_2Si_2O_7$, which showed average ABCTE values of ~4.0 × 10−6 K−1.

Synthesis and functionalization of biocompatible Tb:$CePO_4$ nanophosphors with spindle-like shape

Rodriguez-Liviano, S; Aparicio, FJ; Becerro, AI; Garcia-Sevillano, J; Cantelar, E; Rivera, S; Hernandez, Y; de la Fuente, JM; Ocana, M

Journal of Nanoparticle Research, **15** (2013) 15:1402

Enero, 2013 | **DOI:** 10.1007/s11051-012-1402-7

Monoclinic Tb:$CePO_4$ nanophosphors with a spindle-like morphology and tailored size (in the nanometer and micrometer range) have been prepared through a very simple procedure, which consists of aging, at low temperature (120 °C), ethylene glycol solutions containing only cerium and terbium acetylacetonates and phosphoric acid, not requiring the addition of surfactants or capping agents. The influence of the heating mode (conventional convection oven or microwave oven) and the Tb doping level on the luminescent, structural and morphological features of the precipitated nanoparticles have also been analyzed. This study showed that microwave-assisted heating resulted in an important beneficial effect on the luminescent properties of these nanophosphors. Finally, a procedure for the functionalization of the Tb:$CePO_4$ nanoparticles with aspartic-dextran is also reported. The functionalized nanospindles presented negligible toxicity for Verocells, which along with theirs excellent luminescent properties, make them suitable for biomedical applications.

Combined reactive magnetron sputtering and plasma decomposition of non-volatile precursors to grow luminescent thin films

Gil-Rostra, J; Yubero, F; Ferrer, FJ; Gonzalez-Elipe, AR

Surface and Coatings Technology, **222** (2013) 144-150

Mayo, 2013 | **DOI:** 10.1016/j.surfcoat.2013.02.016

This paper reports a new procedure of the preparation of mixed oxide thin films that combines the traditional reactive magnetron sputtering deposition with the plasma activated decomposition of non-volatile precursors sublimated by means of an effusion cell. The possibilities of this new experimental procedure are illustrated with the preparation of luminescent thin films consisting of rare earth (RE) cations (Tb^{3+}, Eu^{3+}) incorporated in an oxide matrix (TiO_2 and SiO_2). The oxide matrix component was supplied by reactive magnetron sputtering from metallic Ti or Si targets, while the RE cation was dosed by sublimation of acetylacetonate compounds of the selected elements. The obtained mixed oxide thin films have been fully characterized by different methods and their luminescent properties studied as a function of the matrix type and concentration of the RE element present in the film. The advantages of the synthesis procedure are highlighted with regard to its versatility and the possibility of tailoring the properties of complex luminescent materials.

Differences in n-type doping efficiency between Al- and Ga-ZnO films

Quintanilla, M; Nunez, Gabas, M; Landa-Canovas, A; Costa-Kramer, JL; Agullo-Rueda, F; Gonzalez-Elipe, AR; Diaz-Carrasco, P; Hernandez-Moro, J; Lorite, I; Herrero, P; Castillero, P; Barranco, A; Ramos-Barrado, JR

Journal of Applied Physics, **113** (2013) 163709 (9 pages)
Abril, 2013 | **DOI:** 10.1063/1.4803063

A careful and wide comparison between Al and Ga as substitutional dopants in the ZnO wurtzite structure is presented. Both cations behave as n-type dopants and their inclusion improves the optical and electrical properties of the ZnO matrix, making it more transparent in the visible range and rising up its electrical conductivity. However, the same dopant/Zn ratio leads to a very different doping efficiency when comparing Al and Ga, being the Ga cation a more effective dopant of the ZnO film. The measured differences between Al- and Ga-doped films are explained with the hypothesis that different quantities of these dopant cations are able to enter substitutionally in the ZnO matrix. Ga cations seem to behave as perfect substitutional dopants, while Al cation might occupy either substitutional or interstitial sites. Moreover, the subsequent charge balance after doping appear to be related with the formation of different intrinsic defects that depends on the dopant cation. The knowledge of the doped-ZnO films microstructure is a crucial step to optimize the deposition of transparent conducting electrodes for solar cells, displays, and other photoelectronic devices.

Energy transfer efficiency in YF3 nanocrystals: Quantifying the Yb^{3+} to Tm^{3+} infrared dynamics

Quintanilla, M; Nunez, NO; Cantelar, E; Ocana, M; Cuss, F

Journal of Applied Physics, **113** (2013) 174308 (6 pages)
Mayo, 2013 | **DOI:** 10.1063/1.4803540

In this work, we report on the determination of the infrared $Yb^{3+} \rightarrow Tm^{3+}$ energy transfer efficiency in $YF3:Yb^{3+}/Tm^{3+}$ nanocrystals through the study of Yb3+ dynamics. The obtained results are compared to those previously reported in macrocrystals to analyze possible changes related to size reduction. Luminescence lifetimes are much shorter in the nanoparticles than in bulk samples, a behavior that can be related to $Yb^{3+} \rightarrow Yb^{3+}$ migration and the enhanced surface/volume ratio of the nanoparticles. On the other hand, $Yb^{3+} \rightarrow Tm^{3+}$ energy transfer macroparameter remains unaltered, demonstrating that spectroscopic intrinsic parameters such as radiative and non-radiative probabilities are not affected by size reduction. Finally, a formula that describes Yb3+ lifetime dependence with Yb^{3+} and Tm^{3+} concentration is proposed, considering both the effects produced by migration between Yb^{3+} ions and energy transfer from Yb^{3+} to Tm^{3+} ions.

$LaPO_4$:Er microspheres with high NIR luminescent quantum yield
Garcia-Sevillano, J; Cantelar, E; Justo, A; Ocana, M; Cusso, F
Materials Chemistry and Physics, **138** (2013) 666-671
Marzo, 2013 | **DOI:** 10.1016/j.matchemphys.2012.12.036

Er-doped $LaPO_4$ microspheres have been synthesized by spray pyrolysis and the near infrared (NIR) properties have been characterized. It has been found that, following an adequate post-annealing treatment, the emission properties are remarkably improved. The NIR luminescence intensity is highly enhanced and its decay time increases to a value almost coincident with the reported radiative lifetime, which implies that the quantum yield approaches $\eta \approx 100\%$. This improvement in luminescence characteristics is probably related to the suppression of residual OH− radicals, that otherwise act as NIR luminescence quenchers, and to the increase in material's crystallinity.

Enhancement of visible light-induced surface photo-activity of nanostructured N–TiO_2 thin films modified by ion implantation
Romero-Gomez, P; Lopez-Santos, C; Borras, A; Espinos, JP; Palmero, A; Gonzalez-Elipe, AR
Chemical Physics Letters, **582** (2013) 95-99
Septiembre, 2013 | **DOI:** 10.1016/j.cplett.2013.07.025

This work reports the morphological and chemical modifications induced in TiO_2 thin films by bombardment with high energy N+ ions at different temperatures and their different photo-activity responses after implantation under visible and UV light illumination. When implanted samples are illuminated with visible light, no dye photo-decolouration takes place despite that light transformed the surfaces from hydrophobic to hydrophilic. In agreement with the Wenzel model of wetting, correlation is found between visible light photo-activity and film morphology. We conclude that the photo-activity response can be separated into shallow and Schottky barrier photo-activity, this latter involving a thicker layer of material.

Symmetry analysis of the numerical instabilities in the transfer matrix method
Luque-Raigon, JM; Halme, J; Miguez, H; Lozano, G
Journal of Optics, **15** (2013) 125719
Diciembre, 2013 | **DOI:** 10.1088/2040-8978/15/12/125719

This paper discusses the numerical exponential instability of the transfer matrix method (TMM) in the framework of the symmetry formalism. This numerical weakness is attributed to a series of increasingly extreme exponentials that appear in the TMM when it is applied to geometries involving total internal reflection (TIR) or very high absorption. We design a TMM formalism that identifies the internal symmetries of the multilayer geometry. These symmetries suggest particular transformations of a reference system in the TMM that improve its ill-conditioned exponentials. To illustrate the numerical improvements, we present examples with calculations of electric fields.

Small Pt nanoparticles on the TiO_2 (110)–(1 × 2) surface
Sanchez-Sanchez, C; Martin-Gago, JA; Lopez, MF
Surface Science, **607** (2013) 159-163

Enero, 2013 | **DOI:** 10.1016/j.susc.2012.08.028

Scanning tunnelling microscopy (STM) has been used to study the initial stages of Pt deposition on the TiO_2 (110)–(1 × 2) surface. Experimental STM images recorded for Pt coverage of 0.1 and 0.4 ML, suggest a Volmer-Weber growth. For low coverage and RT deposition, small clusters homogeneously distributed on the surface terraces are observed. However, after annealing at 825 K, material agglomeration, with nucleation mainly at the cross-links, is observed as a consequence of Pt diffusion on the surface. Finally, the structure of small clusters has been determined, in good agreement with previous theoretical calculations.

Valence band electronic structure characterization of the rutile TiO_2 (110)-(1 x 2) reconstructed surface

Sanchez-Sanchez, C; Garnier, MG; Aebi, P; Blanco-Rey, M; de Andres, PL; Martin-Gago, JA; Lopez, MF

Surface Science, **608** (2013) 92-96

Febrero, 2013 | **DOI:** 10.1016/j.susc.2012.09.019

The electronic structure of the TiO_2 (110)-(1 × 2) surface has been studied by means of angular resolved ultraviolet photoemission spectroscopy (ARUPS). The valence band dispersion along the high symmetry surface directions, [001] and [1–10], has been recorded. The experimental data show no dispersion of the band-gap Ti 3d states. However, the existence of dispersive bands along the [001] direction located at about 7 eV below the Fermi level is reported. The existence of two different contributions in the emission from the defects-related state located in the gap of the surface is univocally shown for the first time.

Low refractive index SiOF thin films prepared by reactive magnetron sputtering

Garcia-Garcia, FJ; Gil-Rostra, J; Terriza, A; Gonzalez, JC; Cotrino, J; Frutos, F; Ferrer, FJ; Gonzalez-Elipe, AR; Yubero, F

Thin Solid Films, **542** (2013) 332-337

Septiembre, 2013 | **DOI:** 10.1016/j.tsf.2013.07.009

We have studied low refractive index fluorine doped silica thin films prepared by reactive magnetron sputtering. Two experimental parameters were varied to increase the porosity of the films, the geometry of the deposition process (i.e., the use of glancing angle deposition) and the presence of chemical etching agents (fluorine species) at the plasma discharge during film growth. The microstructure, chemistry, optical properties, and porosity of the films have been characterized by scanning electron and atomic force microscopy, Fourier transform infrared spectroscopy, X-ray photoelectron spectroscopy, UV–vis, and spectroscopic ellipsometry. It is found that either the deposition at glancing angles or the incorporation of CF_x species in the plasma discharge during film growth produces a decrease in the refractive index of the deposited films. The combined effect of the two experimental approaches further enhances the porosity of the films. Finally, the films prepared in a glancing geometry exhibit negative uniaxial birefringence.

Preparation and characterization of CrO_2 films by Low Pressure Chemical Vapor Deposition from CrO_3

Aguilera, C; Gonzalez, JC; Borras, A; Margineda, D; Gonzalez, JM; Gonzalez-Elipe, AR; Espinos, JP

Thin Solid Films, **539** (2013) 1-11
Julio, 2013 | **DOI:** 10.1016/j.tsf.2013.04.118

Highly oriented CrO_2 thin films have been heteroepitaxially grown on TiO_2 rutile (110), (100) and (001) single crystalline substrates, by Low Pressure Chemical Vapor Deposition from CrO3 as precursor and flowing oxygen as carrier gas, under a pressure of 67 Pa. The experimental conditions were fine tuned by depositing on polycrystalline Ti foils, to improve the purity of the films and the deposition rate. A maximum deposition rate of 175 nm h− 1 was obtained.
The composition and texture of films, up to 2 µm thick, have been determined by X-ray diffraction (XRD) and Micro Raman, while their microstructure has been examined by Scanning Electron Microcopy and Atomic Force Microscopy, and their magnetic behavior has been tested by superconducting quantum interference device magnetometry. These techniques reveal that the phase purity, texture, microstructure and thickness of these films are dependent on the crystalline face of the rutile substrate and the deposition temperature. Thus, microscopy techniques, XRD and Raman spectroscopy confirm that highly textured CrO_2 films were always obtained on the three rutile substrate faces when deposition temperature ranges between 616 K and 636 K. But these techniques also show that CrO_2 films are unpurified with inclusions or patches of Cr_2O_3, for the most of the substrates and especially at high deposition temperatures. Magnetic measurements conclusively demonstrate that pure CrO_2 films are only obtained when TiO_2 (110) is used as a substrate.

Growth of SiO_2 and TiO_2 thin films deposited by reactive magnetron sputtering and PECVD by the incorporation of non-directional deposition fluxes

Alvarez, R; Romero-Gomez, P; Gil-Rostra, J; Cotrino, J; Yubero, F; Gonzalez-Elipe, AR; Palmero, A

Physica Status Solidi (a), **210** (2013) 796-801
Abril, 2013 | **DOI:** 10.1002/pssa.201228656

We have deposited TiO_2 and SiO_2 thin films by techniques as different as plasma-enhanced chemical vapor deposition (PECVD) and reactive magnetron sputtering under experimental conditions where highly directional deposition fluxes are avoided. The results indicate that whatever the deposition technique employed or even the precursor gas in the PECVD technique, films share common microstructural features: a mounded surface topography and a columnar arrangement in the bulk, with the column width growing linearly with film thickness. With the help of a Monte Carlo model of the deposition, we conclude that these common aspects are explained by solely taking into consideration the incorporation of a low-energy, isotropically directed, deposition flux onto a substrate at low temperature and under a weak plasma/surface interaction environment.

Electrochromism in WOx and WxSiyOz Thin Films Prepared by Magnetron Sputtering at Glancing Angles

Garcia-Garcia, FJ; Gil-Rostra, J; Yubero, F; Gonzalez-Elipe, AR
Nanoscience and Nanotechnology Letters, **5** (2013) 89-93
Enero, 2013 | **DOI:** 10.1166/nnl.2013.1449

This work reports the electrochromic evaluation of WxSiyOz and WOx glad thin films deposited by reactive magnetron sputtering at glancing angle. Their electrochemical properties were assessed by the analysis of cyclic voltammetry and chronoamperometry measurements in 0.1 M HClO4, whereas their optical properties were determined by studying their transmission and absorption spectra under operation conditions. Both types of thin films presented outstanding electrochromic properties characterized by a fast response, a high coloration and a complete reversibility after more than one thousand cycles.

Reserve osmosis membranes oxidation by hypochlorite dioxide: spectroscopic techniques vs. Fujiwara test
Sandin, R; Ferrero, E; Repolles, C; Navea, S; Bacardit, J; Espinos, JP; Malfeito, JJ
Desalination and water treatment, **51** (2013) 318-327
Enero, 2013 | **DOI:** 10.1080/19443994.2012.700010

The aim of this work was the study of degradation of a commercial polyamide membrane by two commonly employed oxidants for disinfection in seawater desalination, hypochlorite, and chlorine dioxide. A conventional reverse osmosis (RO) membrane is a thin film composite membrane composed of three different layers, a polyester support web, a microporous polysulfone interlayer, and a thin cross-linked polyamide barrier layer on the top surface, which is the active layer of the RO membrane. The degree of membrane degradation in seawater was evaluated in terms of decline in membrane performance calculated from permeability and salt rejection. In order to establish a relationship between the hydraulic properties and spectroscopic data, infrared and X-ray photoemission techniques (ATR-FTIR and XPS) were employed. The obtained results were compared with the Fujiwara test which is usually performed in membrane autopsies to check the degradation of polyamides with halogens. The chemical degradation of the surface active layer was analyzed using infrared spectroscopy (ATR-FTIR) by monitoring the changes in the characteristic infrared bands of the polyamide. It is possible to calculate the transmittance bands ratio between peak at 1540 cm(-1) (due to amide II) and peak at 1585 cm(-1) (due to the polysulfone layer) in order to get the comparison of the degraded membranes with a virgin membrane. The amide II band was selected to evaluate the degradation process, because it is the first band that reduces its transmittance value when the degradation process begins. Once the ratio is obtained for the degraded membrane and considering the value obtained from the virgin membrane as the reference point, a new index is calculated named as degradation index. The higher the parameter is, the greater the chemical attacks the polyamide layer. X-ray spectroscopy (XPS) measures the elemental composition and the chemical state of the elements that exist in the surface of a solid. Evaluation of the binding energy is possible to determine if the halogens are attached to the polyamide structure. It was concluded in this work that both spectroscopic techniques ATR-FTIR and XPS could detect the membrane degradation process earlier than Fujiwara test.

