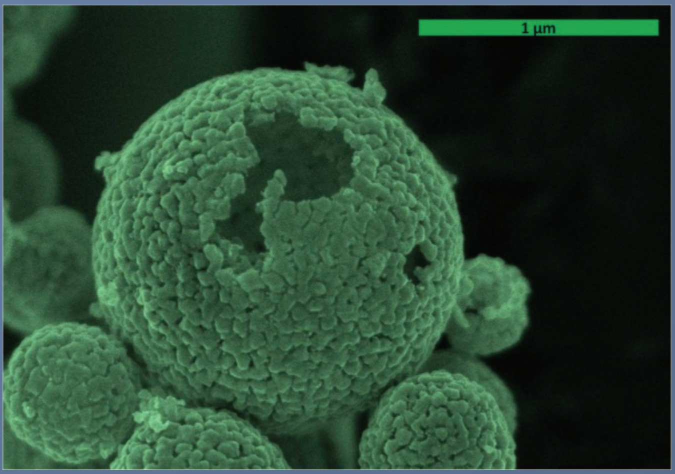
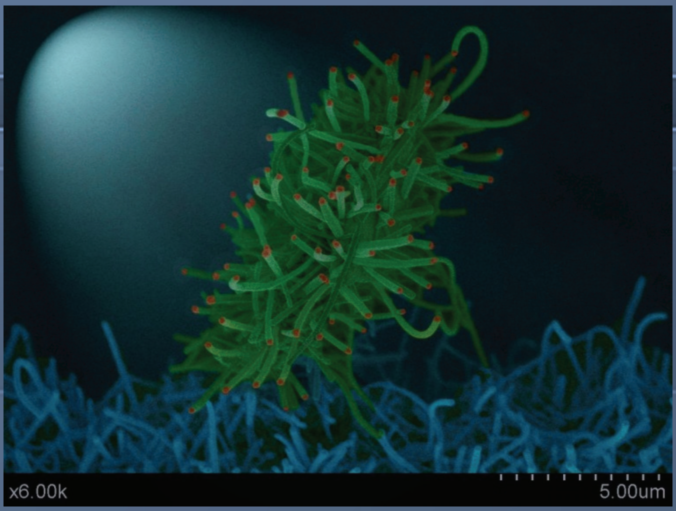
ICMS

Instituto de Ciencia de Materiales de Sevilla





Consejo
Superior de
Investigaciones
Científicas

Universidad
de Sevilla

Junta de
Andalucía

Memoria de Actividades
Annual Report

2012

Consejo Superior de Investigaciones Científicas
Universidad de Sevilla
Junta de Andalucía

INSTITUTO DE CIENCIA DE MATERIALES DE SEVILLA

Memoria de Actividades
Annual Report
2012

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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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DISEÑO Y MAQUETACIÓN
José Carlos Rivero Cabello

FOTOGRAFÍAS DE PORTADA
“Catálisis verde” - Autor: Rosa María Pereñíguez Rodríguez
“Nanoanémona” - Autor: Manuel Macías-Montero
Fotografías premiadas en el concurso Al-NanoPhoto 2012

COMISIÓN MEMORIA-ICMS
Hernán R. Míguez García – Manuel Ocaña Jurado – Pedro José Sánchez Soto

INDICE

TABLE OF CONTENTS

El ICMS en 2012

ICMS in 2012

Presentación Presentation

A través de esta Memoria 2012, 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 136 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 2012, 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 136 people, 48 of which are permanent scientific staff.

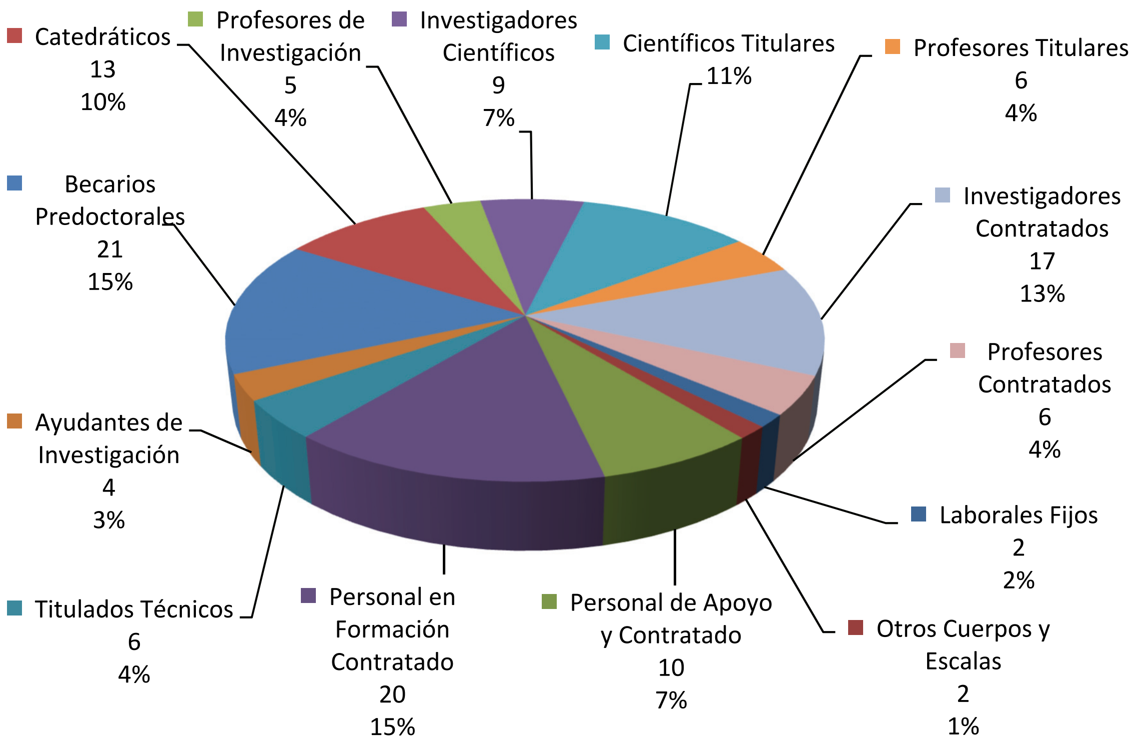
The current Strategic Plan 2010-2013 organizes our Center in 5 lines of research, which related with disciplines of solid state chemistry and physic, as well as materials engineering subjects, are intended to cover the most modern and innovative aspects of the current material science. Our basic commitment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