LIBROS Y OTRAS PUBLICACIONES / BOOKS AND OTHER PUBLICATIONS

Responsive Bragg Reflectors
Mauricio Calvo, Hernán Míguez
En: Responsive Nanostructures, Smart, Nanoscale Optical Materials, (2013) 1-20 (Editorial Royal Society of Chemistry) 1-20
ISBN: 978-1-84973-653-4

Women and chemistry. part I. from antiquity to seventeenth century | Mujeres y química parte I. de la antigüedad al siglo XVII
Páez, A.M.; Garritz, A
Educación Química, **24** (2013) 2-7

Women and chemistry. part IV. Twentieth and Twenty-first centuries | mujeres y química. Parte IV. Siglos XX y XXI
Páez, A.M.; Garritz, A
Educación Química, **24** (2013) 326-334

Women and chemistry. Part II. Eighteenth and nineteenth centuries | Mujeres y química II. Siglos XVIV y XIX
Páez, A.M.; Garritz, A
Educación Química, **24** (2013) 156-162

Plasmas fríos moleculares. Química de plasmas
González-Elipe, A.R.; Gordillo-Vázquez, F.J.; Tabarés, F.L.; Tanarro, I.
Revista Española de Física, **27** (2013) 27-50

Marie Sklodowska-Curie y la radioactividad
Adela Muñoz Páez
Mujeres en la Química – Educ. Quím., **24** (2013) 224-228

El todo y la parte
Rafael Alvarez, Alberto Palmero y José Miguel García-Martín
Química e Industria., **42** (2013) abril-junio

Oro metálico con nanoporosidad controlada: un material de gran interés para eliminación de gases nocivos para el medio ambiente
R. Alvarez; J. M. García-Martín; M. Macías-Montero; L. González-García; J. C. González; V. Rico; J. Perlich; J. Cotrino; A. R. González-Elipe; A. Palmero
Químicos del Sur, 95 (2013) junio

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

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

Materials Research Society Fall Meeting
1 – 6 diciembre [Boston, Estados Unidos de América]

Hernán Míguez García [Symposium Organizer]

COMUNICACIONES EN CONGRESOS Y REUNIONES / COMMUNICATIONS IN CONGRESS AND MEETINGS

ImagineNano 2013 - NanoSpain Conference
23 – 26 abril [Bilbao, España]

Nanoporous gold thin films deposited by magnetron sputtering: tayloring the porosity
R. Álvarez; J. M. García-Martín; M. Macías-Montero; L. González-García; J.C. González; P. Romero-Gómez; V. Rico; J. Perlich; J. Cotrino; A.R. González-Elipe; A. Palmero
Comunicación oral

Highly monodisperse spindle-like calcium hydroxyapatite and fluoroapatite nanoparticles for biomedical applications
Alberto Escudero, Jesús M. de la Fuente, and Manuel Ocaña
Poster

Hybrid and Organic Photovoltaics Conference – HOPV 2013
5 – 8 mayo [Sevilla, España]

Resonant Photocurrent Generation in Nanostructured Sensitized TiO2 Photoconductors by Optical Field Confinement Effects
M. Anaya; M. E. Calvo; J. M. Luque; H. Míguez
Poster

Optical designs of dye solar cells for enhanced light harvesting
Hernán Míguez García; Mauricio Ernesto Calvo; Miguel Anaya Martín; Francisco Gálvez; Silvia Colodrero; María del Carmen López López
Ponencia Invitada

E-MRS 2013 SPRING MEETING
27 – 31 mayo [Strasbourg, Francia]

Optical designs of dye solar cells for enhanced light harvesting
Hernán Míguez García; Mauricio Ernesto Calvo; Miguel Anaya Martín; Francisco Gálvez; Silvia Colodrero; María del Carmen López López
Ponencia Invitada

International Conference on Ion Beam Analysis 2013
23 junio [Seattle, Washington, Estados Unidos de América]

Simultaneous quantification of light elements in thin films deposited on Si substrates using proton-RBS
F.J. Ferrer, M. Alcaire, J. Caballero, F.J. García García, J. Gil Rostra, A. Terriza, V. Godinho, J. García López, A. Barranco, A. Fernández Camacho
Poster

Al-NanoFunc Workshops 2013
1 – 2 julio [Sevilla, España]

Plasmonics for beaming LED emission
G. Lozano, S. R. K. Rodriguez, M. A. Verschuuren and J. Gomez Rivas
Conferencia Invitada

Periodical structures to improve light harvesting in Dye Solar Cell
Carmen López-López, Silvia Colodrero and Hernán Míguez
Comunicación oral

Tailored Luminescent Emission of Dyes Embedded in Porous Resonators
A. Jiménez-Solano, J. M. Luque, M. E. Calvo, F. Fernández-Lázaro, H. Míguez
Comunicación oral

An electron microscopy study of the nanostruture of collagen fibrils
S. Borrego-González, J. Becerra, A. Díaz-Cuenca
Poster

$BiPO_4$ nanostars for luminescent applications
Joaquín Criado, Ana Isabel Becerro, Lionel C. Gontard, Asunción Fernández, Manuel Ocaña
Poster

XVII International Sol-Gel Conference - Sol-Gel 2013
25-30 julio [Sevilla, España]

Sol-gel Synthesized $La_{2-x}H_{ox}Si_2O_7$ phosphors: La/Ho Substitution mechanism by anomalous synchrotron X-ray diffraction and luminescent properties
A.J. Fernández-Carrión, M. Allix, A. N. Fitch, M. Ocaña, A. I. Becerro
Poster

European Conference on Advanced Materials and Processes 2013 - EUROMAT

8 – 13 septiembre [Sevilla, España]

Nanoporous and mesoporus photonic multilayers for applications in sensing
R. Álvarez; J. M. García-Martín; M. Macías-Montero; L. González-García; J.C. González; V. Rico; J. Perlich; J. Cotrino; A.R. González-Elipe; A. Palmero
Comunicación oral

Integration of Gold Nanoparticles in Photonic Crystals: New Base Materials for Optical Sensing Based on the Effect of the Interplay between Plasmonic and Optical
A. Jiménez-Solano, C. López-López, O. Sánchez-Sobrado, J. M. Luque, M. E. Calvo, C. Fernández-López, A. Sánchez-Iglesias, L. M. Liz-Marzán, H. Míguez
Comunicación oral

Flexible one-dimensional photonic crystals based on nanoparticle multilayers for ultraviolet radiation protection purposes
J.R. Castro Smirnov; M. Calvo; H. Míguez
Comunicación oral

Porous metal oxide layers integrated in photonic structures as base materials for optical sensing
Mauricio Calvo Roggiani; Nuria Hidalgo Serrano; Hernan Miguez Garcia
Ponencia “KEY NOTE”

Periodical structures to improve light harvesting of Dye Solar Cells
M. Carmen López López
Comunicación oral

Nanoporous gold thin films deposited by magnetron sputtering: tayloring the porosity
R. Álvarez, J.M. García-Martín, M. Macías-Montero, L. González-García, J.C. González, V. Rico, J. Perlich, J. Cotrino, A.R. González-Elipe, A. Palmero
Comunicación Oral

Solvent-controlled synthesis and luminescent properties of uniform Eu:YVO4 nanophosphors with different morphologies.
Nuria Nuñez, Jad Sabek, Jorge García-Sevillano, Eugenio Cantelar, Alberto Escudero, Manuel Ocaña
Poster

Trends in Nanotechnology – TNT 2013

9 – 13 septiembre [Sevilla, España]

Light Absorption and Emission of Nanomaterials in Porous Photonic Structures
H. Míguez
Conferencia Invitada

Resonant Photocurrent Generation in Dye-Sensitized Periodically Nanostructured Photoconductors by Optical Field Confinement Effects
M. Anaya; M. Calvo; J.M. Luque; H. Míguez
Comunicación oral

Synthesis and functionalization of biocompatible Tb:$CePO_4$ nanophosphors with spindle-like shape
Ana Isabel Becerro, Sonia Rodriguez-Liviano, Sara Rivera, Yulán Hernández, Jesus M. de la Fuente and Manuel Ocaña
Poster

Ionic Liquid Mediated Synthesis and Surface Modification of Multifunctional Mesoporous Eu:GdF3 Nanoparticles for Biomedical Applications
Sonia Rodriguez-Liviano, Nuria. O. Nuñez, Sara Rivera-Fernández, Jesus M. de la Fuente, Manuel Ocaña
Poster

Integration of Gold Nanoparticles in Photonic Crystals: Effect of the interplay between Plasmonic and Optical Cavity Resonances
A. Jiménez-Solano, C. López-López, O. Sánchez-Sobrado, J. M. Luque, M. E. Calvo, C. Fernández-López, A. Sánchez-Iglesias, L. M. Liz-Marzán, H. Míguez
Poster

12th International Conference on Laser Ablation - COLA 2013
6 – 11 octubre [Ischia, Italia]

X-Ray Absorption Spectroscopy Study of the Nb Local Environment in Heavy Metal Oxide Film Glasses Produced by Pulsed Laser Deposition
R. Morea; A. Ruiz de La Cruz; M. Rovezzi; W. Gawelda; J. P. Espinos; J. Gonzalo
Comunicación oral

E-MRS 2013 FALL MEETING
1 – 6 Diciembre [Boston, Estados Unidos de América]

Nanoparticle based porous photonic structures prepared by wet deposition methods
Mauricio Calvo Roggiani; Alberto Jimenez Solano; Jose Castro Smirnov; Hernán Míguez Garcia
Comunicación oral

Photonic structures for highly efficient and transparent dye solar cells
María del Carmen López López; Silvia Colodrero Pérez; Miguel Anaya; Mauricio Calvo Roggiani; Hernán Míguez Garcia
Comunicación oral

Integration of Gold Nanoparticles in Photonic Crystals: New Base Materials for Optical Sensing Based on the Effect of the Interplay between Plasmonic and Optical Cavity Resonances
A. Jiménez-Solano, C. López-López, O. Sánchez-Sobrado, M. E. Calvo, C. Fernández-López, A. Sánchez-Iglesias, L. M. Liz-Marzán, H. Míguez
Comunicación oral

Optical Design of Photo-electrochemical Devices
M. Anaya; M. E. Calvo; H. Míguez
Poster

Formacion / Training

Tesis Doctorales/ Doctor Degree Thesis

Título: **Nanostructured optical materials for dye solar cells**
Autor: M. Carmen López López
Directores: Hernán Míguez García
Calificación: Apto “Cum Laude”
Centro: Universidad de Sevilla

Título: **New Plasma-based synthesis procedures and applications of 1D nanostructures**
Autor: Manuel Macías Montero
Directores: Agustín R. González-Elipe y Ana Isabel Borrás Martos
Calificación: Apto “Cum Laude”
Centro: Universidad de Sevilla

Título: **Funcionalización superficial de polímeros por PECVD para uso biomédico**
Autor: Antonia Terriza Fernández
Directores: Agustín R. González-Elipe y Francisco Yubero Valencia
Calificación: Apto “Cum Laude”
Centro: Universidad de Sevilla

Título: **Porous one dimensional photonic cyrstals for enhanced photovoltaic performance of dye solar cells**
Autor: Silvia Colodero Pérez
Directores: Hernán Míguez García
Calificación: Apto “Cum Laude”
Centro: Universidad de Sevilla

Título: **Láminas delgadas de óxidos mixtos con propiedades ópticas y funcionales obtenidas mediante magnetrón sputtering**
Autor: Jorge Gil Rostra
Directores: Agustín R. González-Elipe y Francisco Yubero Valencia
Calificación: Apto “Cum Laude”
Centro: Universidad de Sevilla

Título: **Control de la nanoestructura en capas esculturales preparadas mediante GLAD: crecimiento y aplicaciones**
Autor: Lola González García
Directores: Agustín R. González-Elipe y Adela Muñoz Páez
Calificación: Apto “Cum Laude”
Centro: Universidad de Sevilla

FORMACIÓN DE GRADUADOS / MASTER DEGREE THESIS

Título: **Generación resonante de fotocorriente en materiales ópticos nanoestructurados**
Autor: Miguel Anaya Martín
Directores: Hernán Ruy Míguez García y Mauricio Calvo Roggiani
Grado: Trabajo Fin de Master
Año Académico: 2012-2013 (11 Julio 2013)

Título: **Grabado de redes de difracción en celdas solares de colorante**
Autor: Reyes Ortiz Marchena
Directores: Hernán Ruy Míguez Gacía
Grado: Trabajo Fin de Master
Año Académico: 2012-2013 (5 Septiembre 2013)

Título: **Síntesis y caracterización de nanopartículas monodispersas luminiscentes**
Autor: Joaquín Criado Reyes
Directores: Manuel Ocaña Jurado y Ana Isabel Becerro Nieto
Grado: Trabajo Fin de Master
Año Académico: 2012-2013 (23 Septiembre 2013)

Título: **Síntesis, funcionalización y propiedades de nanopartículas luminiscentes basadas en $NaGdF_4$**
Autor: María García Medel
Directores: Manuel Ocaña Jurado y Nuria Núñez Alvarez
Grado: Trabajo Fin de Master
Año Académico: 2012-2013 (23 Septiembre 2013)

DOCENCIA / TEACHING

Máster Oficial y Doctorado Interuniversitario en “Biotecnología Avanzada”
Módulo de Nanotecnología
Dra. Aránzazu Díaz Cuenca
Lugar: Universidad de Málaga
Organizado por la Universidad de Málaga y la Universidad Internacional de Andalucía

Máster en Formación del Profesorado de Educación Secundaria
Ciencia, Tecnología y Sociedad
Dra. Adela Muñoz Páez
Lugar: Universidad de Sevilla

PREMIOS Y RECONOCIMIENTOS / PRIZES AND ACKNOWLEDGEMENTS

Premio de Excelencia investigadora de los Jóvenes Sevillanos 2013. La Junta de Gobierno de la Ciudad de Sevilla ha acordado conceder el Premio de Excelencia Investigadora de los Jóvenes Sevillanos 2013 al grupo de Materiales Ópticos Multifuncionales liderado por el investigador científico del CSIC Hernán Ruy Míguez.

La dotación económica del premio son 4000 euros para la promoción y difusión de la excelencia investigadora. El galardón fue convocado con el triple objetivo de reconocer la labor investigadora de los jóvenes sevillanos, dar a conocer sus trabajos fuera de la ciudad y poner en valor la labor investigadora de los jóvenes. Los requisitos para postular al premio eran que al menos el 30% de los integrantes del grupo de investigación tuvieran edades comprendidas entre los 18 y 35 años y que desarrollasen su labor investigadora en Sevilla y Provincia. Los méritos han sido valorados en función de la calidad e interés científico de las líneas de investigación, el nivel de investigación básica, aplicada o tecnológica; los resultados previstos, como transferencias, patentes o impacto social. También, se ha tenido en cuenta los resultados obtenidos y si la investigación está finalizada, además de si está publicada. En este marco, se ha contado con la participación como miembros del jurado de dos investigadores de reconocido prestigio nacional e internacional, como son Miguel Ferrer Baena, coordinador Institucional en Andalucía del Consejo Superior de Investigaciones Científicas, y Francisco Javier Rojo Marcos, Investigador Científico en el Instituto de Investigaciones Químicas, integrante del cicCartuja, y coordinador del Área de Química del CSIC.

El grupo, integrado en un 70% por investigadores menores de 35 años, publica frecuentemente en las revistas más prestigiosas del campo de materiales (Nature Materials, Advanced Materials, Journal of the American Chemical Society, Energy and Environmental Science...) y está financiado por el European Research Council a través de uno de los programas de investigación más competitivos de Europa, el Starting Grant. Su vertiente de investigación fundamental se ve complementada por distintos contratos de I+D+i con empresas del sector de la energía solar. Además, los miembros del grupo han sido galardonados en dos ocasiones con el Premio cicCartuja-Ebro Foods.

M. Carmen López López, segundo accésit, Premio CicCartuja Ebro Foods 2013. La cuarta edición del Premio de Investigación cicCartuja Ebro Foods ha tenido como protagonista a la quí-

mica. Un estudio que aborda la síntesis de moléculas con enlaces múltiples ha logrado el principal galardón; mientras que los accésits han sido otorgados a trabajos desarrollados en áreas como la química organometálica, la nanobiotecnología o la ciencia de los materiales. Estos premios, que pretenden impulsar la carrera de los investigadores noveles, se han entregado hoy en el Centro de Investigaciones Científicas Isla de la Cartuja (cicCartuja), centro mixto del Consejo Superior de Investigaciones Científicas (CSIC), Junta de Andalucía y Universidad de Sevilla.

El trabajo de Carmen López se enmarca en la línea de investigación en torno a los materiales ópticos multifuncionales. Junto a su grupo de investigación, ha diseñado cristales fotónicos para introducirlos en celdas solares de colorante. Según Carmen López, "el punto fuerte" de su estudio no consiste sólo en mejorar el rendimiento de las celdas solares, sino además mantener su transparencia, "con lo cual este nuevo diseño de celda es apropiado para usarse como módulo de ventana fotovoltaico". Asimismo, este diseño forma de una patente que en la actualidad se encuentra transferida a la empresa.

Gabriel Lozano, Premio de Investigación Real Maestranza de Caballería 2013, por sus investigaciones en el Campo de la Física.

El Dr. Gabriel Lozano, Licenciado en Física por la Universidad de Córdoba (2006) y Doctor por la Universidad de Sevilla (2011), ambos con Premio Extraordinario, Investigador Predoctoral en el Instituto de Ciencia de Materiales de Sevilla, es desde el año 2011 Investigador Postdoctoral en el Center for Nanophotonics, FOM Institute AMOLF, Eindhoven (Holanda). Posee más de 27

artículos en revistas indexadas del más alto índice dentro de su especialidad y más de doscientas cincuenta citas; así como tres patentes transferidas. En sus primeros trabajos aborda el estudio de cuestiones fundamentales que afectan a diferentes aspectos de los cristales fotónicos autoensamblados (ópalos artificiales); analiza la respuesta óptica de filtros interferenciales o espejos de Bragg porosos y desarrolla un modelo analítico para determinar la respuesta óptica de estos materiales nanoestructurados. Posteriormente se ha centrado en el estudio de las propiedades de emisión de diferentes fuentes de luz acopladas a nanoestructuras metálicas ordenadas, demostrando un aumento significativo de las funciones de emisores muy eficientes empleando nanoestructuras, y llegando a proponer soluciones relevantes desde un punto de vista tecnológico.

ESTANCIAS Y VISITAS DE PERSONAL DEL ICMS EN OTROS CENTROS
PERSONNEL OF THE ICMS IN OTHER LABORATORIES

FUNDP. Universidad de NAMUR		
Namur, Bélgica	**Alberto Palmero Acebedo**	2 días
	Rafael Alvarez Molina	2 días
Fitzwilliam College. Department of Chemistry University of Cambridge		
Cambridge, Inglaterra	**Manuel Macías Montero**	2 meses
ESRF		
Grenoble, Francia	**Alberto José Fernández Carrión**	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

Dr. José Jesús Benítez Jiménez
Dra. Rosalía Poyato Galán
Dra. T. Cristina Rojas Ruiz

Investigadores Contratados

Dr. Miguel Castillo Rodríguez
Dra. Vanda C. Fortio Godinho
Dr. Eugenio Zapata Solvas
Dr. Lionel Cervera Gontard
Dr. Roland Schierholz

Becarios Predoctorales

Ldo. Jaime Caballero Hernández
Ldo. Carlos Andrés García Negrete

Personal Contratado

Dra. Gisela M. Arzac de Calvo
Ldo. Santiago Domínguez Meister
Ldo. Dirk Hufschmidt
Lda. Mariana Paladini San Martin
Tec. Salah Rouillon
Lda. Lucia T. Castillo Flores
Ing. Tec. M. Rocío García Gil
Dr. Bertrand Lacroix
Dr. Abdelkader Nebatti Ech Chergui
Lda. Inmaculada de Vargas Parody

Proyectos de Investigación / Research Projects

Laboratorio avanzado para el análisis de nanomateriales funcionales Advanced laboratory for the nano-analysis of novel functional materials (AL-NANOFUNC)

Código/Code:	REGPOT-CT-2011-285895
Periodo/Period:	01-10-2011 / 30-03-2015
Organismo Financiador/Financial source:	Unión Europea
Importe total/Total amount:	2.687.409 €
Investigador responsableICMS Research head ICMS:	Asunción Fernández Camacho
Componentes/Research group:	Cristina Rojas, M. Carmen Jiménez, Gisela Arzac, Olga Montes, Inmaculada Rosa, Rafael Alvarez, Vanda Godinho, Juan Carlos Sánchez-López, Hernán Míguez, Agustín R. González-Elipe, Manuel Ocaña, M. Jesús Sayagués, Lionel Cervera, Roland Schierholz, Salah Rouillon, Lucia Castillo, Rocío García, Carlos García-Negrete, Jaime Caballero

RESUMEN / ABSTRACT

El proyecto AL-NANOFUNC ha sido diseñado para poner en marcha en el Instituto de Ciencia de los Materiales de Sevilla (ICMS, CSIC-Univ.Sevilla, España) un laboratorio avanzado para el Nano-análisis de nuevos materiales funcionales. Las técnicas de Nanoscopía avanzada, basadas en equipos de microscopía electrónica de última generación, se dedicarán a la investigación de vanguardia en temas específicos de gran interés: i) Nanomateriales para aplicaciones energéticas sostenibles; ii) películas delgadas multifuncionales y recubrimientos nanoestructurados; iii) materiales nano-estructurados para fotónica y sensores.

Para situar a los laboratorios del ICMS en una posición de liderazgo que sea competitiva en un escenario mundial, el proyecto AL-NANOFUNC contempla la puesta al día del potencial investigador actual en varias direcciones: i) Mejorar las capacidades de equipamiento en relación a la microscopía electrónica analítica de alta resolución; ii) mejorar el impacto de la investigación básica a través de la contratación de investigadores especializados y el intercambio transnacional con los centros de referencia en Europa, iii) desarrollar y mejorar el potencial de innovación de la investigación del ICMS abriendo las nuevas instalaciones a empresas y centros relacionados; iv) organizar talleres, conferencias y actividades de difusión para mejorar la visibilidad de la investigación.

En el proyecto se propone también una estrecha colaboración con centros de referencia y empresas de Lieja (Bélgica), Graz (Austria), Jülich (Alemania), Oxford (Inglaterra), Cambridge (Inglaterra), Dübendorf (Suiza) y Rabat (Marruecos), así como con laboratorios de Universidades Andaluzas.

Cinco empresas en Andalucía colaborarán también en estrecha sinergia para promover las líneas estratégicas de interés a largo plazo de la región en los productos de piedra natural y artificial y los sectores de energía solar y energías renovables.

The AL-NANOFUNC project has been designed to install and fully develop at the Materials Science Institute of Seville (ICMS, CSIC-Univ.Seville, Spain) an advanced laboratory for the Nano-analysis of novel functional materials. Advanced Nanoscopy facilities, based on latest generation electron microscopy equipments, will be devoted to breakthrough research in specific topics of high interest: i) Nanomaterials for sustainable energy applications; ii) protective and multifunctional thin film and nanostructured coatings; iii) nanostructured photonic materials and sensors.

To take the ICMS laboratories to a leading position that is competitive in a world-wide scenario, the AL-NANOFUNC project is contemplated to up-grade the actual research potential in several directions: i) improve equipment capabilities regarding the Analytical High Resolution Electron Microscopy facilities; ii) improve the impact and excellence of basic research through hiring of experienced researchers and transnational exchange with the reference centers in Europe; iii) develop and improve the innovation potential of the ICMS's research by opening the new facilities to companies and stakeholders; iv) organize workshops and conferences, dissemination and take-up activities to improve research visibility.

Close collaborations with reference centers and companies in Liège (Belgium), Graz (Austria), Jülich (Germany), Oxford (England), Cambridge (England), Dübendorf (Switzerland) and Rabat (Morocco), as well as with laboratories at Andalucian Universities, are foreseen in this project.

Five companies in Andalusia will also collaborate in close synergies to promote the long-term strategic lines of interest for the region in the natural and artificial stone products and solar and renowable energy sectors.

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Desarrollo de recubrimientos nanoestructurados protectores para su uso en condiciones extremas (NANOPROTEXT) Development of nanostructured protective coatings for extreme environmental conditions (NANOPROTEXT)

Código/Code:	MAT2011-29074-C02
Periodo/Period:	01-10-2012 / 31-12-2014
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	129.999,98 €
Investigador responsable/Research head:	Juan Carlos Sánchez López
Componentes/Research group:	T. Cristina Rojas Ruiz; Francisco Javier Pérez Trujillo;Maria del Pilar Hierro de Bengoa;Germán Alcalá Penades; Maria Sonia Mato Díaz; Marta Brizuela; Pablo Corengia; José Luis Viviente; Alberto García;Daniel González

RESUMEN / ABSTRACT

En muchas operaciones industriales, los componentes de las máquinas o piezas que están en contacto se hallan sometidos a condiciones extremas de carga, fricción, temperatura o atmósfera variable. La investigación dirige sus esfuerzos hacia al desarrollo de nuevos recubrimientos multicomponentes capaces de aumentar su eficiencia protegiendo su superficie contra el desgaste y la oxidación, que ocasionan fallos ulteriores de funcionamiento. Mediante el control del tamaño y distribución de las fases componentes, la composición química y su microestructura en el rango nanométrico es posible obtener propiedades multifuncionales tales como baja fricción, dureza y estabilidad térmica.

En este proyecto se propone el desarrollo de tres tipos de recubrimientos nanoestructurados mediante el proceso de magnetron sputtering para aplicaciones protectoras en condiciones extremas o singulares de funcionamiento (presión, temperatura, atmósferas oxidantes, vacío, etc). Los sistemas elegidos comprenden cristales de materiales duros (nitruros o carburos) combinados con una segunda fase o elemento que mejore su comportamiento. De este modo se ensayarán recubrimientos nanocomposite formados por nanocristales de WC dispersos en una segunda fase amorfa de tipo calcogenuro (WS2 or WSe2) para su uso como lubricante sólido en aplicaciones espaciales o bajo atmósferas inertes. En el segundo caso, Y ó Zr serán usados como elementos dopantes dentro de recubrimientos de CrAlN con objeto de incrementar la resistencia a la oxidación a baja y alta temperatura, y el comportamiento tribológico, muy válido en numerosos sectores industriales tales como (herramientas de mecanizado, metalúrgico, aeronaútico, automoción, etc.). Finalmente, se desarrollaran recubrimientos nanocomposite duros y transparentes basados en la familia del Al-Si-N para protección de sistemas ópticos.

En todos los casos, el proyecto comprende su síntesis, caracterización estructural y química, así como su validación práctica en ensayos tribológicos y de oxidación que simulan las condiciones finales de operación. En el caso concreto de las capas duras y transparentes también se evaluarán sus propiedades ópticas. El estudio de la relación existente entre la microestructura y las propiedades medidas será un objetivo esencial puesto que permitirá una mayor comprensión de los mecanismos de actuación, y por ende, la optimización de tales sistemas nanoestructurados para su mejor aprovechamiento tecnológico.

In many industrial operations, the machines or tool components in contact are submitted to severe conditions of load, friction, temperature or variable atmosphere. The research efforts are directed towards the development of new multiphase coatings capable to increase their performance by protection of the surface against wear and oxidation that cause failure mechanisms. By appropriate control of the size and distribution of phases, chemical composition and microstructure in the nanometric regime it is possible to obtain multifunctionality as low friction, hardness and thermal stability. To achieve excel in this purpose it is necessary to correlate the macroscopic properties of these coated surfaces (mechanical, tribological, oxidation resistance) with these basic phenomena.

In this project, three types of nanostructured coatings will be prepared using a magnetron sputtering process for protection in running operations under extreme or singular conditions (pressure, temperature, oxidant atmospheres, vacuum, etc.). The chosen systems are constituted by crystals of hard materials (nitrides or carbides) in combination with a second element or phase that improves the practical performance. Thus, nanocomposite coatings consisting of WC nanocrystals dispersed in an amorphous dichalcogenide phase (WS2 or WSe2) are proposed as solid lubricant coatings to run under high vacuum conditions useful for spatial applications or inert environments. In the second case, Y or Zr will be tested as dopant elements in CrAlN coatings with the aim of increasing the corrosion and oxidation resistance and tribological behaviour useful for many industrial fields (machining tools, metallurgy, aeronautic, automotive, etc…). Finally, hard and transparent nanocomposite coatings based on the Al-Si-N system are suggested as protective coatings for optical systems.

In all cases, the project comprises their synthesis, chemical and structural characterization, and validation in tribological and oxidation under extreme condition tests that simulate the final operation conditions. In the case of the hard and transparent coatings, their optical properties will be also analysed. The establishment of the relationships between microstructure and measured properties will be an essential objective, since it enables the better understanding of the action mechanisms, and thus, the optimisation of such nanostructured multifunctional systems for an improved technological benefit.

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Estudio de las interacciones intermoleculares entre hidroxiácidos carboxílicos de cadena larga como modelo para el diseño de poliésteres biomiméticos Fatty hydroxyacids molecular interactions as model for biomimetic polyester design

Código/Code: CTQ2011-24299
Periodo/Period: 01-01-2012 / 31-12-2014
Organismo Financiador/Financial source: Ministerio de Economía y Competitividad
Importe total/Total amount: 99.220 €
Investigador responsable/Research head: Benítez Jiménez, José Jesús
Componentes/Research group: Alejandro Heredia Guerrero, Miguel Angel San Miguel Barrera, Jaime Oviedo López, Miguel Salmerón Batalle

RESUMEN / ABSTRACT

El objetivo de este proyecto de investigación es el estudio y determinación de las interacciones que se ponen de manifiesto entre las moléculas de ácidos carboxílicos lineales de cadena larga en función del nivel y tipo de funcionalización con grupos hidroxilos. Para ello se proponen sistemas modelos basados en capas autoensambladas de estas moléculas sobre un soporte plano de baja energía de interacción para que sean las propias interacciones intermoleculares las que condicionen la estructura del empaquetamiento. La metodología de estudio de estos sistemas confinados combinará, fundamentalmente, las microscopías de sonda de proximidad, las simulaciones atomísticas de dinámica molecular y el análisis químico por espectroscopía infrarroja. La información fundamental obtenida se empleará para el diseño de una ruta de síntesis química de poliésteres miméticos a la cutina vegetal, un biopolímero de barrera hidrófobo, inocuo y completamente biodegradable. Se determinarán las propiedades físicas (mecánicas, permeabilidad, de transporte iónico, etc…) y químicas (grado de esterificación, degradabilidad, etc…) de estos materiales sintéticos y se relacionarán con sus estructuras primarias (red de enlaces ester) y secundarias (interacciones por puente de hidrógeno entre grupos hidroxilos remanentes). A partir de esta relación estructura-función, se modificará el protocolo de síntesis empleando elementos no propagadoras del entrecruzamiento de la red primaria (moléculas con bajo grado de hidroxilación) y modificadores de la red secundaria (ácidos carboxílicos con distinto grado de hidroxilación), con vistas a la obtención de polímeros con propiedades "a la carta". Finalmente, se explorará el potencial de aplicación de estos poliésteres sintéticos como sustitutos de plásticos obtenidos a partir de hidrocarburos con vistas a reducir residuos, tanto en el proceso de fabricación como los asociados a su desecho.

The objective of this Project is to study and characterize the specific interactions between fatty carboxylic acids molecules arising from selective hydroxylation of the alkyl chain. To address this issue, molecular self-assembled systems showing a low interaction with an atomically flat support are proposed as models. The use of low binding energy supports is to ensure the packing structure to be mainly driven by the molecule to molecule interactions rather than the molecule to substrate adsorption. These self-assembled systems will be characterized by scanning probe microscopies such as AFM and STM and infrared spectroscopy. Results will be complemented with molecular dynamics atomistic simulations. Basic information obtained from self-assembled models will be used to design the in-vitro chemical synthesis of cutin mimetic polyesters. Cutin is a non toxic, fully biodegradable barrier biopolymer present at the skin of fruits, leaves and non lignified stems of higher plants. Physical (mechanical, water permeability, ionic transport, etc…) and chemical (ester yield, controlled chemical degradation rate, etc…) properties of synthetic polyesters will be determined. Material features will be related with both, the main structure arising from the ester bonds formation and the secondary network resulting from hydrogen bonding between spare non reacted hydroxyl groups. Structure-function patterns will be used to redesign the synthesis route to obtain polymeric esters with tailored properties. To achieve this goal, both the primary and secondary structural networks will be manipulated. In the first case non hydroxylated molecules will be used to prevent ester bonding propagation. To modify hydrogen bonding crosslinking, additives with selected hydroxylation (primary or secondary) will be employed. The final motivation of this research is to explore the applicability of such mimetic polyester as substitutes for the highly contaminant hydrocarbon based plastics.

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Desarrollo de nuevos materiales y procesos para la generación y uso del hidrógeno principalmente en aplicaciones portátiles Development of novel materials and processes for the generation and use of hydrogen mainly in portable applications

Código/Code:	CTQ2012-32519
Periodo/Period:	01-01-2013 / 31-12-2015
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	190.710 €
Investigador responsable/Research head:	Fernández Camacho, Asunción

Componentes/Research group: Gisela Arzac, Jaime Caballero, Lionel Cervera, Vanda Fortio, Carlos Negrete, Dirk Hufschmidt, Cristina Rojas Ruiz, Roland Schierholz

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. En el presente proyecto se abordará el estudio de los distintos procesos que conducen a la configuración final integrada de sistemas de generación y uso del hidrógeno principalmente en aplicaciones portátiles (y potencialmente escalables para aplicaciones estacionarias). En particular se trabajará en este proyecto en las siguientes líneas de actuación:

a) Investigación en nuevos compuestos ligeros para su uso en procesos de generación de hidrógeno en pequeña escala por vía química (hidrólisis). Típicamente reacciones de hidrólisis de borohidruros (i.e. NaBH4) y compuestos tipo borano de amoníaco, hidrazinas ó borano hidrazina. Este area incluye el desarrollo de catalizadores en la nanoescala utilizando métodos de vía húmeda para su síntesis: Nanoestructuras metal-metaloide (tipo Co-B, Co-B-P y similares) y catalizadores bimetálicos (que incluyan ó no metaloide) de bajo coste potenciando efectos sinérgicos (tipo CoRu, NiPt ó Co-Ru-B). Incluye también el desarrollo de reactores portátiles para estos procesos y el desarrollo de nuevos sustratos y monolitos, estudios de adherencia del catalizador y durabilidad.

b) Investigación en nuevos sistemas anfitrión-huésped (host-guest) que contengan hidrógeno para el almacenamiento reversible (carga/descarga). Principalmente soportes (anfitrión) porosos del tipo "nanoscaffolds" (basados en C ó BN) infiltrados con borohidruros (huésped) (i.e. borohidruro de titanio) típicamente utilizados para el almacenamiento reversible de hidrógeno. Estos nuevos materiales deben presentar cinéticas de carga y descarga mejoradas.

c) Estudios de acoplamiento de un sistema generador de hidrógeno de bajo coste a una celda de combustible. Típicamente un reactor continúo para la hidrólisis del NaBH4 con catalizador Co-B que suministra H2 en condiciones de flujo constante para alimentar directamente una pila de combustible tipo PEM de 60 W.

d) Estudios fundamentales para el desarrollo de catalizadores y soportes para la combustión controlada de hidrógeno. Es una línea nueva en el grupo de investigación que se basa en preparar por vía húmeda catalizadores nanoparticulados de metal noble sobre soportes comerciales de cerámicas porosas (tipo SiC). Incluye el diseño de un reactor para el estudio en escala laboratorio de la producción de calor por combustión controlada de hidrógeno.

e) Desarrollo de la tecnología de pulverización catódica ("magnetrón sputtering") para la preparación de catalizadores y nano-estructuras sobre diversos sustratos de aplicación en los procesos desarrollados en los apartados anteriores. El grupo tiene una amplia experiencia en esta tecnología que se aplicaría de manera novedosa en este proyecto permitiendo una gran versatilidad en cuanto a la nanoestructura, composición y aditivos para mejorar la actividad, durabilidad y selectividad de los catalizadores.

f) Caracterización microestructural y química de los nuevos materiales y catalizadores desarrollados en el proyecto. Se trata típicamente de materiales con una nanoestructura con-

trolada en donde las modernas técnicas nanoscopicas van a jugar un papel fundamental en la fabricación a medida de estos materiales.

Hydrogen as a vector of transport and storage of energy is a very attractive candidate in the context of increased use of renewable and clean energies. This project will address the study of the different processes that lead to the final configuration of an integrated systems for hydrogen generation and use mainly in portable applications (and potentially scalable for stationary applications). In particular, work will be carried out in this project in the following lines:

a) Research on new lightweight compounds for use in hydrogen generation processes on a small scale by chemical routes (hydrolysis). Typically hydrolysis reactions of borohydrides (i.e. $NaBH_4$) and compounds like ammonia borane, hydrazine borane or hydrazine. This line includes the development of catalysts at the nanoscale using wet chemical methods for their synthesis: Metal-metalloid nanostructures (i.e. Co-B, Co-B-P and similar ones) and bimetallic catalysts (including or not metalloid) of low cost which potentiate synergistic effects (i.e. CoRu, NiPt or Co-Ru-B). The topic also includes the development of portable reactors for these processes and the development of new substrates and monoliths, studies of adherence and durability of the catalyst.

b) Research on new host-guest systems containing hydrogen for reversible storage (loading / unloading). Mainly porous supports (host) like the so called "nanoscaffolds" (based on C or BN) infiltrated with borohydrides materials (guest) (i.e. titanium borohydride) typically used for reversible hydrogen storage. These new materials must present improved charging and de-charging kinetics.

c) Studies of coupling a hydrogen generator system with a low cost fuel cell. Typically a continuous reactor for the hydrolysis of $NaBH_4$ with Co-B catalyst for providing H_2 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

Desarrollo de recubrimientos composite de carbono para aplicaciones biomédicas

Development of carbón-based composites for biomedical applications

Código/Code:	P10-TEP 06782 (Proyecto de Excelencia)
Periodo/Period:	15-03-2011 / 15-03-2014
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	96.000 €
Investigador responsable/Research head:	Juan Carlos Sánchez López
Componentes/Research group:	T. Cristina Rojas, Carlos López Cartes, David Abad, Vanda Godinho, Santiago Domínguez, Inmaculada Rosa

RESUMEN / ABSTRACT

El proyecto comprende el desarrollo de recubrimientos basados en carbono desde su síntesis a medida, caracterización, evaluación en tests de desgaste y estudios de biocompatibilidad para su uso en implantes artificiales. El control del tipo de enlace químico del carbono (sp2/sp3) y la composición química, incluyendo metales como (Ag, Ti) u otros elementos (B, N, O), permitirá modular las propiedades mecánicas y tribológicas (dureza, fricción y resistencia al desgaste) con objeto de incrementar su comportamiento final. Para ello se propone el empleo de la técnica de pulverización catódica (del inglés magnetron sputtering) para depositar estos recubrimientos avanzados sobre los materiales usados en los implantes (acero, aleaciones de Ti o polímeros) bajo diferentes condiciones de síntesis. Seguidamente, éstos composites de carbono serán evaluados de forma comparativa en ensayos de fricción y desgaste que simulen las condiciones que estos materiales se encontrarán en la aplicación final. De esta manera será posible establecer una correlación entre el comportamiento observado y las características químicas y estructurales de las capas preparadas bajo diferentes condiciones de síntesis. Finalmente, la biocompatibilidad será estudiada en ensayos de adhesión celular, citotoxicidad y actividad antibacteriana. Este completo conjunto de análisis aportará una excelente perspectiva de las posibilidades de transferencia tecnológica de estos materiales avanzados a la biomedicina.

This project pursues the development of carbon-based coatings including the tailored synthesis, characterization, evaluation in wear tests and biocompatibility study for the application in artificial implants. The control of the carbon chemical bonding (sp2/sp3) and the chemical composition, including metals as (Ag, Ti) or other elements (B, N, O) will enable to tune the mechanical and tribological properties (hardness, friction and wear resistance) with the aim of improving the final performance. To achieve this goal, the use of magnetron sputtering technique is envisaged to deposit advanced coatings under different synthesis conditions. Next, these carbon composites will be evaluated comparatively in friction and wear tests that simulate the conditions that these materials will face in the final application. In this way, it will be possible to establish the correlation between the observed behavior and chemical and structural characteristics of the prepared layers in cell adhesion tests, cytotoxicity and antibacterial activity. This complete characterization will provide an excellent overview of the possibilities of technological transfer of these advanced materials to the biomedicine.