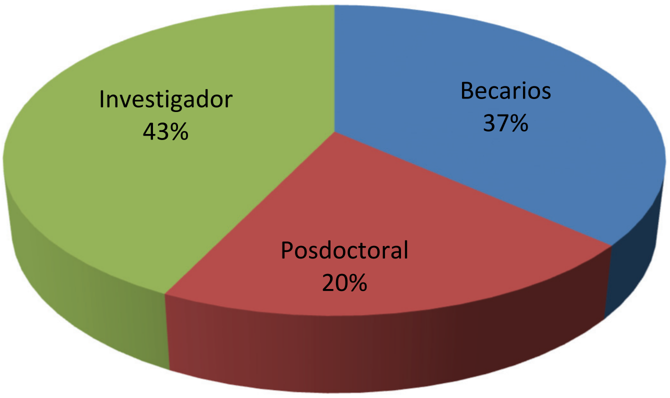
Dr. **Alfonso Caballero Martínez**
Director del Instituto de Ciencia de Materiales de Sevilla

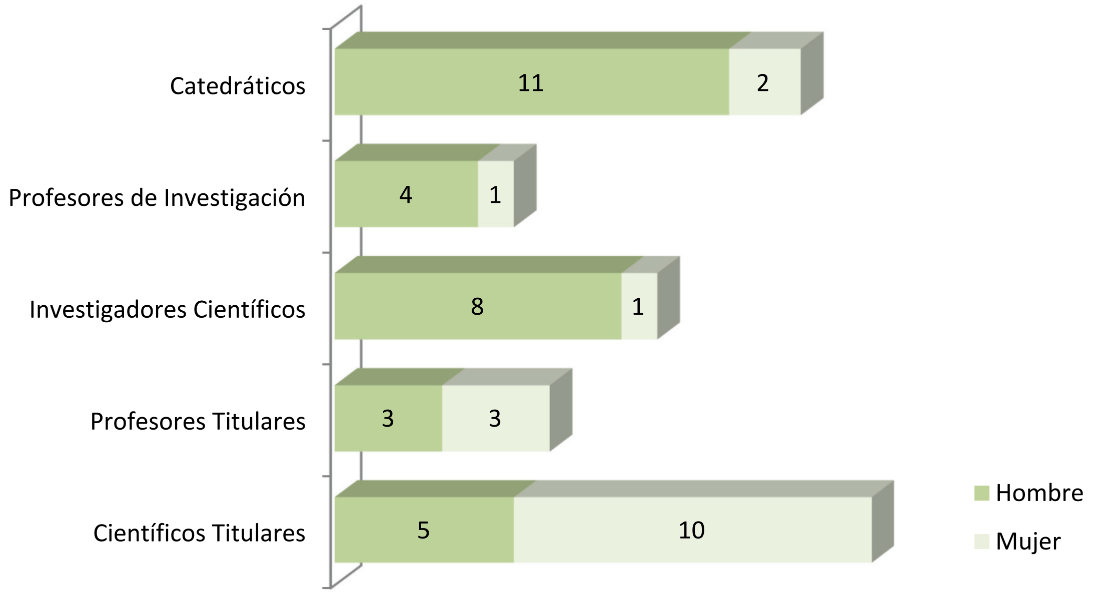
Datos Estadísticos del ICMS
Statistical Data of ICMS

RECURSOS HUMANOS / HUMAN RESOURCES

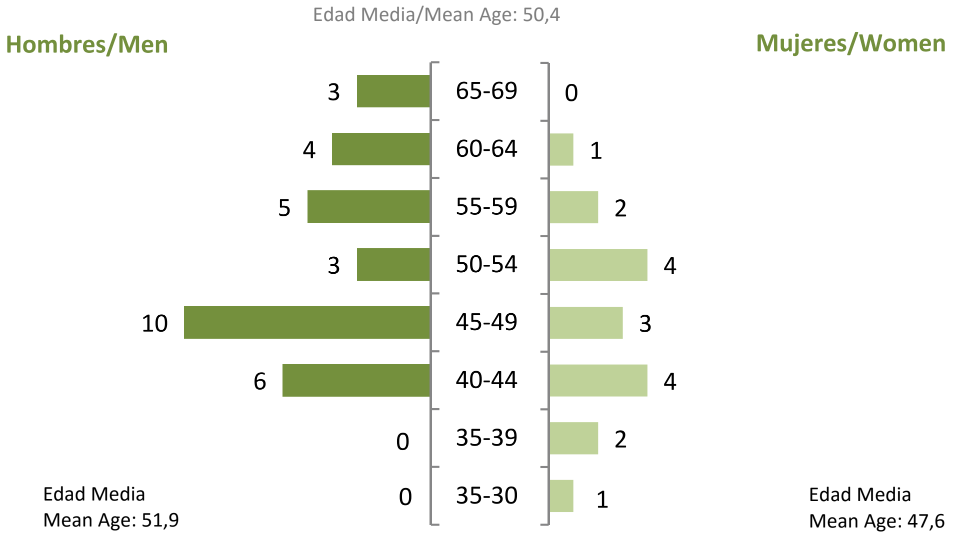
Distribución del personal por categorías
Distribution of by cathegories