Nanopartículas funcionalizadas para aplicaciones de hipertermia y evaluación de su ecotoxicidad Functionalized for hyperthermia applications and evaluation of ecotoxicity

Código/Code:	P09-FQM-4554 (Proyecto de Excelencia)
Periodo/Period:	03-02-2010 / 02-02-2013
Organismo Financiador/Financial source:	Junta de Andalucía (Proyecto Internacional)
Importe total/Total amount:	172.598,68 €
Investigador responsable/Research head:	Asunción Fernández Camacho
Componentes/Research group:	J. Blasco, M. Hampel, C. López, L.M. Lubián, I. Moreno, M.A. Muñoz, D. Philippon, T. Cristina Rojas, Carlos García-Negrete, Inmaculada Rosa

RESUMEN / ABSTRACT

En este Proyecto de Excelencia se parte de la experiencia previa del grupo TEP-217 en el desarrollo y caracterización de nanopartículas funcionalizadas potencialmente biocompatibles y se pretende avanzar en cuatro direcciones. a) Continuar con el desarrollo de nanopartículas basadas principalmente en Au, Ag y óxidos magnéticos con distintas funcionalizaciones y microestructura. b) Profundizar en la fisico-química de su interacción con campos electromagnéticos (en un amplio rango de frecuencias desde kHz a GHz) para producir calentamientos localizados. Actualmente se han propuesto distintos mecanismos (corrientes inducidas, histéresis, relajación de momentos magnéticos y movimiento browniano) sin que existan todavía suficientes datos para comprender e interpretar los resultados experimentales. c) Establecer una colaboración multidisciplinar con el grupo RNM-306, especialista en ensayos de ecotoxicidad, que permita mejorar el conocimiento del impacto ambiental de las nanopartículas (principalmente de oro y plata) en los ecosistemas marinos, que son el sumidero final de una buena parte de los nanomateriales producidos en la actualidad. d) Realizar estudios preliminares de la toxicidad de las nanopartículas en función del campo electromagnético aplicado. En cualquier proyecto dedicado a la nanotecnología resulta extremadamente valioso introducir estudios que nos permitan determinar el impacto toxicológico y ambiental de los nuevos materiales que se están desarrollando en la actualidad.

Un objetivo fundamental de este proyecto es la formación de personal investigador a través de la realización de una Tesis Doctoral en el Instituto de Ciencia de Materiales de Sevilla.

This Excellence project is taking profit of the previous experience of the group TEP-217 in the development and characterization of functionalized biocompatible nanoparticles and potentially trying to advance in four directions. a) Continue with the development of nanoparticle based mainly on Au, Ag and magnetic oxides with different functionalizations and microstructure. b) To deepen the physical-chemical interaction with electromagnetic fields (in a wide range of frequencies from kHz to GHz) to produce local heating. Currently, various mechanisms have been proposed (Eddy current, hysteresis, relaxation of magnetic moments and Brownian motion) without enough data yet existing to understand and interpret the experimental results. c) Establish a multidisciplinary collaboration with the group RNM-306, a specialist in ecotoxicity testing, to improve the knowledge of the environmental impact of nanoparticles (mainly gold and silver) in marine ecosystems, which are the ultimate sink for a good part of nanomaterials currently produced. d) Conduct preliminary studies of the toxicity of nanoparticles as a function of applied magnetic field. In any project dedicated to nanotechnology is extremely valuable to introduce studies to determine the toxicological and environmental impact of new materials being developed at present.

A key objective of this project is the training of research personnel through the implementation of one doctoral thesis at the Institute of Materials Science of Seville.

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 / 15-05-2015
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.

CONVENIOS Y CONTRATOS / CONTRACTS AND AGREEMENTS

Diseño, construcción y puesta en marcha de un sistema para la producción de hidrógeno por hidrólisis de borohidruro en condiciones de alimentar una pila de combustible tipo PEM

Periodo/Period:	15-12-2012 / 15-05-2013
Organismo Financiador/Financial source:	Abengoa Hidrógeno, S.A.
Investigador responsable/Research head:	Asunción Fernández Camacho
Componentes/Research group:	Gisela Arzac, Dirk Hufschmidt

Diseño, construcción y puesta en marcha de un sistema para la producción de hidrógeno por hidrólisis de borohidruro en condiciones de alimentar una pila de combustible tipo PEM

Periodo/Period:	01-07-2010 / 30-05-2013
Organismo Financiador/Financial source:	Abengoa Hidrógeno, S.A.
Investigador responsable/Research head:	Asunción Fernández Camacho
Componentes/Research group:	Gisela Arzac, Dirk Hufschmidt

Tribological behaviour and charaterisation of carbon-based coatings for lubrication under heating conditions.

Periodo/Period:	23-10-2013 / 13-12-2013
Organismo Financiador/Financial source:	Robert Bosch Gmbh
Investigador responsable/Research head:	Juan Carlos Sánchez López
Componentes/Research group:	Santiago Domínguez Meister

Patentes / Patents

CO_2 and SO_2 capture method
Inventores: Luis M. Esquivias Fedriani y Victor M. Morales López
Tipo de Patente: Patente de Invención
Fecha Solicitud: 8 noviembre 2013
Entidad/es Titular/es: Universidad de Sevilla, Universidad de Cádiz

Cooperación Internacional y Otros International Cooperation and Others

Mise au point de revêtements bactéricides sur microbilles de verre par technique plasma base pression - ABBEADS

Periodo/Period:	1-10-2012 / 30-09-2014
Código/Code:	ECV320600FD013F/1217577
Entidad Financiadora/Financial source:	Direction des Progammes Regionaux de Wallonie (Bélgica)
Investigador responsable/Research head:	Fabian Renaux
Participantes/Participants:	Juan Carlos Sánchez López

Artículos Publicados en Revistas SCI / Papers In SCI Journals

Characterisation of Co@Fe3O4 core@shell nanoparticles using advanced electron microscopy
Knappett, BR; Abdulkin, P; Ringe, E; Jefferson, DA; Lozano-Perez, S; Rojas, TC; Fernandez, A; Wheatley, AEH
Nanoscale, **5** (2013) 5765-5772
Julio, 2013 | **DOI:** 10.1039/C3NR33789H

Cobalt nanoparticles were synthesised via the thermal decomposition of Co2(CO)8 and were coated in iron oxide using Fe(CO)5. While previous work focused on the subsequent thermal alloying of these nanoparticles, this study fully elucidates their composition and core@shell structure. State-of-the-art electron microscopy and statistical data processing enabled chemical mapping of individual particles through the acquisition of energy-filtered transmission electron microscopy (EFTEM) images and detailed electron energy loss spectroscopy (EELS) analysis. Multivariate statistical analysis (MSA) has been used to greatly improve the quality of elemental mapping data from core@shell nanoparticles. Results from a combination of spatially resolved microanalysis reveal the shell as Fe3O4 and show that the core is composed of oxidatively stable metallic Co. For the first time, a region of lower atom density between the particle core and shell has been observed and identified as a trapped carbon residue attributable to the organic capping agents present in the initial Co nanoparticle synthesis.

High temperature plasticity in yttria stabilised tetragonal zirconia polycrystals (Y-TZP)
Dominguez-Rodriguez, A; Gomez-Garcia, D; Wakai, F
International Materials Reviews, **58** (2013) 399-417
August, 2013 | **DOI:** 10.1179/1743280413Y.0000000018

The literature data on the superplastic deformation of high purity yttria stabilised tetragonal zirconia polycrystals is reviewed in detail. It is shown that, based on the existence of a threshold stress, the single mechanism of grain boundary sliding (GBS) accommodated by diffusional processes can explain the superplasticity of these materials over all the ranges of temperature, stress, grain size, and surrounding atmosphere that have been studied. The origin of the threshold stress and its quantitative dependence on temperature and grain size is explained in terms of the segregation of yttrium atoms at the grain boundaries. A new model for GBS accommodated by lattice or grain-boundary diffusion is presented which can explain the transition of the stress exponent from 2 to 1.

Spectroscopic properties of electrochemically populated electronic states in nanostructured TiO2 films: anatase versus rutile
Berger, T; Anta, JA; Morales-Florez, V
Physical Chemistry Chemical Physics, **15** (2013) 13790-13795
Septiembre, 2013 | **DOI:** 10.1039/C3CP52324A

A thorough characterization of nanostructured materials under application-relevant conditions is a prerequisite for elucidating the interplay between their physicochemical nature and their functional properties in practical applications. Here, we use a spectroelectrochemical approach to study the population of electronic states in different types of nanostructured anatase and rutile TiO2 films in contact with an aqueous electrolyte. The spectroscopic properties of the two polymorphs were addressed under Fermi level control in the energy range between the fundamental absorption threshold and the onset of lattice absorption (3.3–0.1 eV). The results evidence the establishment of an equilibrium between localized Ti3+ centers absorbing in the vis/NIR and shallow (e−)(H+) traps absorbing in the MIR upon electron accumulation in anatase electrodes. The absence of the MIR-active (e−)(H+) traps on all rutile electrodes points to a crystal structure-dependent electron population in the films.

Behaviour of Au-citrate nanoparticles in seawater and accumulation in bivalves at environmentally relevant concentrations
Garcia-Negrete, C. A.; Blasco, J.; Volland, M.; Rojas, T. C.; Hampel, M.; Lapresta-Fernandez, A.; Jimenez de Haro, M. C.; Soto, M.; Fernandez, A.
Environmental Pollution, **174** (2013) 134-141
Marzo, 2013 | **DOI:** 10.1016/j.envpol.2012.11.014

The degree of aggregation and/or coalescence of Au-citrate nanoparticles (AuNPs, mean size 21.5 ± 2.9 nm), after delivery in simulated seawater, are shown to be concentration-dependent. At low concentrations no coalescence and only limited aggregation of primary particles were found. Experiments were performed in which the marine bivalve (Ruditapes philippinarum) was exposed to AuNPs or dissolved Au and subsequently, bivalve tissues were studied by Scanning and Transmission Electron Microscopy and chemical analyses. We show that the bivalve accumulates gold in both cases within either the digestive gland or gill tissues, in different concentrations (including values of predicted environmental relevance). After 28 days of exposure, electron-dense deposits (corresponding to AuNPs, as proven by X-ray microanalysis) were observed in the heterolysosomes of the digestive gland cells. Although non-measurable solubility of AuNPs in seawater was found, evidence is presented of the toxicity produced by Au3+ dissolved species (chloroauric acid solutions) and its relevance is discussed.

A new bottom-up methodology to produce silicon layers with a closed porosity nanostructure and reduced refractive index

Godinho, V; Caballero-Hernandez, J; Jamon, D; Rojas, TC; Schierholz, R; Garcia-Lopez, J; Ferrer, FJ; Fernandez, A

Nanotechnology, **24** (2013) 275604

Julio, 2013 | **DOI:** 10.1088/0957-4484/24/27/275604

A new approach is presented to produce amorphous porous silicon coatings (a-pSi) with closed porosity by magnetron sputtering of a silicon target. It is shown how the use of He as the process gas at moderated power (50–150 W RF) promotes the formation of closed nanometric pores during the growth of the silicon films. The use of oblique-angle deposition demonstrates the possibility of aligning and orientating the pores in one direction. The control of the deposition power allows the control of the pore size distribution. The films have been characterized by a variety of techniques, including scanning and transmission electron microscopy, electron energy loss spectroscopy, Rutherford back scattering and x-ray photoelectron spectroscopy, showing the incorporation of He into the films (most probably inside the closed pores) and limited surface oxidation of the silicon coating. The ellipsometry measurements show a significant decrease in the refractive index of porous coatings (n500 nm = 3.75) in comparison to dense coatings (n500 nm = 4.75). The capability of the method to prepare coatings with a tailored refractive index is therefore demonstrated. The versatility of the methodology is shown in this paper by preparing intrinsic or doped silicon and also depositing (under DC or RF discharge) a-pSi films on a variety of substrates, including flexible materials, with good chemical and mechanical stability. The fabrication of multilayers of silicon films of controlled refractive index in a simple (one-target chamber) deposition methodology is also presented.

Surface properties of anatase TiO_2 nanowire films grown from a fluoride-containing solution

Berger, T; Anta, JA; Morales-Florez, V

Chemphyschem, **14** (2013) 1676-1685

Junio, 2013 | **DOI:** 10.1002/cphc.201300024

Controlling the surface chemistry of nucleating seeds during wet-chemical synthesis allows for the preparation of morphologically well-defined nanostructures. Synthesis conditions play a key role in the surface properties, which directly affect the functional properties of the material. Therefore, it is important to establish post-synthesis treatments to facilitate the optimization of surface properties with respect to a specific application, without losing the morphological peculiarity of the nanostructure. We studied the surface properties of highly crystalline and porous anatase TiO2 nanowire (NW) electrodes, grown by chemical-bath deposition in fluoride-containing solutions, using a combined electrochemical and spectroscopic approach. As-deposited films showed low capacity for catechol adsorption and a poor photoelectrocatalytic activity for water oxidation. Mild thermal annealing at 200 °C resulted in a significant improvement of the electrode photoelectrocatalytic activity, whereas the bulk properties of the NWs (crystal structure, band-gap energy) remained unchanged. Enhancement of the functional properties of the material is discussed on the basis of adsorption capacity and electronic properties. The temperature-induced decrease of recombination centers, along with the concomitant increase of adsorption and reaction sites upon thermal annealing are called to be responsible for such improved performance.

Evidence of nanograin cluster coalescence in spark plasma sintered α-Al2O3
Morales-Rodriguez, A; Poyato, R; Gallardo-Lopez, A; Munoz, A; Dominguez-Rodriguez, A
Scripta Materialia, **69** (2013) 529-532
Octubre, 2013 | **DOI:** 10.1016/j.scriptamat.2013.06.019

The aim of this study is to elucidate the coarsening kinetics involved during densification of fine-grained pure α-alumina by spark plasma sintering. Low temperature and short dwell time sintering conditions were used to preserve the nanocrystalline structure of the starting commercial powder (about 50 nm). Notwithstanding the above, submicron grain coarsened microstructures have been developed. The microstructure evolution of alumina under different sintering conditions points to a nanograin rotation densification mechanism as being responsible for the fast grain growth observed.

In situ imaging and strain determination during fracture in a SiC/SiC ceramic matrix composite
Ramirez-Rico, J; Stolzenburg, F; Almer, JD; Routbort, JL; Singh, D; Faber, KT
Scripta Materialia, **69** (2013) 497-500
Octubre, 2013 | **DOI:** 10.1016/j.scriptamat.2013.05.032

A combined imaging and microdiffraction technique using high-energy synchrotron X-rays is described and used to reveal microstructure, damage and strain evolution around notches in SiC/SiC composites. This technique allows for monitoring the material for cracks while loading and mapping the strain distribution in fibers and matrix with a resolution of tens of microns. We show that at current resolutions this technique is capable of measuring the strain distribution near crack tips in ceramic matrix composites and observe load transfer effects.

Removal of basic yellow cationic dye by an aqueous dispersion of Moroccan stevensite
Ajbary, M; Santos, A; Morales-Florez, V; Esquivias, L
Applied Clay Science. **80-81** (46-51)
Agosto, 2013 | **DOI:** 10.1016/j.clay.2013.05.011

The aim of this study was to investigate the adsorption of basic yellow, a cationic dye, from aqueous solution by natural stevensite, with 104 m2/g of specific surface area. The kinetics and the effects of several experimental parameters such as the pH of the solution, adsorbent dose and initial dye concentration were researched using a batch adsorption technique. The results showed that an alkaline pH favoured basic yellow adsorption and the adsorption reached equilibrium in about 20 min. It was concluded that the adsorption process was governed by the electrostatic interaction. The isothermal data were fitted by means of Langmuir and Freundlich equations, and a monolayer adsorption capacity of 454.54 mg/g was calculated. Finally, a good agreement was found between the pseudo-second order model and the experimental data. A high maximum adsorption capacity was obtained (526 mg/g) and a maximum surface density of ~ 9 dye molecules/nm2 was estimated, involving a columnar arrangement of the adsorbed molecules.

Exploring the benefits of depositing hard TiN thin films by non-reactive magnetron sputtering
Martinez-Martinez, D; Lopez-Cartes, C; Fernandez, A; Sanchez-Lopez, JC
Applied Surface Science, **275** (2013) 121-126
Junio, 2013 | **DOI:** 10.1016/j.apsusc.2013.01.098

The aim of this paper is to compare the mechanical and tribological properties of TiN coatings prepared in a conventional magnetron sputtering chamber according to two different routes: the usual reactive sputtering of a Ti target in an Ar/N2 atmosphere vs. the comparatively more simple sputtering of a TiN target in a pure Ar atmosphere. Improved properties in term of hardness and wear rates were obtained for films prepared by non-reactive sputtering route, due to the lower presence of oxynitride species and larger crystalline domain size. Additionally, a significant hardness enhancement (up to 45 GPa) is obtained when a −100 V d.c. bias is applied during growth. This behaviour is explained by non-columnar growth and small grain size induced by effective ion bombarding. These results demonstrate that non-reactive sputtering of TiN target appears a simple and efficient method to prepare hard wear-resistant TiN films.

Structure and tribological properties of MoCN-Ag coatings in the temperature range of 25–700 °C
Shtansky, DV; Bondarev, AV; Kiryukhantsev-Korneev, PV; Rojas, TC; Godinho, V; Fernandez, A
Applied Surface Science, **273** (2013) 408-414
Mayo, 2013 | **DOI:** 10.1016/j.apsusc.2013.02.055

The preparation of hard coatings with low friction coefficient over a wide temperature range is still a challenge for the tribological community. The development of new nanocomposite materials consisting of different metal-ceramic phases, each of which exhibiting self-lubricating characteristics at different temperatures, may help to solve this problem. We report on the structure and tribological properties of MoCN-Ag coatings deposited by magnetron co-sputtering of Mo and C (graphite) targets and simultaneous sputtering of an Ag target either in pure nitrogen or in a gaseous mixture of Ar + N2. The structure and elemental composition of the coatings were studied by means of X-ray diffraction, scanning and transmission electron microscopy, X-ray photoelectron spectroscopy, energy dispersive spectroscopy, Raman spectroscopy, and glow discharge optical emission spectroscopy. The tribological properties of the coatings against an Al2O3 ball were investigated first at discrete temperatures of 25, 500, and 700 °C, and then during continuous heating in the temperature range of 25–700 °C. The coating structure and their respective wear tracks were also examined to elucidate their phase transformations during heat treatments. The lowest friction coefficients (<0.4) were observed in the temperature ranges of 25–100 °C and 400–700 °C and can be explained by the presence of a free amorphous carbon phase, which served as a lubricant at low temperatures, and by a positive role of silver and two phases forming at elevated temperatures, molybdenum oxide and silver molybdate, which provided lubrication above 400 °C. In the temperature range between 100 and 400 °C, the friction coefficient was relatively high. This problem is to be addressed in future works.