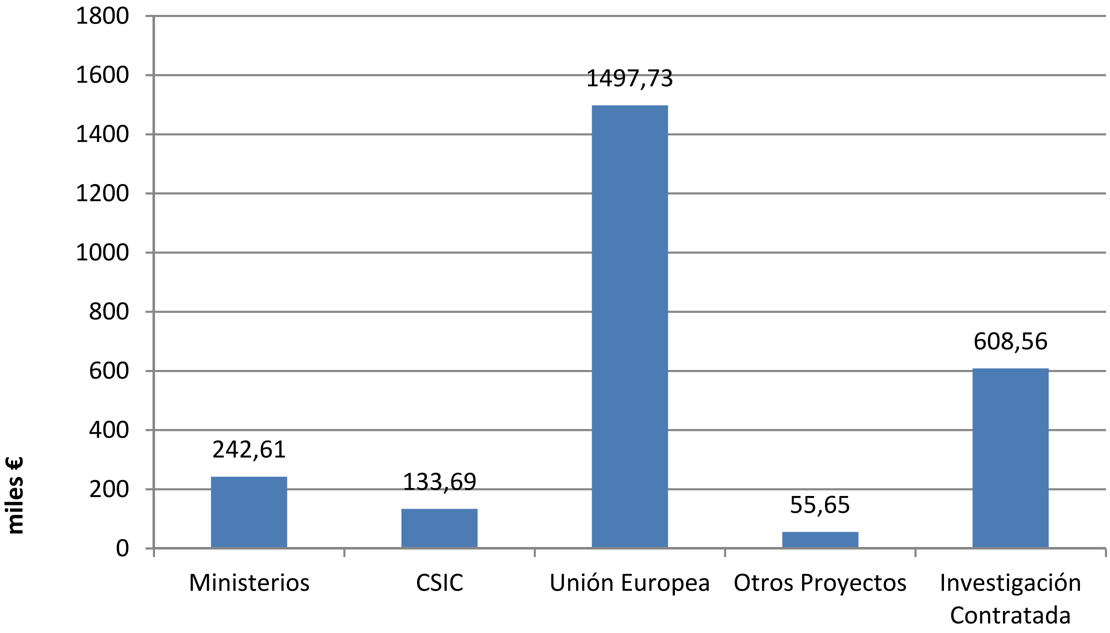


Distribución del personal investigador por edad
Scientific staff by age

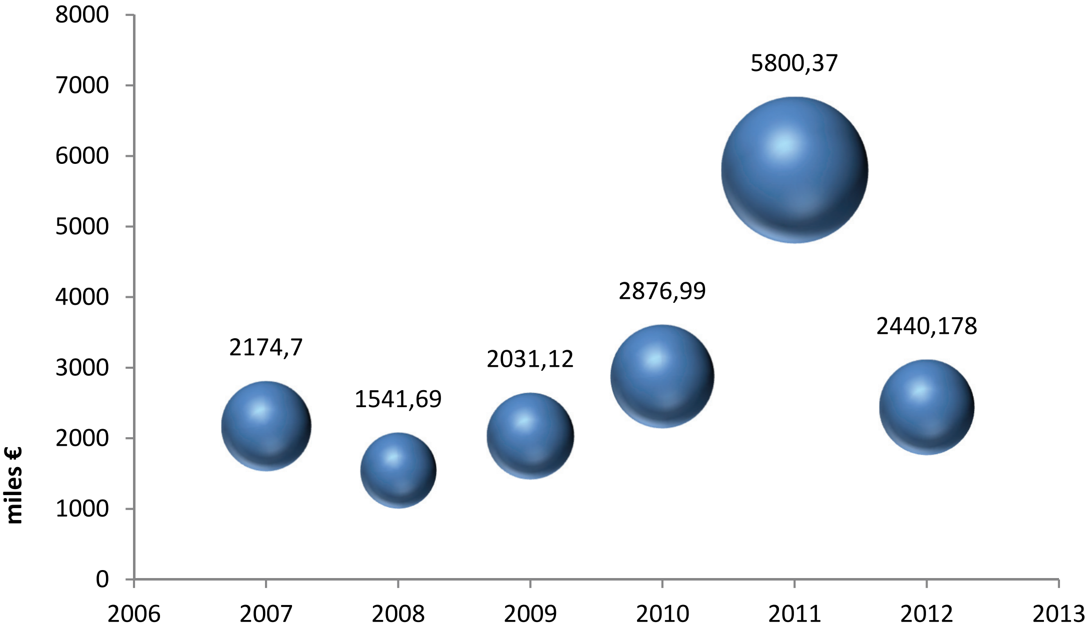


FINANCIACIÓN / FUNDING

Financiación del ICMS por Organismo Financiador 2012 (Sede-cicCartuja)
Funding of ICMS classified by source 2012 (Sede-cicCartuja)



Evolución 2007-2012 de la Financiación del ICMS (PCO)
Evolution of the funding of the ICMS in the period 2007-2012 OK



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 REVIEWS	1	40,197
NATURE MATERIALS	1	32,841
NANO LETTERS	1	13,198
ADVANCED FUNCTIONAL MATERIALS	1	10,179
ENERGY & ENVIRONMENTAL SCIENCE	1	9,610
TRAC-TRENDS IN ANALYTICAL CHEMISTRY	1	6,273
JOURNAL OF PHYSICAL CHEMISTRY LETTERS	1	6,213
CHEMICAL COMMUNICATIONS	2	6,169
JOURNAL OF MATERIALS CHEMISTRY	2	5,968
CHEMISTRY-A EUROPEAN JOURNAL	1	5,925
APPLIED CATALYSIS B-ENVIRONMENTAL	9	5,625
ENVIRONMENT INTERNATIONAL	1	5,297
ENVIRONMENTAL SCIENCE & TECHNOLOGY	1	5,228
CHEMCATCHEM	1	5,207
ACTA BIOMATERIALIA	1	4,865
JOURNAL OF PHYSICAL CHEMISTRY C	11	4,822
CRYSTAL GROWTH & DESIGN	1	4,720
INORGANIC CHEMISTRY	1	4,601
SOLAR ENERGY MATERIALS AND SOLAR CELLS	1	4,542
ACS APPLIED MATERIALS & INTERFACES	2	4,525
LANGMUIR	4	4,186
INTERNATIONAL JOURNAL OF HYDROGEN ENERGY	1	4,054
NANOTECHNOLOGY	2	3,979
APPLIED CATALYSIS A-GENERAL	6	3,903
APPLIED PHYSICS LETTERS	1	3,844
ELECTROCHIMICA ACTA	2	3,832
TALANTA	1	3,794
JOURNAL OF PHYSICAL CHEMISTRY B	1	3,696
PHYSICAL CHEMISTRY CHEMICAL PHYSICS	1	3,573
ULTRASONICS SONOCHEMISTRY	1	3,567
CHEMICAL ENGINEERING JOURNAL	1	3,461
CATALYSIS TODAY	3	3,407

REVISTA **JOURNAL**	ARTÍCULOS PAPERS	FACTOR DE IMPACTO IMPACT FACTOR (*)
MICROPOROUS AND MESOPOROUS MATERIALS	2	3,285
CHEMOSPHERE	1	3,206
JOURNAL OF COLLOID AND INTERFACE SCIENCE	1	3,070
MICROSCOPY AND MICROANALYSIS	2	3,007
CATALYSIS COMMUNICATIONS	1	2,986
JOURNAL OF MOLECULAR CATALYSIS A-CHEMICAL	2	2,947
FUEL PROCESSING TECHNOLOGY	2	2,945
JOURNAL OF THE MECHANICAL BEHAVIOR OF BIOMEDICAL MATERIALS	1	2,814
SOLID STATE IONICS	2	2,646
CHEMISTRY AND PHYSICS OF LIPIDS	1	2,571
JOURNAL OF PHYSICS D-APPLIED PHYSICS	1	2,544
APPLIED CLAY SCIENCE	2	2,474
JOURNAL OF PHOTOCHEMISTRY AND PHOTOBIOLOGY A-CHEMISTRY	1	2,421
PHYSICS OF THE EARTH AND PLANETARY INTERIORS	1	2,319
JOURNAL OF THE EUROPEAN CERAMIC SOCIETY	2	2,312
MATERIALS LETTERS	1	2,307
JOURNAL OF ALLOYS AND COMPOUNDS	3	2,289
JOURNAL OF THE AMERICAN CERAMIC SOCIETY	2	2,272
AICHE JOURNAL	1	2,261
CATALYSIS LETTERS	1	2,242
AMERICAN MINERALOGIST	2	2,169
JOURNAL OF APPLIED PHYSICS	2	2,160
JOURNAL OF SOLID STATE CHEMISTRY	2	2,159
JOURNAL OF BIOMEDICAL MATERIALS RESEARCH PART A	1	2,147
MATERIALS RESEARCH BULLETIN	1	2,105
RHEOLOGICA ACTA	1	2,027
JOURNAL OF MATERIALS SCIENCE	1	2,015
MATERIALS SCIENCE AND ENGINEERING A-STRUCTURAL MATERIALS PROPERTIES MICROSTRUCTURE AND PROCESSING	4	2,003
SURFACE SCIENCE	1	1,994
SURFACE & COATINGS TECHNOLOGY	4	1,867
THERMOCHIMICA ACTA	1	1,805
JOURNAL OF MAGNETISM AND MAGNETIC MATERIALS	1	1,780
INTERNATIONAL JOURNAL OF PHOTOENERGY	1	1,769

REVISTA JOURNAL	ARTÍCULOS PAPERS	FACTOR DE IMPACTO IMPACT FACTOR (*)
CERAMICS INTERNATIONAL	2	1,751
INTERNATIONAL JOURNAL OF REFRACTORY METALS & HARD MATERIALS	2	1,693
CERAMICS INTERNATIONAL	2	1,751
JOURNAL OF THERMAL ANALYSIS AND CALORIMETRY	1	1,604
POLYMER TESTING	1	1,600
JOURNAL OF POLYMER SCIENCE PART B-POLYMER PHYSICS	1	1,531
MICRON	1	1,527
CHEMIE DER ERDE-GEOCHEMISTRY	1	1,447
NUCLEAR INSTRUMENTS & METHODS IN PHYSICS RESEARCH SECTION B-BEAM INTERACTIONS WITH MATERIALS AND ATOMS	1	1,211
SURFACE AND INTERFACE ANALYSIS	1	1,180
JOURNAL OF MATERIALS SCIENCE-MATERIALS IN ELECTRONICS	1	1,076
JOURNAL OF ADVANCED OXIDATION TECHNOLOGIES	1	0,806
PHYSICS OF THE SOLID STATE	2	0,711
JOURNAL OF NANO RESEARCH	1	0,630
ACTA PHYSICA POLONICA A	1	0,444
STUDIES IN CONSERVATION	1	0,400
INTERNATIONAL JOURNAL OF ARCHITECTURAL HERITAGE	1	0,235
REVISTA DE METALURGIA	1	0,202
CHEMPLUSCHEM	1	---

(*) Factor de Impacto correspondiente al año 2011
Journal Citation Reports of 2011