Dissociation of basal dislocations in 4 H - SiC single crystals deformed around the transition temperature

Castillo-Rodriguez, M; Lara, A; Munoz, A; Dominguez-Rodriguez, A

Journal of the American Ceramic Society, **96** (2013) 2921-2925

Septiembre, 2013 | **DOI:** 10.1111/jace.12434

The dislocation microstructure was studied in 4H–SiC samples plastically deformed by basal slip activation around the transition temperature (1000°C–1100°C). Dissociation of basal dislocations takes place over a wide temperature range (800°C–1300°C), but its influence on dislocation motion is different in the high- and low-temperature regimes due to the difference in mobility of partials. Consequently, this material exhibits a completely different mechanical behavior below and above its transition temperature, indicating a change in the deformation mechanism. In this work, the dislocation microstructure was studied around the transition temperature at which both mechanisms are still operative, thus providing a richer number of different configurations generated by dissociation of basal dislocations. They were observed and analyzed by means of the complementary use of weak-beam dark-field imaging and high-resolution transmission electron microscopy. Firstly, 3C band nucleation in the 4H–SiC matrix was identified and its appearance discussed from an energy standpoint. Secondly, the attractive interaction between partials in dipoles and the difference in mobility between the leading and the trailing partial have remarkable effects on the dissociation width, and explain the absence of work hardening above the transition temperature.

Mechanical properties of ZrB2- and HfB2-based ultra-high temperature ceramics fabricated by spark plasma sintering

Zapata-Solvas, E; Jayaseelan, DD; Lin, HT; Brown, P; Lee, WE

Journal of the European Ceramic Society, **33** (2013) 1373-1386

Julio, 2013 | **DOI:** 10.1016/j.jeurceramsoc.2012.12.009

Flexural strengths at room temperature, at 1400 °C in air and at room temperature after 1 h oxidation at 1400 °C were determined for ZrB2- and HfB2-based ultra-high temperature ceramics (UHTCs). Defects caused by electrical discharge machining (EDM) lowered measured strengths significantly and were used to calculate fracture toughness via a fracture mechanics approach. ZrB2 with 20 vol.% SiC had room temperature strength of 700 ± 90 MPa, fracture toughness of 6.4 ± 0.6 MPa, Vickers hardness at 9.8 N load of 21.1 ± 0.6 GPa, 1400 °C strength of 400 ± 30 MPa and room temperature strength after 1 h oxidation at 1400 °C of 678 ± 15 MPa with an oxide layer thickness of 45 ± 5 µm. HfB2 with 20 vol.% SiC showed room temperature strength of 620 ± 50 MPa, fracture toughness of 5.0 ± 0.4 MPa, Vickers hardness at 9.8 N load of 27.0 ± 0.6 GPa, 1400 °C strength of 590 ± 150 MPa and room temperature strength after 1 h oxidation at 1400 °C of 660 ± 25 MPa with an oxide layer thickness of 12 ± 1 µm. 2 wt.% La2O3 addition to UHTCs slightly reduced mechanical performance while increasing tolerance to property degradation after oxidation and effectively aided internal stress relaxation during spark plasma sintering (SPS) cooling, as quantified by X-ray diffraction (XRD). Slow crack growth was suggested as the failure mechanism at high temperatures as a consequence of sharp cracks formation during oxidation.

Preliminary investigation of flash sintering of SiC
Zapata-Solvas, E; Bonilla, S; Wilshaw, PR; Todd, RI
Journal of the European Ceramic Society, 33 (2013) 2811-2816
Noviembre, 2013 | **DOI:** 10.1016/j.jeurceramsoc.2013.04.023

The feasibility of flash sintering a covalent ceramic, SiC, has been investigated for the first time. Flash sintering involves the application of an electrical potential difference across a powder compact during heating, which leads to sintering at low furnace temperatures in a few seconds and has only been demonstrated with ionic ceramics previously. Near-theoretical density was achieved using Al_2O_3 + Y_2O_3 sintering aids at a furnace temperature of only 1170 degrees C and in a time of 150 s. Specimen temperatures were significantly higher than the furnace temperature owing to Joule heating and consequently heat loss limited densification in the near surface region. It was not possible to reach high densities using "ABC" sintering aids (aluminium-boron-carbon) or pure SiC. The mechanisms involved and potential commercial advantages are briefly discussed.

Thermal properties of La_2O_3-doped ZrB2- and HfB_2-based ultra-high temperature ceramics
E. Zapata-Solvas, D.D. Jayaseelan, P.M. Brown, W.E. Lee
Journal of the European Ceramic Society, **33** (2013) 3467-3472
Diciembre, 2013 | **DOI:** 10.1016/j.jeurceramsoc.2013.06.009

Thermal properties of La2O3-doped ZrB2- and HfB2-based ultra high temperature ceramics (UHTCs) have been measured at temperatures from room temperature to 2000 °C and compared with SiC-doped ZrB2- and HfB2-based UHTCs and monolithic ZrB2 and HfB2. Thermal conductivities of La2O3-doped UHTCs remain constant around 55–60 W/mK from 1500 °C to 1900 °C while SiC-doped UHTCs showed a trend to decreasing values over this range.

Structure of supercritically dried calcium silicate hydrates (C–S–H) and structural changes induced by weathering
Morales-Florez, V; de la Rosa-Fox, N
Journal of Materials Science, **48** (2013) 5022-5028
Julio, 2013 | **DOI:** 10.1007/s10853-013-7289-6

The nanostructure of supercritically dried calcium silicate hydrates was researched. This particular drying procedure was used to avoid nanostructure modifications due to conventional drying processes. Thus, in this study, the as-precipitated cementitious C–S–H structure was obtained for the first time. A specific surface area 20 % larger than conventionally dried C–S–H was measured. Given the importance of this nanostructured phase for the properties of hydrated cements, especially when in contact with CO2-rich environments, the supercritically dried C–S–H was weathered for 2 weeks. The structural effects of this weathering process on the C–S–H were researched and calcium carbonate microcrystal precipitation or the presence of silica by-product are reported. Calcite and aragonite polymorphs were observed, as well as nanoporous silica forming globular arrangements. In addition, 2 weeks of weathering was not enough to carbonate the entire C–S–H sample.

Strong quantum confinement effects in SnS nanocrystals produced by ultrasound-assisted method
Azizian-Kalandaragh, Y; Khodayari, A; Zeng, ZP; Garoufalis, CS; Baskoutas, S; Gontard, LC
Journal of Nanoparticle Research, **15** (2013) 1388
Enero, 2013 | **DOI:** 10.1007/s11051-012-1388-1

Nanocrystalline SnS powder has been prepared using tin chloride (SnCl2) as a tin ion source and sodium sulfide (Na2S) as a sulfur ion source with the help of ultrasound irradiation at room temperature. The as-synthesized SnS nanoparticles were quantitatively analyzed and characterized in terms of their morphological, structural, and optical properties. The detailed structural and optical properties confirmed the orthorhombic SnS structure and a strongly blue shifted direct band gap (1.74 eV), for synthesized nanoparticles. The measured band gap energy of SnS nanoparticles is in a fairly good agreement with the results of theoretical calculations of exciton energy based on the potential morphing method in the Hartree–Fock approximation.

Synthesis and tribological properties of WSex films prepared by magnetron sputtering
Dominguez-Meister, S; Justo, A; Sanchez-Lopez, JC
Materials Chemistry and Physics, **142** (2013) 186-194
Octubre, 2013 | **DOI:** 10.1016/j.matchemphys.2013.07.004

WSex films with variable Se/W ratio were deposited by non-reactive r.f. magnetron sputtering from WSe2 target changing the applied d.c. pulsed bias conditions and substrate temperature. The structural and chemical properties were measured by cross-sectional scanning electron microscopy (X-SEM), energy dispersive analysis (EDX), X-ray diffraction (XRD), Raman and X-ray photoelectron spectroscopy (XPS). The tribological properties were measured in ambient air (RH = 30–40%) and dry nitrogen by means of a reciprocating ball-on-disk tribometer. A clear correlation was found between the Se/W ratio and the measured friction coefficient displaying values below 0.1 (in ambient air) and 0.03 (in dry N2) for ratios Se/W ≥ 0.6 as determined by electron probe microanalysis (EPMA). The results demonstrated that notable tribological results could be obtained even in ambient air (friction ≤ 0.07 and wear rate ≈10−7 mm3 Nm−1) by controlling the film microstructure and chemical composition. By incorporating carbon, wear and chemical resistance can be gained by formation of non-stoichiometric carbides and/or alloying into the defective WSex hexagonal structure. The existence of a WSe2 rich interfacial layer (either on the ball scar or embedded in the film track) was evidenced by Raman in low friction conditions. The improvement in tribological performance is therefore obtained by means of layered WSex, the formation of gradient composition from metallic W (hard) to WSe2 (lubricant) and carbon incorporation.

Segregation to the grain boundaries in YSZ bicrystals: A Molecular Dynamics study
Gonzalez-Romero, RL; Melendez, JJ; Gomez-Garcia, D; Cumbrera, FL; Dominguez-Rodriguez, A
Solid State Ionics, **237** (2013) 8-15
Abril, 2013 | **DOI:** 10.1016/j.ssi.2013.02.002

A Molecular Dynamics study about the segregation of yttrium at 1500 K to a Σ5 grain boundary in 8 mol% YSZ has been performed. Segregation has been induced by explicitly taking into account the excess energy associated to the elastic misfit effect for yttrium cations located nearby the grain boundary planes. After an initial transient, a steady regime is reached, in which the number of yttrium cations does not increase with time. Accumulation of yttrium cations is accompanied by that of zirconium ones and oxygen vacancies at some distance of the grain boundary planes. The changes in the radial distribution functions for different ionic pairs are discussed, as also the effect of segregation on oxygen diffusion along the grain boundaries and in volume. Finally, the possibility that segregated yttrium located at available free sites at the grain boundaries is pointed out.

Tribological properties of surface-modified Pd nanoparticles for electrical contacts
Abad, MD; Sanchez-Lopez, JC
Wear, **297** (2013) 943-951
Enero, 2013 | **DOI:** 10.1016/j.wear.2012.11.009

A fully comprehensive study of the tribological behavior of palladium nanoparticles (Pd NPs) capped by tetrabutylammonium chains using a ball-on-disk tribometer under different conditions of applied load, concentration, tribometer motion, linear speed and nature of the counterface is revised. A low concentration of NPs (2 wt%) in tetrabutylammonium acetate was found sufficient to improve the tribological properties due to the formation of a protective transfer film (TF) comprised of metallic Pd. The increase of the applied load (up to 20 N, 1.82 GPa of contact pressure) confirmed the excellent extreme-pressure behavior avoiding the counterfaces from severe wear. After a running-in period whose duration depends on the operating conditions, the TF build-up allows to maintain a low contact electrical resistance through the contact (<0.1 kΩ) during the entire test. When the Pd NPs are used with ceramic counterfaces, the nanoparticles increase the load-bearing capabilities and performance of the base without forming TF, likely by mixed or boundary lubrication and healing effects. Finally, the Pd NPs are demonstrated to be useful as a thin solid lubricant film in reciprocating motion yielding a comparable tribological behavior. Hence, the presented surface Pd NPs can be very helpful to extend life of sliding components due to their high strength resistance providing a gateway to electrical conduction as well.

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

COMUNICACIONES EN CONGRESOS Y REUNIONES / COMMUNICATIONS IN CONGRESS AND MEETINGS

7th International Symposium Hydrogen & Energy

21 – 25 enero [Suiza]

Catalytic hydrolysis of NaBH4 in a continuous reactor for long time stable and versatile generation of hydrogen
D. Hufschmidt; G.M. Arzac; A. Fernández; B. Sarmiento; M.A. Jiménez
Comunicación oral

PRIMO 17 "Pollutant Responses in Marine Organisms"

5 – 8 mayo [Faro, Portugal]

Evaluation of lethal and sublethal effects of exposure to gold nanoparticles and dissolved gold on aquatic organisms
M. Hampel; J. Otero; C.García Negrete; A. Fernández-Camacho; J. Blasco
Poster

Research Potential capitalization exercise: Thematic workshop on capacity building – Stairway to excellence

25 junio [Bélgica]

Case studies of REGPOT synergies with Structural Funds: AL-NANOFUNC
A. Fernández
Conferencia Invitada

International Conference on Ion Beam Analysis 2013

23 junio [Seattle, Washington, Estados Unidos de América]

Simultaneous quantification of light elements in thin films deposited on Si substrates using proton-RBS

F.J. Ferrer, M. Alcaire, J. Caballero, F.J. García García, J. Gil Rostra, A. Terriza, V. Godinho, J. García López, A. Barranco, A. Fernández Camacho

Poster

13th International Conference of the European Ceramic Society – ECERS

23-27 Junio [Limoges, Francia]

Microstructure and electrical conductivity of Spark Plasma Sintered SWNT/3YTZP nanocomposites

R. Poyato, A. García-Valenzuela, R.L. González-Romero, A. Gallardo-López, A. Muñoz, A. Domínguez-Rodríguez

Póster

3Y-TZP with 0.5 and 1 vol.% SWNTs: sintering, microstructure and room-temperature mechanical properties

A. Gallardo-López, A. Muñoz, A. Morales-Rodríguez, R. Poyato, A. Domínguez-Rodríguez

Póster

AI-NanoFunc Workshops: "II Nanomaterials for sustainable energy and protection of the enviroment and III Advanced microstructural characterization of nanomaterials"

1 – 2 julio [Sevilla, España]

Characterisation of Co@Fe_3O_4 core@shell nanoparticles using advanced electron microscopy

Benjamin R. Knappett; Pavel Abdulkin; Emilie Ringe; David A. Jefferson; Sergio Lozano-Perez; T. Cristina Rojas; Asunción Fernández; Andrew E. H. Wheatley

Comunicación oral

Main contributions of the TEM techniques to determine the nanostructure and chemical composition of the complex catalytic and magnetic systems: Co-B and Co-Ru-B

G.M. Arzac; T.C. Rojas; L. Cervera; A. Fernández

Comunicación oral

Characterization of amorphous porous Silicon coatings by Transmission Electron Microscopy techniques

Roland Schierholz; Jaime Caballero; Vanda Godinho; Asunción Fernández

Comunicación oral

High-resolution quantitative characterization of nanoparticles

Sergio Lozano-Perez; Kate MacArthura; Haibo Ea; Benjamin R. Knappett; Pavel Abdulkin; Andrew E. H. Wheatley; Cristina Rojas-Ruiz; Asuncion Fernandez
Comunicación oral

Investigation of the catalyzed hydrolysis of Ammonia Borane in a continuous flow reactor for the hydrogen production at medium scale
M. Paladini; G.M. Arzac; D. Hufschmidt; G. Adame; M.A. Jiménez; A. Fernández
Poster

Mean inner potential and skeletical density of zeolite MCM- 41 using TEM
Lionel C. Gontard; Rafal E. Dunin-Borkowski; T. Kasama
Poster

Study of deposition parameters on the microstructure of magnetron sputtered amorphous silicon coatings with closed porosity
J. Caballero; R. Schierholz; V. Godinho; M. Duchamp; R. Dunin-Borkowski; A. Fernández
Poster

Al-NanoFunc Workshops 2013
1 – 2 julio [Sevilla, España]

$BiPO_4$ nanostars for luminescent applications
Joaquín Criado, Ana Isabel Becerro, Lionel C. Gontard, Asunción Fernández, Manuel Ocaña
Poster

Microscopy Conference 2013
25 – 30 agosto [Regensburg, Alemania]

Understanding the behaviour ot a CoB catalyst tor sodium borohydride hydrolysis: An electron microscopy study
A. Femández; G.M.Arzac; E. Deprez; T.C. Rojas; M.C. Jiménez
Conferencia Invitada

Characterization of amorphous and porous Silicon coatings by (S)TEM and EELS
R. Schierholz; J. Caballero-Fernández; V. Godinho; A. Fernández
Poster

International Conference on Computational Modelling of Nanostructured Materials
3 – 6 septiembre [Frankfurt, Alemania]

3rd ECNF & AL-NANOFUNC Cconference. Highlighted research topics
Vanda Godinho, Asunción Femández
Comunicación oral

European Conference on Advanced Materials and Processes 2013 - EUROMAT

8 – 13 septiembre [Sevilla, España]

Simulation and growth of porous silicon coatings by magnetron sputtering
V. Godinho; J. Caballero-Hernández; R. Schierholz; P. Moskovkin; R. Álvarez; B. Bera; J. Demarche; A. Palmero; S. Lucas; A. Fernández
Comunicación oral

Influence of deposition parameters on the closed porous structure of magnetron sputtered amorphous silicon coatings
J. Caballero-Hernández; R. Schierholz; V. Godinho; M. Duchamp; R. Dunin-Borkowski; A. Fernández
Poster

Effect of single wall carbon nanotubes on the electrical properties of 3YTZP based ceramic nanocomposites
R. Poyato, A. García-Valenzuela, J. Macías-Delgado, R.L. González-Romero, A. Gallardo-López, A. Muñoz, A. Domínguez-Rodríguez
Póster

Spark Plasma Sintering and microstructure of low content Single-Walled Carbon Nanotube reinforced alumina
A. Gallardo-López, A. Morales-Rodríguez, R. Poyato, A. Muñoz, A. Domínguez-Rodríguez
Póster

Single wall carbon nanotubes in a 3YTZP matrix. High temperature mechanical behaviour
M. Castillo-Rodríguez, A. Muñoz, A. Morales-Rodríguez, A. Gallardo-López, R. Poyato, A. Domínguez-Rodríguez
Póster

Tribological behaviour of single-walled carbon nanotubes reinforced alumina
F. Gutiérrez-Mora, R. Poyato, A. Morales-Rodríguez, A. Gallardo-López, A. Muñoz, A. Domínguez-Rodríguez
Póster

Supported Co catalysts prepared by magnetron sputtering for hydrogen production through sodium borohydride or ammonia borane hydrolysis
M. Paladini; G.M. Arzac; V. Godinho; A. Fernández
Poster

E-MRS 2013 FALL MEETING

16 – 20 septiembre [Varsovia, Polonia]

A multi-technique microstructural study of additives/catalysts used In hydrogen storage processes based on borohydride materials
A. Femández; G.M.Arzac; E. Deprez; T.C. Rojas; M.C. Jiménez
Conferencia Invitada

Microscopy at the Frontiers of Science 2013
17 – 20 septiembre [Tarragona, España]

Location study of gold NPS in bivalve tissues by STEM mode in a SEM-FEG microscope
C.A. García-Negrete; M.C. Jiménez de Haro; J. Blasco; A.Fernández
Comunicación oral

CONGRESOS Y REUNIONES NACIONALES / NATIONAL CONGRESSES AND MEETINGS

COMUNICACIONES EN CONGRESOS Y REUNIONES / COMMUNICATIONS IN CONGRESS AND MEETINGS

Reunión de la Sociedad Española de Catálisis SECAT 2013
26 – 28 junio [Sevilla, España]

Deposición de capas de Co en membranas de PtFe por pulverización catódica para la producción y separación de hidrógeno mediante la hidrólisis catalítica de Borohidruro sódico
D. Hufschmidt; M. Paladini; G.M. Arzac; V. Godinho; A. Fernández
Poster

Estudio de la reacción de hidrólisis catalizada de NH3BH3 en reactor continuo para la producción de hidrógeno a mediana escala
M. Paladini; G.M. Arzac; D. Hufschmidt; G. Adame; M.A. Jiménez; A. Fernández
Poster

9° Congreso Ibérico y 6° Iberoamericano de Contaminación y Toxicología Ambiental
1 – 4 julio [Valencia, España]

Uptake. effect and elimination of engineered gold nanoparticles in the marine bivalve clam Rudifapes philippinarum
M. Volland; C. Trombini; C. Garcia-Negrete; A. Fernandez; T. Gomes; M. J. Bebianno; M. Hampel; J. Blasco
Poster

Formacion / Training

Tesis Doctorales/ Doctor Degree Thesis

Título:	**Estudio de la reacción de hidrólisis del borohidruro sódico como medio de producción de hidrógeno para aplicaciones portátiles**
Autor:	Gisela Mariana Arzac Di Tomaso
Directores:	M. Asunción Fernández Camacho y Cristina T. Rojas Ruiz
Calificación:	Sobresaliente “Cum Laude”
Centro:	Universidad de Sevilla

Formación de Graduados / Master Degree Thesis

Título:	**Estudio de percolación eléctrica en compuestos de 3YTZP reforzada con nanotubos de carbono monocapa**
Autor:	Aurelio García Valenzuela
Directores:	Rosalía Poyato Galán y Ángela Gallardo López
Grado:	Trabajo Fin de Master
Año Académico:	2012-2013 (11 Julio 2013)

Docencia / Teaching

Máster en Ciencias y Tecnologías Químicas

El hidrógeno como vector de transporte y almacenamiento de la energía sostenible del futuro: un reto científico

Dra. M. Asunción Fernández Camacho

Lugar: Universidad de Cádiz

Máster Oficial y Doctorado Interuniversitario en “Biotecnología Avanzada”

Módulo de Nanotecnología

Dra. Dr. José Jesús Benítez Jiménez

Lugar: Universidad de Málaga

Organizado por la Universidad de Málaga y la Universidad Internacional de Andalucía

ESTANCIAS Y VISITAS DE PERSONAL DEL ICMS EN OTROS CENTROS

PERSONNEL OF THE ICMS IN OTHER LABORATORIES

FELMI-Graz, Austrian Centre for Electron Microscopy and Nanoanalysis

Graz, Austria	**Carlos García Negrete**	1 semana

Ernst Ruska-Centre for Microscopy and Spectroscopy with Electrons

Jülich, Alemania	**Lionel Cervera Gontard**	2 semanas
	Roland Schierholz	2 semanas

Japan Synchrotron Radiation Research Institute

Sayo, Japón	**Roland Schierholz**	2 semanas

Ernst Ruska-Centre for Microscopy and Spectroscopy with Electrons, Institute for Microstructure Research Centre Jülich

Jülich, Alemania	**Lionel Cervera Gontard**	2 semanas

EMPA. Eidgenössische Materialprüfungs- und Forschungs-anstalt. Section Hydrogen & Energy

Zurich, Suiza	**Asunción Fernández Camacho**	3 meses

FUNDP. Universidad de NAMUR

Namur, Bélgica	**Vanda Fortio Godinho**	5 meses

Fitzwilliam College. Department of Chemistry University of Cambridge

Cambridge, Inglaterra	**Carlos García Negrete**	7 meses

ESTANCIAS Y VISITAS DE INVESTIGADORES EN EL ICMS

PERSONNEL OF THE OTHER LABORATORIES IN THE ICMS

Universidad de Cádiz

Cádiz, España	**José Calvino**	1 día

Department of Materials. University of Oxford

Oxford, Reino Unido	**Karen Kruska**	1 semana
	Sergio Lozano	1 semana

Ernst Ruska-Centre for Microscopy and Spectroscopy with Electrons, Institute for Microstructure Research Centre Jülich

Jülich, Alemania	**Martial Duchamp**	1 semana

University of Cambridge Cambridge, Reino Unido	**Benjamin Knappett**	1 semana
	Adam Fraser	1 semana
	Zineb Saghi	3 semanas
Universidad de Minho Guimaraes, Portugal	**Sebastián Calderón**	1 mes
Instituto Federal do Amazonas Brasil	**Yurimiler Leyet Ruiz**	1 mes
Universidad Hassan II Marruecos	**Risite Heriarivelo**	2 meses

Equipamiento Científico Available Equipment

Spin coater
UV/Ozonone cleaner
Horno a vacío de baja temperatura
Cromatógrafo
Pila de combustible (100W)
Medidor y sonda de temperatura por fluorescencia
Cabeza magnetrón de 2”
Bomba FLODOS
Electrónica y controladores de flujo másico para gases

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.

Cámaras de deposición

Tres cámaras de deposición de recubrimientos y películas delgadas por la técnica de pulverización catódica (magnetrón sputtering). Con una dotación de 7 cabezas magnetrón, 2 fuentes DC, 2 fuentes RF y 1 fuente pulsada, portamuestras girables y calentables.

Laboratorio de síntesis de NPs y catalizadores por vía química
Reactores y material de vidrio convencional
Rotavapor, sistemas de filtrado
Cámara seca MBRAUN
Reactores de producción de hidrógeno

Laboratorio AFM/STM
Microscopio AFM aire/liquido modelo Explorer marca Topometrix.
Microscopio AFM aire/atmósfera controlada modelo Cervantes con unidad de control Dulcinea marca Nanotec.
Microscopio STM aire/atmósfera controlada con unidad de control Dulcinea marca Nanotec.
Microscopio STM aire/líquido modelo Discoverer con controlador TMX2000 marca Topometrix.
Balanza Langmuir-Blodgett modelo Minitrough termostatizada con dipper controlado por software marca KSV.

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

PROYECTOS DE INVESTIGACIÓN / RESEARCH PROJECTS

Unión Europea
Fondo Europeo de Desarrollo Regional
"Una manera de hacer Europa"
MINISTERIO DE ECONOMÍA Y COMPETITIVIDAD

Propiedades termomagnéticas de materiales y optimización de su eficiencia energética Thermomagnetic properties of materials and optimization of energy efficiency

Código/Code:	MAT2010-20537
Periodo/Period:	01-01-2011 / 30-06-2014
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	121.000 €
Investigador responsable/Research head:	Alejandro Conde Amiano
Componentes/Research group:	Clara F. Conde Amiano, Josefa María Borrego Moro, Victorino Franco García, Javier S. Blázquez Gámez, Rafael Caballero Flores, Jhon J. Ipus Bados

RESUMEN / ABSTRACT

Se propone un estudio de propiedades termomagnéticas de materiales magnéticos blandos, preparados por técnicas de solidificación rápida y aleado mecánico. Se trata de profundizar en la relación composición-microestructura-propiedades como vía para la optimización de materiales en dos direcciones: magnéticos blandos para aplicaciones a alta temperatura y materiales para refrigeración magnética en el entorno de la temperatura ambiente. En el primer caso se trata de aumentar el límite térmico de comportamiento blando del material y de disminuir el coeficiente de temperatura de sus propiedades. En el segundo caso se trata de acercar la temperatura de transición magnética del material a la temperatura ambiente y de maximizar su respuesta magnetocalórica. Se abordará también la modelización de diferentes procesos implicados en la formación de aleaciones (aleado mecánico, cinética de nanocristalización), de las interacciones entre partículas y sus efectos en el comportamiento termomagnético del material, de la dependencia de la respuesta magnetocalórica con el campo magnético aplicado, etc.

A study of the thermomagnetic properties of soft magnetic materials obtained by rapid quenching and mechanical alloying techniques is proposed. The aim of the project is to improve the knowledge of the composition-microstructure-properties relationship as a way to optimize the material properties in two lines: soft magnetic materials for high temperature applications and materials for magnetic refrigeration near room temperature. In the first case, the aim is to enhance the thermal limit for the soft magnetic behavior of the materials and to decrease the temperature coefficient of their properties. In the second case, the aim is to bring the magnetic transition temperature of the material close to room temperature and to maximize the magnetocaloric response. It will be also contemplated modeling of different processes involved in the alloy formation (mechanical alloying, nanocrystallization kinetics), of the interactions between particles and their effects on the thermomagnetic behavior of the material, of the dependency of the magnetocaloric response with the applied magnetic field, etc.

Materiales magnéticos blandos; su optimización para un uso eficiente de la energía Soft magnetic materials; their optimization for an efficient use of energy

Código/Code:	P10-FQM-6462 (Proyecto de Excelencia)
Periodo/Period:	15-03-2011 / 31-12-2014
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	153.190,75 €
Investigador responsable/Research head:	Alejandro Conde Amiano
Componentes/Research group:	Clara F. Conde Amiano, Josefa María Borrego Moro, Victorino Franco García, Javier S. Blázquez Gámez, Jhon J. Ipus Bados

RESUMEN / ABSTRACT

El proyecto plantea abordar problemas relativos al comportamiento termomagnético de materiales amorfos y nanocristalinos magnéticamente blandos, preparados por técnicas de solidificación rápida y de aleado mecánico. En las aleaciones nanocristalinas obtenidas por desvitrificación parcial de un amorfo precursor se trata de profundizar en los mecanismos de formación de los nanocristales, analizando los efectos composicionales en la cinética del proceso y en la microestructura resultante.

The aim is to optimize in terms of composition and microstructure the thermomagnetic behaviour of materials in two directions: a) extension of the high temperature limit for soft magnetic behaviour, and b) optimization of the magnetocaloric properties in order to their use for magnetic refrigeration at room temperature. Also, in the context of the relation microstructure-properties, we will try to modelize the involved processes in the formation of the alloys, the particle interactions and thir effects on the thermomagnetic behaviour of materials, the field dependence of the magnetocaloric effects, etc. following previous results of the group.

Otros Proyectos / Other Projects

Magnetocaloric effect in amorphous and multiphase materials: experiments and models

Código/Code: N00014-11-1-0311
Periodo/Period: 1-05-2011 / 31-01-2013
Oganismo Financiador/Financial source: OTROS PROGRAMAS, ORGANISMOS PÚBLICOS (EXTERNOS) OFFICE OF NAVAL RESEARCH (USA)
Importe total/Total amount: 99.432 $ USA
Investigador responsable/Research head: Victorino Franco García
Componentes/Research group: Clara F. Conde Amiano, Alejandro Conde Amiano, Javier S. Blázquez Gámez, B. Ingale

Convenios y Contratos / Contracts and Agreements

Characterization of the physical properties of materials

Periodo/Period: 20-07-2013 / 19-07-2014
Organismo Financiador/Financial source: ThyssenKrupp Steel Europe AG
Investigador responsable/Research head: Victorino Franco García

Characterization of magnetocaloric materials

Periodo/Period: 14-05-2013 / 13-05-2014
Organismo Financiador/Financial source: ERASTEEL
Investigador responsable/Research head: Victorino Franco García

Cooperación Internacional y Otros International Cooperation and Others

Proyecto NANOKOP

Periodo/Period: 1-01-2012 / 31-12-2013
Entidad Financiadora/Financial source: UE. Intercambio de estancias entre el Institute of Experimental Physics of Slovak Academy of Sciences, Kosice, Eslovaquia, y la Universidad de Sevilla
Investigador responsable/Research head: Victorino Franco García

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

Mechanical and magnetocaloric properties of Gd-based amorphous microwires fabricated by melt-extraction
Qin, F. X.; Bingham, N. S.; Wang, H.; Peng, H. X.; Sun, J. F.; Franco, V.; Yu, S. C.; Srikanth, H.; Phan, M. H.
Acta Materialia, **61** (2013) 1284-1293
DOI: 10.1016/j.actamat.2012.11.006

We report a systematic study of a new class of melt-extracted Gd53Al24CO20Zr3 amorphous microwires in terms of fabrication, structural characterization and evaluation of mechanical and magnetic properties. The tensile properties of the wires are characterized by a precision video gauge method and analyzed using the Weibull and lognormal methods. The three-parameter Weibull model and lognormal model based on the median rank value show consistent results and prove to be superior to the two-parameter Weibull model for the studied microwire with a smaller variation. The statistical mean tensile strength and fracture strain are calculated to be similar to 1200 MPa and similar to 2.0%, respectively, which are comparable with those of other metallic glasses. Remarkably, the microwires exhibit a large and reversible magnetocaloric effect (MCE), with the isothermal magnetic entropy change (-Delta S-m) and refrigerant capacity (RC) reaching the large values of 5.32 J kg(-1) K-1 and 467 J kg(-1) for a field change of 3 T. Albeit with a low Curie temperature, these values are superior to those reported for pure Gd and other Gd-based bulk or ribbon-shaped glasses. The mean field model-based and Langevin function analyses reveal that the second-order magnetic transition behavior of the studied wire originates from the local anisotropy associated with the fine size of the wire and derogation and fluctuation of the exchange integral. These results demonstrate the adaptability and overall excellence of the newly developed Gd-based microwires, making them multifunctional elements for MCE-based cooling applications, especially for liquid nitrogen liquefaction.

Influence of magnetic interactions between phases on the magnetocaloric effect of composites
Romero-Muniz, C; Franco, V; Conde, A
Applied Physics Letters, **102** (2013) 082402
DOI: 10.1063/1.4793663

Magnetocaloric materials with coexisting magnetic phases appear either due to the phase coexistence in first order phase transitions, or due to the development of composites, which are known to enhance the refrigerant capacity and produce table-like magnetocaloric effect. However, interactions between phases are rarely considered. We have modeled the influence of interactions on the magnetocaloric effect of a biphasic composite by implementing a mean field model. Interactions shift the peak magnetic entropy change to higher temperatures than those of the pure phases and enhance the table-like character of the curves. Although there is no qualitative change of the magnetocaloric response of the composites due to interactions, the optimal fraction of phases which produces the largest enhancement of the refrigerant capacity is shifted to compositions richer in the low Curie temperature phase. This shift can be used to estimate the magnitude of the interactions in composites measured experimentally.

Role of starting phase of boron on the mechanical alloying of FeNbB composition
Ipus, JJ; Blazquez, JS; Franco, V; Lozano-Perez, S; Conde, A
Journal of Alloys and Compounds, **553** (2013) 119-124
DOI: 10.1016/j.jallcom.2012.11.099

Mechanical alloyed Fe75Nb10B15 systems, prepared with crystalline or commercial amorphous boron and a similar composition with the same Fe/Nb ratio but no boron, have been studied as a function of milling time in the aim of enhancing the homogenization of boron and determining its role on the mechanical alloying process. Neither boron addition nor boron phase used affect the developed microstructure at very early stages. After 4 h milling at 350 rpm, the formation of an amorphous phase was observed for boron containing compositions while for the boron-free alloy a supersaturated solid solution was found in the final microstructure. The alloy prepared using commercial amorphous boron showed a larger fraction of amorphous phase than that prepared using crystalline boron for the same milling time, suggesting that amorphous boron accelerates the formation of the amorphous phase during mechanical alloying.

Active transient cooling by magnetocaloric materials
Law, JY; Franco, V; Keblinski, P; Ramanujan, RV
Applied Thermal Engineering, **52** (2013) 17-23
DOI: 10.1016/j.applthermaleng.2012.11.001

The magnetocaloric effect (MCE) has been intensively studied for novel energy efficient thermal management systems. The present study demonstrates a proof-of-concept magnetic cooling setup for active cooling of the thermal spikes of a heated resistor. Using Gd as the MCE material, the device was capable of actively cooling thermal spikes within one cycle since the dynamics of magnetic phase transition in Gd (a second-order magnetic phase transition material) are favorable to effect a fast MCE response. Enhanced cooling rate of the heated resistor of up to similar to 85% for active cooling by MCE compared to passive cooling was achieved. The cooling curve of the resistor was found to follow an exponential decrease. Our results show that magnetic cooling systems can be an efficient solution to cool thermal spikes in active transient cooling systems.

The use of amorphous boron powder enhances mechanical alloying in soft magnetic FeNbB alloy: A magnetic study
Ipus, JJ; Blazquez, JS; Franco, V; Conde, A
*Journal of Applied Physics, **11**3* (2013) 17A330
DOI: 10.1063/1.4798794

Saturation magnetization and magnetic anisotropy have been studied during mechanical alloying of Fe75Nb10B15 alloys prepared using crystalline and commercial amorphous boron.

The evolution of saturation magnetization indicates a more efficient dissolution of boron into the matrix using amorphous boron, particularly for short milling times. The magnetization of the crystalline phase increases as boron is incorporated into this phase. Two milling time regimes can be used to describe the evolution of magnetic anisotropy: a first regime governed by microstrains and a second one mainly governed by crystal size and amorphous fraction.

Magnetic properties and magnetocaloric effects in GdCo9Si2 compound with multiple magnetic phase transitions
Zheng, Z. G.; Zhong, X. C.; Zhang, J. L.; Liu, Z. W.; Franco, V.; Zeng, D. C.
Journal of Applied Physics, ***11**3* (2013) 17A938
DOI: 10.1063/1.4799973

The structure and magnetic properties of polycrystalline GdCo9Si2 compound have been investigated. It has a BaCd11 structure and undergoes two magnetic phase transitions: an antiferromagnetic to ferrimagnetic transition occurring at similar to 93 K, and a ferrimagnetic to paramagnetic transition at 420 K, which results in a positive and a negative magnetic entropy change, respectively. The two peak values of magnetic entropy change are -0.6 and 1.1 J.kg(-1).K-1 for Delta H = 5 T. Furthermore, there exists a metal-semiconductor transition temperature (T-P), below which the resistance increases with increasing temperature, while the semiconductor characteristic is observed above T-P. The magnetic domain structures are characterized by stripe and grid structures 1 mu m wide. Although the MCE is small for applications, its study is useful to clearly understand the nature of multiple magnetic phase transitions in the GdCo9Si2 compound.

Low hysteresis and large room temperature magnetocaloric effect of Gd5Si2.05-xGe1.95-xNi2x (2x=0.08, 0.1) alloys
Zhong, X. C.; Min, J. X.; Liu, Z. W.; Zheng, Z. G.; Zeng, D. C.; Franco, V.; Ramanujan, R. V.
Journal of Applied Physics, ***11**3* (2013) 17A916
DOI: 10.1063/1.4795434

Gd5Si2.05-xGe1.95-xNi2x (2x = 0.08, 0.1) alloys were prepared by arc melting followed by annealing at 1273K for 96 h. Mixed monoclinic Gd5Si2Ge2-type phase, orthorhombic Gd5Si4-type phase, and a small amount of Gd5Si3-type phase were obtained in these alloys. Gd5Si2.01Ge1.91Ni0.08 alloy undergoes a second-order transition (T-C) around 300 K, whereas Gd5Si2Ge1.9Ni0.1 alloy exhibits two transitions including a first-order transition (T-C(II)) at similar to 295K and second-order transition (T-C(I)) at similar to 301K. Ni substitution can effectively reduce the thermal hysteresis and magnetic hysteresis while maintaining large magnetic entropy change. The maximum magnetic entropy changes (vertical bar Delta S-M(max)vertical bar) of Gd5Si2.05-xGe1.95-xNi2x alloys with 2x = 0.08 and 0.1 are 4.4 and 5.0 J kg(-1) K-1, respectively, for 0-2 T, and are 8.0 and 9.1 J kg(-1) K-1, respectively, for 0-5 T. Low hysteresis performance and relatively large magnetic entropy change make these alloys favorable for magnetic refrigeration applications.