COMPOSICIÓN Y ESTRUCTURA
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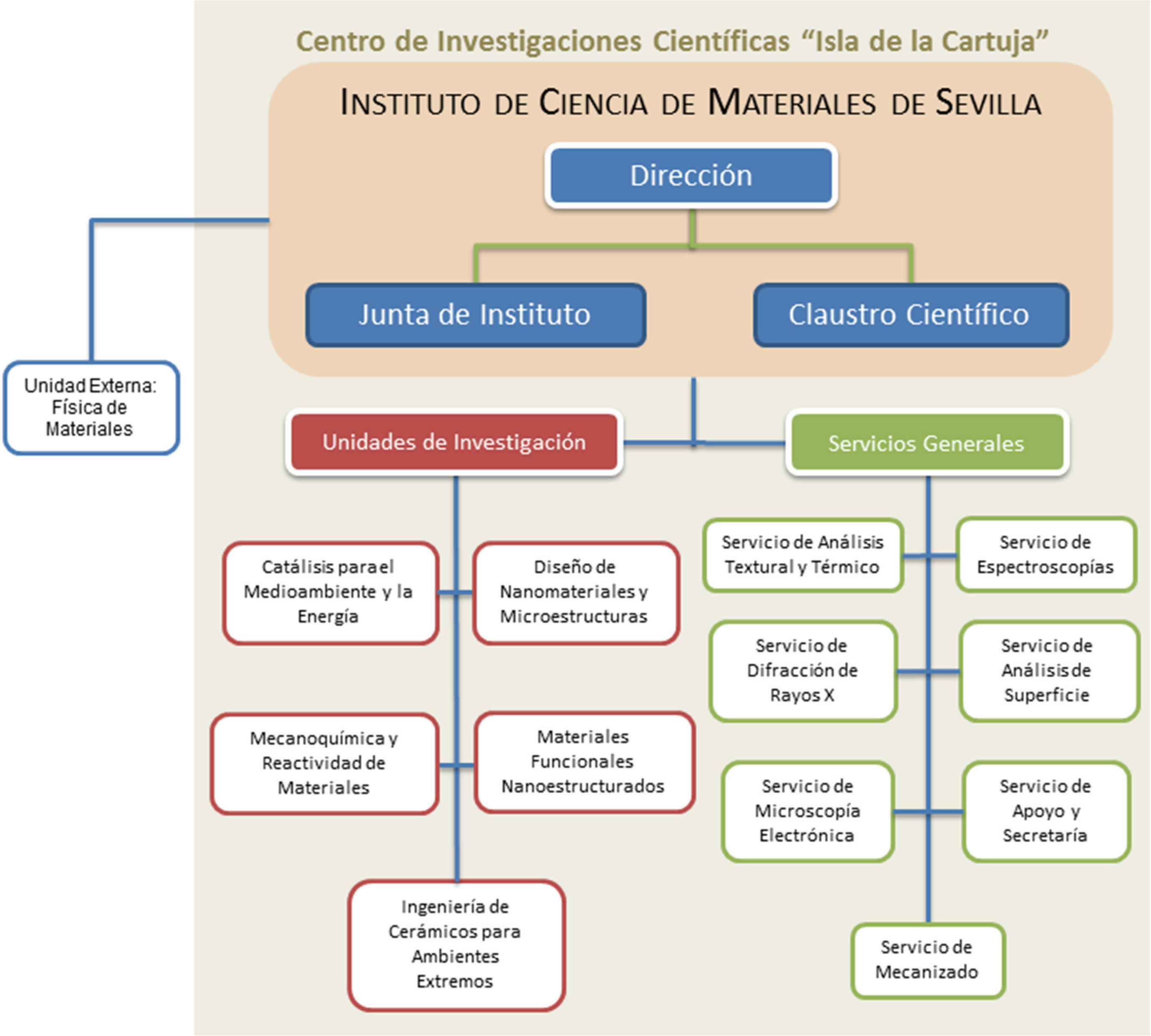
ORGANIGRAMA / ORGANIZATION CHART



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Lda. Olga Montes Amorín (cicCartuja)
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Visitas Institucionales y Prevención de Riesgos Laborales
Dr. Antonio Ruiz Conde

Unidad “Mecanoquímica y Reactividad de Materiales”
Lda. Belinda Sigüenza Carballo

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Dr. Victor J. Rico Gavira

Transferencia del Conocimiento (JAE Transfer)
Dra. Ana García Navarro
(Vicepresidencia Adjunta de Transferencia del Conocimiento del CSIC)

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

Personal / Personnel:

Catedrático

Dr. Alejandro Conde Amiano
Dra. Clara F. Conde Amiano
Dr. Jaime del Cerro González
Dr. Alberto Criado Vega
Dr. Arturo Domínguez Rodríguez
Dr. Victorino Franco García (desde 25 junio)
Dr. Antonio Muñoz Bernabé
Dr. Saturio Ramos Vicente

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 (desde 13 febrero)

UNIDADES DE INVESTIGACIÓN
RESEARCH UNITS

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

PROYECTOS DE INVESTIGACIÓN / RESEARCH PROJECTS

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MINISTERIO DE ECONOMÍA Y COMPETITIVIDAD

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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Materiales foto-activos para el desarrollo de la energía solar en procesos de interés medioambiental Photo-active materials to exploit solar energy for photocatalytic processes of environmental interest

Código/Code:	CTQ2011-26617-C03-02
Periodo/Period:	1-01-2012 / 31-12-2012
Organismo Financiador/Financial source:	Otros Programas del Plan Nacional I+D. Ministerio de Economía y Competitividad
Importe total/Total amount:	14.520 €
Investigador responsable/Research head:	José Antonio Navío Santos
Componentes/Research group:	Mª del Carmen Hidalgo López, Manuel Macías Azaña, Julie J. Murcia Mesa, Sebastián Murcia López

RESUMEN / ABSTRACT

La fotocatálisis heterogenea ha mostrado sobradamente su potencial para destoxificación y desinfección de medios acuosos y gaseosos. Sin embargo, su desarrollo tecnológico ha sido muy limitado debido a una serie de dificultades que pueden agruparse en dos grupos principales:

1. Dificultades para separar el catalizador del medio al finalizar el proceso, para su reactivación y reutilización.

2. Bajo rendimiento del proceso, que sólo aprovecha un porcentaje muy pequeño de los fotones útiles, siendo estos además una pequeña parte del espectro natural.

En nuestra propuesta se coordinan tres subproyectos liderados por tres grupos que aunan amplia experiencia en: Síntesis, modificación y caracterización de materiales fotocatalíticos (principalmente el grupo de la Universidad de Sevilla), Preparación y caracterización de óxidos metálicos en lámina delgada sobre diferentes substratos(principalmente el grupo del CIEMAT) y Modificación, caracterización espectroscópica de centros activos y estudios de fotorreactividad en fase acuosa y gaseosa (principalmente el grupo de la ULPGC).