Tuning the Curie temperature in gamma-FeNi nanoparticles for magnetocaloric applications by controlling the oxidation kinetics (vol 113, 17A918, 2013)
Ucar, H; Ipus, JJ; Laughlin, DE; McHenry, ME
Journal of Applied Physics, ***11**3* (2013) 159902
DOI: 10.1063/1.4801929

Mechanically alloyed Fe70 Ni 30 and Fe72 Ni 28 alloys were characterized in terms of their structural and magnetic properties. Previous studies showed that single phase FCC γ-FeNi alloys with ~26-30 at. % Ni have Curie temperatures, Tc, near room temperature. Having Tc near room temperatures along with large magnetization makes γ-FeNi alloys attractive for room temperature magnetocaloric cooling technologies. To obtain a single γ-phase, particles were solution annealed in the γ-phase field and water quenched. The preferential oxidation of Fe during ball milling was used as a means to tune the Curie temperature, Tc, of the alloy. Refrigeration capacities, RCFWHM, of the Fe70 Ni 30 and the Fe72 Ni 28 alloys were calculated to be ≈470 J/kg and 250 J/kg at 5 T, with peak temperatures ≈363 K and ≈333 K, respectively. The RCFWHM for the Fe70 Ni 30 is higher than the previously reported Nanoperm (Fe70 Ni 30)89Zr7B4 type alloy and on the same order of magnitude with other Fe-based alloys. The maximum magnetic entropy change values observed for the Fe70 Ni 30 and the Fe72 Ni 28 are 0.65 and 0.5 J kg−1 K−1, respectively, at a field of 5 T. These are smaller than those of rare earth magnetic refrigerants showing first order transformation behavior. The larger RCFWHM value results mainly from the width of the magnetic entropy curve in these types of materials. We discuss the economic advantage of these rare earth free refrigerants

Magnetocaloric effect and critical behavior of amorphous (Gd4Co3)(1-x)Si-x alloys
Zheng, ZG; Zhong, XC; Liu, ZW; Zeng, DC; Franco, V; Zhang, JL
Journal of Magnetism and Magnetic Materialss, **343** (2013) 184-188
DOI: 10.1016/j.jmmm.2013.04.087

The amorphous alloys (Gd4Co3)(1-x)Si-x (x=0, 0.05, and 0.10) were prepared by melt spinning. The Curie temperature can be tuned from 213 to 198 K with increasing Si content, while the maximum value of magnetic entropy change for a magnetic field change of 0-5 T can reach 7.3, 7.2 and 6.4 J kg(-1) K-1 for x=0, 0.05 and 0.10 respectively. Large values of the refrigerant capacity are obtained in these alloys. The saturation magnetization at 5 K equals 26.5 mu(B), 24.2 mu(B) and 22.4 mu(B) per (Gd4Co3)(1-x)Si-x formula with x=0, 0.05 and 0.10, respectively. All samples undergo a second order ferri paramagnetic phase transition. The critical behavior around the transition temperature is investigated by means of the modified Arrott plots technique, the Kouvel-Fisher method and the Widom scaling relation. The results indicate that for x=0 and 0.05, the critical exponents are close to those of the 3D Ising model, while the critical exponents for x=0.1 are between those of the 3D Ising model and mean-field model.

The Role of Microstructure and Processing on Magnetic Properties of Materials
Willard, MA; Franco, V
JOM, **52** (2013) 17-23
DOI: 10.1016/j.applthermaleng.2012.11.001

Metastable Soft Magnetic Materials Produced by Mechanical Alloying: Analysis Using an Equivalent Time Approach
Blazquez, JS; Ipus, JJ; Lozano-Perez, S; Conde, A
JOM, **65** (2013) 870-882
DOI: 10.1007/s11837-013-0616-1

Mechanical alloying, a ball milling technique where heterogeneous starting powders are homogenized and alloyed, has become a versatile technique for the production of metastable microstructures: nanocrystalline and/or amorphous alloys, supersaturated solid solutions, etc. Ball milling is affected by many experimental parameters and the analysis of the dynamics of milling is needed to compare different experiments and to optimize time and costs. In this work, a short overview on the microstructural techniques used to characterize the powder is supplied. The soft magnetic properties of mechanically alloyed powders are also described, as well as the magnetocaloric response of these systems.

LIBROS Y OTRAS PUBLICACIONES / BOOKS AND OTHER PUBLICATIONS

Calorimetric study of avalanche criticality in the martensitic phase transition of $Cu_{67.64}Zn_{16.71}Al_{15.65}$
M.C. Gallardo, F.J. Romero, J. Manchado, J.M. Martín-Olalla, A. Planes, E. Vives, E.K.H. Salje, R. Romero and M. Stipcich
Materials Science Forum, **738-739** (2013) 46-50
DOI: 10.4028/www.scientific.net/MSF.738-739.46

The first-order diffusionless structural phase transition in Cu67.64Zn16.71Al15.65 is characterized by jerky propagation of phase front related to the appearance of avalanches. In this work we describe a full analysis of this avalanche behaviour using calorimetric heat-flux measurements and the results are compared with acoustic emission (AE) measurements.

Three different scanning temperature rates have confirmed that the phase front of the transition in Cu67.64Zn16.71Al15.65 is developed in two regimens, a continuous background and the avalanche jerks. The smooth background is reproducible even at ultra low scanning temperature rate. The jerks are well defined when the scanning temperature rate is lower, so that the statistic analysis is more reliable and the plateau obtained on heating by ML method is enhanced. Nevertheless on cooling no plateau is obtained even for the smallest temperature scanning rate.

Although that avalanches seen in calorimetry are vastly more energetic than those seen in AE, the statistical analysis is identical for both experimental techniques, it becomes clear that the full range of the power law extends over several decades.

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

COMUNICACIONES EN CONGRESOS Y REUNIONES / COMMUNICATIONS IN CONGRESS AND MEETINGS

12th Joint MMM/Intermag Conference

14 –18 enero [Chicago, Estados Unidos de América]

Influence of magnetic interactions on the magnetocaloric effect of composites
C. Romero-Muñiz, V. Franco, A. Conde
Comunicación oral

The use of amorphous boron powder enhances mechanical alloying in soft magnetic FeNbB alloy
J.J. Ipus, J.S. Blázquez, V. Franco, A. Conde
Poster

Low hysteresis and large room temperature magnetocaloric effect of Gd5Si2.05-xGe1.95-xNi2x (2x=0.08, 0.1) compounds
J. Min, X. Zhong, Z. Liu, Z. Zheng, D. Zeng, V.Franco,R.V. Ramanujan
Poster

Magnetic properties and magnetocaloric effects in GdCo9Si2 compound with multiple magnetic phase transitions
Z. Zheng, X. Zhong, V. Franco, J. Zhang, Z. Liu, D. Zeng
Poster

142nd Annual Meeting & Exhibition - TMS 2013

3 – 7 marzo [San Antonio, Texas, Estados Unidos de América]

Optimization of the Mechanical Alloying Process of Soft Magnetic Fe-Based Powders: An Equivalent Time Approach
J.S. Blázquez, J.J. Ipus, V. Franco, A. Conde
Conferencia Invitada

15th Czech and Slovak Conference on Magnetism - CSMAG'13

17 – 21 junio [Kosice, República Eslovaca]

The magnetocaloric effect of multiphase materials and nanostructures
V. Franco, A. Conde
Conferencia Invitada

20th International Symposium on Metastable, Amorphous and Nanostructured Materials - ISMANAM 2013

30 junio – 5 julio [Turin, Italia]

Mechanical alloying as an effective method to produce soft magnetic amorphous and nanocrystalline alloys
C.F. Conde, J.S. Blázquez, J.J. Ipus, V. Franco, A. Conde
Conferencia Invitada

Enthalpy relaxation and Curie temperature behaviour in Fe(Co)SiAlGaPCB amorphous alloys upon low temperature annealing
J.M. Borrego, J.S. Blázquez, C.F. Conde, A. Conde
Poster

Development of metalloid free amorphous and nanocrystalline alloys for high temperature applications as soft magnets
J.S. Blázquez, J.J. Ipus, C.F. Conde, D. Cabrera, V. Franco, A. Conde
Poster

Evolution of Fe environments in mechanical alloyed Fe-Nb-(B) compositions
J.S. Blázquez, J.J. Ipus, C.F. Conde, A. Conde
Poster

XXII International Materials Research Congress (IMRC) 2013

11 – 15 agosto [Cancún, México]

Analysis of direct adiabatic temperature change measurements using ac techniques
E. Espejo, V. Franco, A. Conde
Comunicación oral

The use of the magnetocaloric response to study compositional changes in mechanically alloyed samples
J.J. Ipus, J.S. Blázquez, V. Franco, A. Conde
Poster

Joint European Magnetic Symposia - JEMS 2013

25 – 30 agosto [Rodas, Grecia]

Gaining information of phase transitions and critical phenomena via magnetocaloric studies
V. Franco, A. Conde
Conferencia Invitada

58th Annual Conference on MMM 2013

4 – 8 noviembre [Denver, Estados Unidos de América]

Magnetic properties of amorphous Co62Nb6Zr2B30 obtained by mechanical alloying
L. Moreno, J.S. Blázquez, V. Franco, A. Conde
Comunicación oral

Deconvolution of magnetocaloric effect of multiphase materials
V. Franco, J.S. Blázquez, A. Conde, J. Marcin, I. Skorvanek
Comunicación oral

Long-range ferromagnetism in Gd53Al24Co20Zr3 amorphous microwires: Role of structural disorder
A. Biswas, P. Zhang, Y.Y. Tu, N.S. Bingham, H. Wang, F. Qin, H.X. Peng, J.F. Sun, S.C. Yu, V. Franco, H. Shrianth, M.H. Phan
Poster

Milling effects on magnetic properties of melt spun Fe-Nb-B alloy
J.J. Ipus, J.S. Blázquez, V. Franco, A. Conde
Poster

Broadening the magnetic entropy change and its correlation with the Curie temperatura distributuion on high-energy ball milled R2Fe17 alloys
P. Álvarez-Alonso, J.L. Sánchez-Llamazares, C.F. Sánchez-Valdés, G.J. Cuello, V. Franco. P. Gorría, J.A. Blanco
Poster

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 ESPECTROSCOPÍAS / SPECTROSCOPY SERVICE

El Servicio de Espectroscopía 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.

RESONANCIA MAGNÉTICA NUCLEAR EN SU CONFIGURACIÓN DE ESTADO SÓLIDO / NUCLEAR MAGNETIC RESONANCE IN ITS SOLID-STATE CONFIGURATION

La Resonancia Magnética Nuclear en estado sólido es utilizada para el estudio de la estructura atómica de materiales amorfos o poco cristalinos. Se basa en el comportamiento de los momentos magnéticos de ciertos núcleos atómicos bajo la influencia de un campo magnético externo y de los campos locales creados por los núcleos vecinos y por la distribución de carga electrónica en torno al núcleo. Los espectros más comunes son representaciones de la intensidad de absorción frente a la frecuencia de resonancia y presentan señales cuya posición, forma y tamaño están íntimamente relacionados con el entorno químico del núcleo estudiado. Permite el estudio del orden a corto alcance de materiales estructuralmente desordenados y diferencia átomos con números atómicos similares.

Solid State Nuclear Magnetic Resonance is a widely use technique to study the structural properties of materials, including amorphous and not very crystalline solids. This technique is based on the behavior of the magnetic moments of the nuclei under an external magnetic fields and the local magnetic field due to the environment. Thus, the position, shape and intensity of the signals inform of the chemical environment of the nuclei.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- Espectrómetro de RMN multinuclear Bruker, mod. DRX-400
  Multinuclear RMN Spectrometer (Bruker, mod. DRX-400)
- Sonda CP/MAS de 4 mm dotada de doble canal, 1H y BB (en el rango de frecuencia desde 15N a 31P)
  CP/MAS sound of 4 mm with double channel, 1H and BB (at a frequency range from 15N to 31P)
- Unidad neumática, que permite alcanzar velocidades de giro bajo ángulo mágico de hasta 15000 Hz
  Neumatic unit, that allows to reach turn velocities under a magic angle up to 15000 Hz

- Accesorio de temperatura variable dotado de una línea propia de N_2, así como de un sistema de control de temperatura que permite trabajar en un rango entre 223 K y a 373 K.
  Accessory of variable temperature with a line of its own of N_2, and with a temperature control system that allows to work at a range of 223 K - 373 K.

Responsable Científico/Scientific Responsible: Dra. María Dolores Alba Carranza
Personal Técnico/Technical Assistant: Dr. Miguel Angel Avilés Escaño

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 than excited in infrared. 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 bounds, whenever it occur 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 of 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) report 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 tres técnicas: Análisis Termogravimétrico (TG), Análisis Térmico Diferencial (ATD) y Calorimetría Diferencial de Barrido (CDB).

Thermal analysis techniques allows 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.
Three different techniques are available: Thermogravimetry (TG), Differential Thermal Analysis (DTA) and Differential Scanning Calorimetry (DSC).

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
- Equipo de calorimetría diferencial de barrido CDB, TA Instruments Q20
  Differential scanning calorimeter DSC, TA Instruments Q20

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), acopladas una y otra con las Espectroscopías de Energía Dispersiva de Rayos X (EDS) y de Pérdida de Energía de Electrones (EELS).
Las tareas ofrecidas por el personal técnico comprenden la preparación de muestras, su estudio por la batería de técnicas disponibles (TEM, SEM, HRTEM, SAED, EDS, EELS), la adquisición de imágenes y espectros, y en algunos casos, la interpretación de los datos experimentales.

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 and Scanning Electron Microscopy, which are equipped with a Electron Energy Loss Spectrometre and a Energy Dispersive X-ray Analyser, respectively.
The technicians working on this service carryied out different tasks, as sample preparation, image and spectral adquisition, and in some particular cases, interpretation of experimental results.

MICROSCOPÍA ELECTRÓNICA DE BARRIDO / SCANNING ELECTRON MICROSCOPY

La microscopía electrónica de barrido proporciona información microestructural, morfológica y de composición química en escala microscópica (hasta una resolución lateral de ~ 5 nm). 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.

The scanning electron microscopy provides information about the microstructure, morphology and chemical composition at the microscopic scale of solid samples (upto a lateral resolution of ~ 5 nm). 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.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- Microscopio SEM, modelo JEOL JSM-5400, dotado de analizador EDX Oxford Isis, con resolución de 138 eV. Cañón termoiónico de filamento de W
  JEOL JSM-5400 microscope equipped with an Oxford Isis EDX analyzer with a resolution of 138 eV. Thermoionic W electron gun

- Microscopio SEM, modelo Hitachi S4800 SEM-FEG de alta resolución (1-3 nm). Dotado de analizador EDX Bruker X Flash Detector 4010 con una resolución de 133 eV. Cañón de emisión de campo y detector STEM
  Hitachi S4800 SEM-FEG microscope of high resolution (1-3 nm). Equipped with a Bruker X flash 4010 EDX detector with a resolution of 133 eV, field emission gun and STEM detector.
- Equipamiento anexo para la preparación de muestras: metalizadores, cortadoras, etc.
  Sample preparation equipment: metallization devices, cutting tools, etc.

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, pudiéndose obtener información sobre morfología, tamaño de grano, composición química, grado de cristalinidad e identificación de fases cristalinas en todo tipo de materiales. La espectroscopia de pérdida de energía de electrones (EELS) acoplada al microscopio TEM constituye una herramienta adicional para determinar la estructura electrónica, el tipo de enlace y la formación de fases.

The transmission electron microscopy is a widely used technique for the microstructural and chemical characterization at micro and nanoscales, giving information about morphology, grain size, chemical composition, crystallinity and identification of crystalline phases in all type of materials. The energy loss spectroscopy (EELS), coupled to the TEM microscope, constitutes an additional tool for the determination of electronic structure, type of bond and phase formation.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- Microscopio JEM-2000EX con una resolución estructural de 2.1 Å
  JEM-2000EX microscope with a structural resolution of 2.1 Å.
- Microscopio Philips CM200 con una resolución estructural entre puntos de 2.3 Å, portamuestras de uno y dos giros, calentamiento y enfriamiento. Acoplado al equipo se dispone de un sistema de análisis por Energías Dispersivas de Rayos X (EDX) y Espectrómetro de Pérdidas de Energía Electrónica (PEELS), de GATAN modelo 766-2 keV
  Philips CM200 microscope with a structural resolution of 2.3 Å. Sample holder with one and two angles, heating and cooling. It is equipped with a X-ray Energy Dispersive Analyser (EDX) and a Electron Energy Loss Spectrometre (PEELS) (Gatan, model 766-2 keV.

- Laboratorio de preparación de muestras para microscopía, que comprende: Evaporador de carbón, pulidora, cortadoras de disco y ultrasónica, adelgazador cóncavo-convexo, adelgazadores iónicos: Balttec (modelo RES 100) y Fischione (modelo 1010)
  Sample preparation laboratory, equipped with carbón evaporator, cutting and polishing tools, dimple, ion milling guns Balttec (model RES100) and Fischione (model 1010)

Responsable Científico/ Scientific Responsible: Dra. Asunción Fernández Camacho
Personal Técnico/ Technical Assistant: Dª Olga Montes Amorín (CicCartuja) y Dª María Inmaculada Roja Cejudo

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 incluye las Unidades de Espectroscopía de Fotoelectrones de Rayos X (XPS) y de Espectroscopia de Descarga Luminiscente (GDL). 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 two untis: the X-ray Photoelectron Spectroscopy (XPS) and the Glow Discharge Luminescence. 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
Personal Técnico/Technical Assistant: D. David Benítez Sánchez

SERVICIO DE ESPECTROMETRÍA DE EMISIÓN ÓPTICA POR DESCARGA LUMINISCENTE/ GLOW DISCHARGE OPTICAL EMISSION SPECTROMETRY SERVICE (GD-OES)

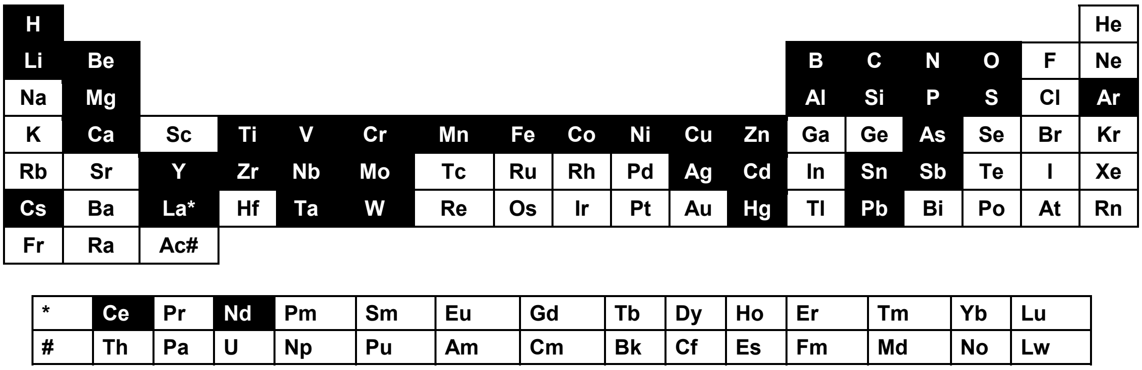
La técnica de Espectrometría por Descarga Luminiscente constituye una potente herramienta para el análisis rápido de sólidos, tanto en masa como en superficie. Además se pueden determinar concentraciones elementales en función de la profundidad lo que permite realizar perfiles de concentración sobre materiales avanzados: metales tratados, materiales recubiertos, multicapas, superficies pintadas, capas finas, procesos de corrosión y oxidación, etc.. La descarga luminiscente es un plasma autosustentado que se genera en un gas encerrado a baja presión (1 kPa aproximadamente) entre dos electrodos que se encuentran a una diferencia de potencial del orden de 1 kV. La diferencia de potencial puede lograrse tanto mediante la aplicación de corriente continua (DC), como mediante radiofrecuencia (RF), pudiéndose medir tanto muestras conductoras como no conductoras.