Sobre la base de esta experiencia acumulada y de las principales tendencias en el desarrollo de la fotocatálisis heterogénea, nuestro consorcio se plantea como objetivo central de este proyecto: Sintetizar materiales basados en TiO_2, SnO_2, ZnO y materiales ternarios tipo titanato de bismuto (BITs), en forma de polvo con tamaño manométrico con alta actividad fotocatalítica y su fijación sobre sustratos adecuados (vidrio, membranas, láminas metálicas, etc.) recubiertos con películas delgadas de diferentes óxidos metálicos que faciliten el anclaje de estas partículas y/o actúen como semilla para la formación o cristalización de estas partículas, para poder utilizar estos sistemas de forma eficiente en procesos de descontaminación fotocatalítica en fase acuosa y gaseosa.

The heterogeneous photocatalysis has extensively shown its potential for detoxification and disinfection of aqueous and gaseous media. However, technological development has been very limited due to a number of difficulties that can be grouped into two main groups:

1. Difficulties inseparating the catalyst from the reacting medium for recovery and re-use once completed the process.

2. Poor performance of the process, which uses only a very small percentage of photons useful, and these are only a small part of the natural spectrum.

Our proposal is composed by three sub-projects led by three groups which combine extensive experience in: Synthesis, modification and characterization of photocatalytic materials (mainly the group at the University of Seville), Preparation and characterization of metal oxide thin films on different substrates (mainly the group from CIEMAT) and Modification, spectroscopic characterization of active centers and photoreactivity studies in aqueous and gaseous phase (mainly the group from the ULPGC).

Based onthe experience gained and the main trends in the development of heterogeneous photocatalysis, our consortium proposal has a central objective: Synthesize materials based on TiO_2, SnO_2, ZnO and ternary materials such as bismuth titanate (BITs), in powder form with nanometric size, with high photocatalytic activity and its support on suitable substrates (glass, membrane, metal sheets, etc.) coated with thin films of different metal oxides to facilitate the fixation of powder particles and/or acting as seed for the formation or crystallization of these particles in order to use these systems efficiently in photocatalytic detoxification processes in aqueous phase and gas phase.

Integración de reactores catalíticos de microcanales para la producción de hidrógeno a partir de alcoholes
Integration of microchannel catalytic reactors for hydrogen production from alcohols

Código/Code:	ENE2009-14522-C05-01 (subprograma CON)
Periodo/Period:	1-08-2009 / 31-07-2012
Organismo Financiador/Financial source:	Programa Nacional de Proyectos de Investigación Fundamental, Subprograma de Proyectos de Investigación Fundamental no Orientada (Ministerio de Economía y Competitividad)
Importe total/Total amount:	314.600 €
Investigador responsable/Research head:	José Antonio Odriozola Gordón
Componentes/Research group:	M.A. Centeno, S. Ivanova, F. Romero Sarria, M.I. Domínguez, Sandra Palma, Oscar Laguna, Ana Penkova, Sylvia Cruz, W.Yesid Hernández, Luis Bobadilla

RESUMEN / ABSTRACT

El uso masivo y generalizado de dispositivos eléctricos y electrónicos portátiles aumenta la necesidad de fuentes de potencia autónomas y eficientes, de hasta unos 50 We, capaces de reemplazar la tecnología actual basada en el uso de baterías. El uso de combustibles o productos químicos convencionales, hidrocarburos o alcoholes por ejemplo, es una alternativa prometedora cuando se combina con los recientes desarrollos en intensificación de procesos basados en la tecnología de reactores de microcanales.

El desarrollo de la tecnología de microcanales para la producción de hidrógeno, in situ y a demanda, a partir de alcoholes, se comenzó a estudiar en el proyecto anterior (MAT2006-12386-C05). Este estudio permitió la construcción de reactores de microcanales para las reacciones de reformado catalítico de metanol y oxidación preferente de CO (PROX).

En el presente proyecto se pretenden aplicar los conocimientos adquiridos para acoplar los microrreactores entre sí integrando flujos térmicos y materiales, escalarlos, y unirlos a una celda de combustible comercial de 50 We (PEMFC). En paralelo, se desarrollarán reactores de microcanales para el reformado catalítico de etanol y la reacción de desplazamiento del gas de agua (WGS) lo que permite aumentar la versatilidad el dispositivo diseñado.

La viabilidad de estas fuentes de potencia autónomas requiere el estudio no sólo de la fabricación, escalado de los microreactores e integración de los flujos térmicos y materiales sino también explorar el uso de materiales de mayor disponibilidad (aceros ferríticos adaptados al uso), su durabilidad (aceros, catalizadores, soldaduras, juntas, …) y el desarrollo de un algoritmo de control para el conjunto formado por el procesador de combustible (reformado + eliminación de CO) y la pila de combustible.

The widespread use of portable electric and electronic devices increases the need for efficient autonomous power supplies (up to 50 We) that replace the currently predominant battery technology. The use of common fuels/chemicals, such as hydrocarbons or alcohols, as

an energy source is a promising alternative when combined with the recent developments in microchannel reactor technology.

In the previous project (MAT2006-12386-C05) we began to explore the use of microchannel reactor technology to generate hydrogen on site and on demand by processing alcohols which has allowed the manufacturing of microreactors for the catalytic steam reforming of methanol and CO preferential oxidation (PROX) reactions.

In the present project, the main focus is set on the scaling up of the already designed microreactors which will allow the fueling of a 50 We commercial fuel cell (PEMFC) and the integration of both, the material and thermal flows generated in the fuel processor and the fuel cell, including the production and cleaning steps required by the PEMFC. In addition to this, the development of microreactors for the catalytic steam reforming of ethanol and the water-gas-shift (WGS) reactions is considered in this project for increasing the versatility of the designed device.

The feasibility of this kind of autonomous power supplies would require the study of the manufacturing, scaling up of the microreactors and material and thermal flows integration, but also to explore the use of easily available materials (new steels adapted to use), the ageing behaviour of devices (steel, catalysts, sealings, …) and the development of a control algorithm of the fuel processor/fuel cell system

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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 fragances 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 basa-

dos 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 fragrances fields, such as the production of l-carvone from catalytic oxidation of d-limonene.

Reformado Catalítico de Glicerina
Catalytic reforming of glycerol

Código/Code:	P09-TEP-5454 (Proyecto de Excelencia)
Periodo/Period:	01-01-2010 / 31-12-2012
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	284.545,68 €
Investigador responsable/Research head:	José Antonio Odriozola Gordón
Componentes/Research group:	Luis F. Bobadilla Baladrón, Sylvia A. Cruz Torres, M. Isabel Domínguez Leal, Anna D. Penkova, Francisca Romero Sarria, Andrea Alvarez Moreno

RESUMEN / ABSTRACT

Este proyecto pretende la producción de hidrógeno a partir del reformado de glicerina. La glicerina es el producto secundario principal en la producción de bio-diesel a través de la transesterificación de ácidos grasos. Si tenemos en cuenta el desarrollo actual, la producción de bio-combustibles se estima en 9.9 Mtoe para 2010, lo que representa el 50% de los objetivos de la Unión Europea.

Los sistemas de energía actuales necesita el desarrollo de modelos energéticos alternativos. El empleo de hidrógeno como vector energético representa una de esas alternativas, aunque para asegurar la sostenibilidad se requiere que el hidrógeno se produzca a partir de fuentes renovables. La principal ventaja del planteamiento que proponemos, aprovechamiento de la glicerina, reside en que además de sostenible el balance de carbono es prácticamente neutro. Además, su valorización debe conducir a aumentar la rentabilidad de las bio-refinerías que de otro modo se verían afectadas por el incremento de costes asociados a la eliminación de este producto

The main objective of this Project is the production of Hydrogen from glicerol steam reforming. Glycerol is the most important by-product of the biodiesel production from the transterification of fatty acids. In the year 2010, the estimated production of biofuels was about 9.9 millions of tonnes, which represents 50% of the aims of the European Union objectives. The current energy system needs the development of alternative energetic models. The use of hydrogen as energetic vector is one of these alternatives, but, to assure the sustainability, its production must be from renewable sources. Among the possible renewable sources of hydrogen, the main advantage of the use of glycerol is the almost neutral carbon balance. In addition, the glycerol valorisation must lead to increase the profitability of the bio-refineries that, differently, would meet affected by the increase of costs associated with the elimination of this product.

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:	M. 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.

OTROS PROYECTOS / OTHER PROJECTS

Desarrollo de nuevos catalizadores a partir de fosfatos naturales marroquíes para las reacciones de oxidación de alcoholes y producción de hidrógeno

Código/Code:	C/030491/10
Periodo/Period:	27-01-2011 / 27-01-2012
Organismo Financiador/Financial source:	Otros programas de la Agencia Española de Cooperación Internacional (AECI), Ministerio de Asuntos Exteriores y Cooperación
Importe total/Total amount:	11.000 €
Investigador responsable/Research head:	Alfonso Caballero Martínez

Valorización de fosfatos naturales marroquíes en aplicaciones catalíticas energéticas y medioambientales

Código/Code:	A1/035614/11
Periodo/Period:	13-12-2011 / 12-12-2012
Organismo Financiador/Financial source:	Otros programas de la Agencia Española de Cooperación Internacional (AECI), Ministerio de Asuntos Exteriores y Cooperación
Importe total/Total amount:	55.650 €
Investigador responsable/Research head:	Alfonso Caballero Martínez

Cooperación Internacional y Otros
International Cooperation and Others

Design and characterization of gold catalysts for pure hydrogen production for fuel cell application

Código/Code:	2009BG0024
Periodo/Period:	01-01-2011 / 31-12-2012
Organismo Financiador/Financial source:	Proyecto conjunto con instituciones extranjeras CSIC-Academia de Ciencias Búlgara
Importe total/Total amount:	3.378,5 €
Investigador responsable/Research head:	José Antonio Odriozola Gordón
Componentes/Research group:	Svetlana Ivanova, Francisca Romero Sarria, Miguel Angel Centeno Gallego, Tomás Ramírez Reina

Reformado catalítico de glicerol: Estudio IR de la influencia de las propiedades de los sólidos sobre el mecanismo de reacción

Código/Code:	FR2009-0105
Periodo/Period:	01-01-2010 / 31-12-2012
Organismo Financiador/Financial source:	Acciones Integradas con Francia
Importe total/Total amount:	3.300 €
Investigador responsable/Research head:	Francisca Romero Sarria
Componentes/Research group:	José Antonio Odriozola Gordón, Miguel Angel Centeno Gallego, Svetlana Ivanova, Francisca Romero Sarria, Leidy Marcela Martínez Tejada, Luis Bobadilla Baladrón

TRANSFERENCIA / TECHNOLOGICAL TRANSFER

PATENTES / PATENTS

Catalizadores de oro y uso del mismo para la reacción de desplazamiento del gas de agua
Inventores: J.A. Odriozola, T. Ramírez Reina, M.A. Centeno, S. Ivanova, V. Idakiev, T. Tabakova, L.F. Bobadilla, F. Romero Sarria
Tipo de Patente: Internacional
Número de presentación: 300073297
Solicitud número PCT: PCT/ES2012/070717
Fecha Solicitud: 17 Octubre 2012
Entidad/es Titular/es: Universidad de Sevilla y CSIC

CONVENIOS Y CONTRATOS / CONTRACTS AND AGREEMENTS

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

Oxidación selectiva de diferentes terpenos de interés en química de fragancias

Código/Code:	020165110009
Periodo/Period:	09-05-2011 / 09-05-2012
Organismo Financiador/Financial source:	Destilaciones Bordas Chinchurreta, SA
Importe total/Total amount:	38.940 €
Investigador responsable/Research head:	Juan Pedro Holgado Vázquez

PUBLICACIONES / PUBLICATIONS

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

Advanced nanoarchitectures for solar photocatalytic applications
Kubacka, A; Fernandez-Garcia, M; Colon, G
Chemical Reviews, **112** (2012) 1555-1614
March, 2012 | **DOI**: 10.1021/cr100454n

Advanced nanostructured materials that demonstrate useful activity under solar excitation in fields concerned with the elimination of pollutants, partial oxidation and the valorization of chemical compounds, water splitting and CO_2 reduction processes, are discussed. Point defects present in nanoparticulated anatase present both 5-fold- and 6-fold-coordinated titanium atoms, as well as 2-fold- and 3-fold-coordinated oxygens. The requirement of using sunlight as the excitation source for the degradation reaction demands, as a principal requirement, the modification of the electronic characteristics of a UV absorber system such as anatase-TiO_2. Some reports also indicate the need for large doping concentrations for N-doping in specific cases where notable changes in the valence band onset are subsequently observed. The effect of cetyltrimethylammonium bromide (CTAB) on the crystallization is reported by Yin et al. They showed that the presence of CTAB induces the appearance of BiOBr during the synthesis at 80°C using an aqueous method.