Glow discharge optical emission spectrometry (GDOES) has emerged as a major technique for surface and depth profile analysis, owing to the unique combination of fast sputtering rate, high depth resolution, excellent sensitivity and multi-element capability. Glow discharge is a selfsustained plasma that is generated in a gas enclosed to low pressure (1 kPa) between two electrodes at a potential difference of 1 kV. This potential difference can be achieved by the application of a direct current (DC.) or by radio frequency (RF), which offers the advantage that both conducting and non-conducting samples can be analyzed.

Instrumental Disponible / Available Equipment

- Espectrómetro de descarga luminiscente LECO GDS 750A equipado con dos policromadores holográficos de 3600 y 1800 líneas, óptica a vacío que permite el análisis en el UV y 43 fotomultiplicadores que permiten analizar los elementos resaltados en negro en la tabla mostrada abajo. Puede trabajar en modo de funcionamiento en DC y RF.

  Glow Discharge Spectrometer LECO GDS 750ª equipped with two holographic diffraction gratings of 1800 lines/mm and 3600 lines/mm, optics in vacuum and 43 photomultipliers giving the ability to detect the elements highlighted in the table below. It is equipped with a RF and a DC sputter source.



H																	He
Li	**Be**											**B**	**C**	**N**	**O**	F	Ne
Na	**Mg**											**Al**	**Si**	**P**	**S**	Cl	**Ar**
K	**Ca**	Sc	**Ti**	**V**	**Cr**	**Mn**	**Fe**	**Co**	**Ni**	**Cu**	**Zn**	Ga	Ge	**As**	Se	Br	Kr
Rb	Sr	**Y**	**Zr**	**Nb**	**Mo**	Tc	Ru	Rh	Pd	**Ag**	**Cd**	In	**Sn**	**Sb**	Te	I	Xe
Cs	Ba	**La***	Hf	**Ta**	**W**	Re	Os	Ir	Pt	Au	**Hg**	Tl	**Pb**	Bi	Po	At	Rn
Fr	Ra	Ac#															

*	**Ce**	Pr	**Nd**	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu
#	Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lw

Responsable Científico/ Scientific Responsible: Miguel Angel Centeno Gallego

Servicio de Mecanizado/ Mechanical 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

Además de otras herramientas menores para cortar, soldar, doblar, taladrar, etc., el taller de mecanizado cuenta con los siguientes equipos:
Besides other minor tools for cutting, soldering, folding, drilling, etc, this workshop is equipped with:

- Centro de mecanizado de control numérico computerizado (CNC), 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 manual EMCO SUPER 11CD y torno semiautomático PINACHO SMART TURN180
  A couple of turning machines: models PINACHO TURN180 and SUPER 11 CD EMCO

Responsable/ Responsible: D. Enrique Jiménez Roca
Personal Técnico: D. Juan Carlos Sánchez Martín

ACTIVIDADES DIVULGATIVAS Y FORMATIVAS
OUTREACH AND TEACHING ACTIVITIES

DOCENCIA / TEACHING

CURSOS DE POSTGRADO / POSTGRADUATE COURSES

MÉTODOS FÍSICOS DE ANÁLISIS DE CAPAS FINAS Y SUPERFICIES DE SÓLIDOS

Curso de Postgrado del CSIC
Programa 2010

Métodos Físicos de Análisis de Capas Finas y Superficies de Sólidos

Sevilla, del 28 de Junio al 2 de Julio de 2010

Este curso de postgrado pretende familiarizar a los asistentes con algunos de los métodos físicos de análisis más utilizados en la actualidad para la caracterización de capas finas y superficies. El curso, de una semana de duración, constará de clases teóricas y sesiones prácticas. Será impartido por profesores de la Universidad de Sevilla e investigadores del CSIC, en las instalaciones y con el equipamiento científico disponible en el Instituto de Ciencia de Materiales de Sevilla y el Centro Nacional de Aceleradores.

Está dirigido a científicos e ingenieros especializados en temas de análisis, diagnóstico e investigación en materiales y superficies. También se considera especialmente adecuado para alumnos de tercer ciclo de ingenierías de materiales.

Temáticas

Aplicaciones Tecnológicas de las Películas Delgadas
Técnicas de Preparación de Películas y Recubrimientos (CVD, PVD)
Espectroscopías de Fotoemisión (XPS, UPS)
Microscopías Electrónicas de Transmisión y Barrido (TEM y SEM) Espectroscopías Electrónicas (Auger, REELS, EDX, PEELS)
Difracción de Rayos X
Absorción de Rayos X (EXAFS, XANES)
Dispersión de Iones (ISS, RBS)
Microscopías de Aproximación de puntas (AFM, STM)

Directores:

Dr. Juan Pedro Espinós Manzorro
Instituto de Ciencia de Materiales de Sevilla
jpespinos@icmse.csic.es

Dr. Guillermo Munuera Contreras
Instituto de Ciencia de Materiales de Sevilla
Dpto. Química Inorgánica

Instituto de Ciencia de Materiales de Sevilla
c/ Américo Vespucio, 49, 41092 (Sevilla)
Telf: 954 48 95 27 • Fax: 954 46 06 65
http://www.icmse.csic.es/cursocapas.htm
CUOTA DE INSCRIPCIÓN: 300 €

Hay disponible un número limitado de becas para alumnos de tercer ciclo (adjuntar curriculum vitae y una carta de presentación de un investigador cualificado)

Organizado por el Instituto de Ciencia de Materiales de Sevilla
Directores: Dr. Juan Pedro Espinós y Dr. Guillermo Munuera
Fecha de Celebración: 24 al 28 de junio de 2013
Horas lectivas: 25 de teoría + 15 de prácticas
Dirigido a: Grado de Doctor, Estudiante de Doctorado o Especialista de Empresa

Este curso de postgrado pretende familiarizar a los asistentes con algunos de los métodos físicos de análisis más utilizados en la actualidad para la caracterización, de materiales en general, y de capas finas y superficies en particular. El curso, de carácter intensivo y una semana de duración, consta de clases teóricas y sesiones prácticas. Estas últimas, desarrolladas con grupos de reducidos de alumnos, se realizarán con el equipamiento científico disponible en el Instituto de Ciencia de Materiales de Sevilla, el Centro Nacional de Aceleradores y el Servicio de Espectroscopía de Fotoelectrones de la Universidad de Sevilla.
El contenido y enfoque de los temas y otras actividades del curso son eminentemente prácticos, estando dirigido a científicos e ingenieros especializados en temas de análisis, diagnóstico, investigación en superficies, etc. También se considera especialmente adecuado para alumnos de tercer ciclo relacionados con la temática tratada.

Teoría**:**
Interacción de Fotones, Electrones e Iones con la Materia Condesada. Métodos Físicos de Análisis de Capas Finas y Superficies de Sólidos | Dr. Agustín R. González-Elipe
Espectroscopía de Fotoemisión de Rayos X: Composición Superficial | Dr. Juan Pedro Espinós
Espectroscopía de Fotoemisión de Rayos X: Estado Químico en la Superficie de Sólidos | Dr. Agustín R. González-Elipe
Determinación de Perfiles de Composición a partir del Análisis de Forma de Pico en XPS: Método de Tougaard | Dr. Francisco Yubero Valencia
Plasmas en Tecnología de Materiales| Dr. José Cotrino Bautista
Microscopías Efecto Túnel y de Fuerzas Atómicas | Dr. José Jesús Benítez Jiménez

Microscopía Electrónica de Transmisión: Fundamentos y Aplicaciones Generales | Dra. Concepción Real Pérez
Microscopía Electrónica: PEELS y EFTEM| Dra. Cristina Rojas Ruiz
Microscopía Electrónica de Barrido| Dra. María del Carmen Jiménez de Haro
La Difracción de Rayos X: Generalidades | Dr. Angel Justo Erbez
Análisis Tribológico y Mecánico de Capas Delgadas | Dr. Juan Carlos Sánchez López
Métodos de Determinación de Espesores de Capas Finas | Dr. Juan Pedro Espinós Manzorro
Absorción de Rayos X: Orden a Corto Alcance en Superficie y Láminas Delgadas| Dr. Juan Pedro Holgado Vázquez
Retrodispersión Rutherford: Perfiles de Composición | Dr. Miguel Angel Respaldiza (CNA)

Prácticas:
a. Microscopías Electrónicas de Transmisión y Barrido | Dra. Cristina Rojas Ruiz | Dra. Carmen Jiménez de Haro | Lda. Olga Montes
b. Espectroscopía de Fotoemisión, XPS | Dr. Juan Pedro Espinós | Dr. Francisco Yubero | Dr. Juan Pedro Holgado
c. Difracción y Fluorescencia de Rayos X | Dr. Angel Justo | Ldo. José M. Martínez
d. Absorción de Rayos X | Ldo. Victor González
e. Retrodispersión Rutherford | Dr. Javier García
f. Microscopía de Fuerzas Atómicas y Túnel | Dra. Ana Borrás | Dr. Carlos Sánchez | Dr. Rafael Alvarez, Lda. Antonia Terriza

MÁSTER / MASTER

MÁSTER EN CIENCIA Y TECNOLOGÍA DE NUEVOS MATERIALES

Organizado por la Universidad de Sevilla
Coordinador: Dr. José Antonio Odriozola Gordón
Fecha de Celebración: Curso Académico 2012-13
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 Europeos: 3.25) | Dra. María Dolores Alba Carranza | Dr. Miguel Angel Castro | Dra. Pilar Malet
Fisica del Estado Sólido (Créditos: 2.85) | Dra. Angela Gallardo López | Dra. Josefa Borrego | Dr. Manuel Jiménez Melendo

Técnicas de Caracterización de Materiales (Créditos: 5.20) | Dr. Juan Pedro Holgado Vázquez | Dr. Julián Martínez Fernández | Dr. Juan M. Montes (US)
Síntesis de Materiales y Nanoestructuras (Créditos: 4.55) | Dra. Asunción Fernández Camacho | Dr. Francisco J. Gotor | Dr. Juan Pedro Espinós | Dr. Manuel Ocaña
Catalizadores para la Energía y el Medio Ambiente (Créditos: 3.25) | Dr. Miguel Angel Centeno Gallego | Dr. José Antonio Navío
Materiales con Funcionalidad Superficial (Créditos: 3.25) | Dr. Agustín R. González-Elipe
Corrosión y Recubrimientos Protectores (Créditos: 5) | Dr. Antonio Paúl (US) | Dr. Juan Carlos Sánchez López | Dr. L. Soria Conde (US) | Dr. Enrique Herrera (US)
Recuperación y Transformación de Materiales (Créditos: 3.25) | Dr. José Antonio Odriozola | Dra. María Isabel Domínguez Leal | Dr. Luis A. Pérez-Maqueda
Procesado de Materiales Estructurales (Créditos: 3.25) | Dr. Alfonso Bravo León | Dr. José Antonio Rodríguez Ortíz
Comportamiento Mecánico (Créditos: 3.25) | Dr. Diego Gómez García
Comportamiento Magnético (Créditos: 3.25) Dr. Javier S. Blázquez Gámez | Dr. Victorino Franco García
Comportamiento Térmico, Dieléctrico y Óptico de Materiales (Créditos: 2.85) | Dr. Hernán Míguez García | Dr. Francisco Javier Romero Landa | Dra. Carmen Gallardo Cruz
Computación en Ciencia de Materiales (Créditos: 3.25) | Dra. Regla Ayala Espinar | Dr. Antonio Córdoba Zurita (US) | Dr. Felipe Gutiérrez Mora

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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 en la Universidad de Sevilla.

Otros cursos / Other Courses

Introducción a la protección del conocimiento

Organizado por la Vicepresidencia Adjunta de Transferencia del Conocimiento (delegación de Andalucía) y el Instituto de Ciencia de Materiales de Sevilla (proyecto Al-NanoFunc)
Directores: Dr. J.M. Armesto y Dra. V. Godinho
Fecha de Celebración: 18 de febrero de 2013
Horas lectivas: 4 horas
Dirigido a: Investigadores y gestores de centros de investigación, centros tecnológicos y empresas.

Tratamiento del IPR en los proyectos europeos: Los Acuerdos de Consorcio.
Dr. Francisco Orozco (Unidad Legal Programas Europeos, VICYT - CSIC).
Bases de datos de información sobre patentes.
Dr. Björn Jürgens (Agencia IDEA - CITPIA PATLIB Centre).
Protección de los resultados de investigación en la Universidad de Sevilla.
Dr. Raquel Delgado (OTRI - US).
Explotación de resultados: Contratos de I+D - CDAs - MTAs - Licencias.
Dr. Ana García Navarro (VATC -CSIC, Inst. de Ciencia de Materiales de Sevilla).
Introducción a la protección del conocimiento.
Dr. Juan Martínez Armesto (VATC -CSIC, Delegación en Andalucía).
Presentación de servicios de la VATC del CSIC.
Dr. Juan Martínez Armesto (VATC -CSIC, Delegación en Andalucía).

CONFERENCIAS INVITADAS IMPARTIDAS POR PERSONAL DEL ICMS
INVITED CONFERENCES BY PERSONNEL OF THE ICMS

Modelo para la evaluación del potencial de rehabilitación de la arquitectura rural
Pedro J. Sánchez Soto, E. Garzón
Universidad de la Rioja
Logroño (España), Febrero

A microestructural study of additives / catalysts used in hydrogen storage processes based on hydride materials
Asunción Fernández Camacho
EMPA (Swiss Federal Laboratory for Materials Science and Technology)
Dübendorf (Suiza), Enero

CONFERENCIAS Y SEMINARIOS IMPARTIDOS EN EL ICMS
CONFERENCES AND SEMINAR IN THE ICMS

29 de enero| **Plasmons in metallic hole arrays and nanoparticles in an optical trap**
Dra. Sol Carretero-Palacios
Photonics and Optoelectronics Group. Faculty of Physics and Center for Nanoscience (CeNS). Ludwing-Maximilians-Universität München

Subwavelength apertures periodically arranged in a metal film may transmit electromagnetic waves beyond the cutoff wavelength of the holes, with a much higher intensity than if they were isolated. It has been established that resonant excitation of surface plasmons creates huge electric fields at the metallic surface, forcing light through the holes and giving very high transmission coefficients. This is the so-called Extraordinary Optical Transmission.

I will present a detailed theoretical study of the physical mechanisms controlling the transmission process in a Bull's Eye Geometry (a single subwavelength circular hole surrounded by concentric annular grooves).

I will also show final results for other systems containing annular holes, such us arrays of annular dimples (of finite thickness) designed for detection in the terahertz regime, and a description for resonances appearing at unusual wavelengths beyond the cutoff of the holes. Also the nonlinear optical response in arrays of subwavelength slits in combination with nonlinear Kerr-type dielectrics will be shown.

Finally, I will describe the Dark Field Microscopy technique and present some of its applications for manipulating micro- and nano-particles (metallic, dielectric, and hybrid ones).

3 de mayo| **Espectroscopía de Neutrones, aplicaciones en Química, Ciencia de Materiales y Catálisis**

Dr. Timmy Ramírez-Cuesta

ISIS Facility, STFC, Rutherford Appleton Laboratories, UK

Las técnicas de neutrones son muy útiles en el estudio de la dinámica de los átomos y moléculas de hidrogeno. La naturaleza de la interacción entre el neutrón y átomo es responsable de la gran sección eficaz del núcleo del átomo de hidrogeno. En particular, el neutrón inspecciona simultáneamente la estructura y dinámica de los átomos del solido, en ambos casos la conexión entre los cálculos dinámicos utilizando métodos de DFT se corresponden rigurosamente con los espectros inelásticos dado que el neutrón interactúa directamente con el núcleo atómico, a diferencia del caso de las espectroscopias ópticas donde la dinámica del núcleo es determinada indirectamente a través de la interacción del fotón con la nube electrónica.

Otra característica muy importante de la interacción del neutrón es que es una prueba muy penetrante, por ejemplo, casi todos los metales poseen secciones eficaces muy pequeñas. Esto permite hacer experimentos utilizando contenedores de muestras de acero, aluminio etc., sin necesidad de tener ventanas ópticas, en consecuencia el uso de gases para dosificar solidos porosos, catalizadores y otros es trivial.

En esta charla se presentaran los aspectos básicos de la espectroscopia inelástica de neutrones y el uso de DFT para interpretar los experimentos como así también algunos ejemplos de aplicaciones al estudio de materiales solidos, gases adsorbidos en matrices porosas (hidrogeno, captura de CO2, separación de etileno y acetileno entre otros)

26 de septiembre | **Enhancing Particle Applications by Surface Modification**

Prof. Richard Partch

Chemistry Center for Advanced Materials Processing Clarkson University

Gas, aerosol and liquid dispersion methods for particle synthesis and surface modification carried out by the speaker´s research group at Clarkson University will be presented. Briefly discussed will be colloids having a wide variety compositions, shapes and sizes useful for : 1) metal matrix and rubber composites, 2) protecting cores from acidic and humid environments, 3) improving print toner charging and reducing energy use by copy machines and fluorescent lights, 4) reducing polishing defects in microelectronic processing, 5) medical imaging, treatment of chemical overdoses and cancer and artificial organs and 6) security.

Otras Actividades / Other Activities

Feria de la Ciencia / Fair of Science

La Feria de la Ciencia (9 al 11 de mayo de 2013, en Sevilla) 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: "De la Microscopía óptica a la microscopía electrónica: Un sorprendente viaje del micro al nanomundo".
Coordinadora: Asunción Fernández Camacho. Participaron un total de 16 monitores.

The Fair of Science (9 to 11 May 2013, 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: "From optical to electron microscopy. A voyage from the micro- to the nano-world"
Coordination: Asunción Fernández Camacho.

SEMANA DE LA CIENCIA Y LA TECNOLOGÍA / SCIENCE AND TECHNOLOGY WEEK

Jornadas de puertas abiertas (Semana del 4 al 17 de Noviembre de 2013, en Sevilla). 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.

Visitas guiadas

Coordinadores Drs. A. Ruiz Conde y P.J. Sánchez Soto

Charlas

12 de Noviembre de 2013. Dr. José Cotrino Bautista

Titulo: "Tecnología de plasmas y materiales”

15 de Noviembre de 2013. Dras.Gisela Arzac

Titulo: “El hidrógeno como combustible sostenible”

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

4 de noviembre de 2013. Dr. Juan Carlos Sánchez López. Coloquio con estudiantes. Tema: "Reduciendo el desgaste con nanomateriales: ¡divide y vencerás!"

IES Hipatia. IES La Campiña

Exposición de Fotografías

La Semana de la Ciencia, ha incluido en su programa de actividades una exposición de fotografía científica, titulada "Microscopía electrónica: todo un arte". La exposición coordinada por Asunción Fernández Camacho, investigadora del Instituto de Ciencia de Materiales de Sevilla (ICMS) y responsable de la Red Al-NanoFunc (dedicada al estudio internacional de los nanomateriales funcionales).

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