Evidence of upconversion luminescence contribution to the improved photoactivity of erbium doped TiO_2 systems
Obregon, S; Colon, G
Chemical Communications, **48** (2012) 7865-7867
August, 2012 | **DOI**: 10.1039/C2CC33391K

Er^{3+}–TiO_2 synthesized by a surfactant free hydrothermal method exhibits good photoactivities under sun-like excitation for the degradation of phenol. The presence of Er^{3+} does not affect the structural and morphological features of the TiO_2 significantly. The best photocatalytic performance was attained for the samples with 2 wt% of Er. Different photocatalytic runs indicated that the incorporation of the Er^{3+} cation would be responsible for the enhanced photocatalytic activity, which participates in different mechanisms under UV and NIR excitation.

Hydrothermal synthesis of $BiVO_4$: Structural and morphological influence on the photocatalytic activity
Obregon, S; Caballero, A; Colon, G
Applied Catalysis B-Environmental, **117** (2012) 59-66
May, 2012 | **DOI:** 10.1016/j.apcatb.2011.12.037

$BiVO_4$ hierarchical heterostructures are synthesized by means of a surfactant free hydrothermal method having good photoactivities for the degradation of methylene blue under UV-vis irradiation. From the structural and morphological characterization it has been stated that $BiVO_4$ present the monoclinic crystalline phase with different morphologies depending on the pH value, type of precipitating agent and hydrothermal temperature and treatment time. The best photocatalytic performance was attained for the samples with needle-like morphology.

Effect of the active metals on the selective H-2 production in glycerol steam reforming
Araque, M; Martinez, LM; Vargas, JC; Centeno, MA; Roger, AC
Applied Catalysis B-Environmental, **125** (2012) 556-566
August, 2012 | **DOI:** 10.1016/j.apcatb.2012.06.028

The production of hydrogen by glycerol steam reforming was studied using CeZr(Co, CoRh) catalysts. The effect of Co and Rh presence on the properties of the mixed oxides and the effect on the catalytic behavior were considered. The catalysts were characterized before and after testing by XRD, Raman, TPR, H_2-TPD, TPD-TPO and HRTEM. It was observed that the presence of Co allowed the selective H_2 production related with the presence of a metallic phase at the beginning of the reaction. The presence of Rh favored even more the H_2 production and also increased the stability of the catalyst. For CeZrCoRh, the presence of both metals enhanced the catalyst reduction capacity, a characteristic that significantly improved the catalytic behavior for glycerol steam reforming. The selective H_2 production was related to the capacity of the catalyst to activate H_2O under the reaction conditions. The progressive loss of this capacity decreases the production of H_2, and glycerol decomposition is actually favored over glycerol steam reforming. According to the initial distribution of products, and its evolution with time on stream, two main reaction pathways were proposed.

Effect of deposition of silver on structural characteristics and photoactivity of TiO2-based photocatalysts
Melian, EP; Diaz, OG; Rodriguez, JMD; Colon, G; Navio, JA; Macias, M; Pena, JP
Applied Catalysis B-Environmental, **127** (2012) 112-120
October, 2012 | **DOI:** 10.1016/j.apcatb.2012.08.007

The homemade bare TiO_2 photocatalyst obtained in a previous work was modified with nanosized silver particles by liquid impregnation and photodeposition methods to obtain different noble metal loadings (0.3–1 at.%). Characterization of the synthesized photocatalysts was carried out by the BET method, XPS, TEM, SEM-EDX, XRD and diffuse reflectance measurements. Photocatalytic activity of these silver-deposited TiO_2 nanoparticles was tested by photocatalytic degradation of phenol as a reference model representing phenolic pollutants. The noble metal content on the TiO_2 surface affected the efficiency of the photocatalytic process, and the photocatalytic activity of noble metal-modified TiO_2 was considerably better than that of bare TiO_2. Phenol decomposition rate was higher with TiO_2 modified by the liquid impregnation method than with TiO_2 modified by the photodeposition method.

Insights towards the influence of Pt features on the photocatalytic activity improvement of TiO_2 by platinisation

Murcia, JJ; Navio, JA; Hidalgo, MC
Applied Catalysis B-Environmental, **126** (2012) 76-85
September, 2012 | **DOI:** 10.1016/j.apcatb.2012.07.013

The influence of Pt features, such as particle size, dispersion, oxidation state and amount of metal, on the improvement of the photoactivity of TiO2 for phenol and methyl orange degradation was studied.

The size of Pt deposits was precisely controlled by changing deposition time under medium light intensity during the photodeposition, with sizes ranging from 3 to 6 nm. Pt oxidation state was also strongly dependent on the photodeposition time.

Photocatalytic activity results showed that the fraction of metallic platinum (Pt0) was the crucial factor for the improvement of the activity. When the fraction of Pt0 was similar, metal deposit size became the dominant parameter influencing the activity.

The influence of the substrate to be degraded (phenol or methyl orange) was also studied.

Selective photooxidation of alcohols as test reaction for photocatalytic activity

Lopez-Tenllado, F. J.; Marinas, A.; Urbano, F. J.; Colmenares, J. C.; Hidalgo, M. C.; Marinas, J. M.; Moreno, J. M.
Applied Catalysis B-Environmental, **128** (2012) 150-158
November, 2012 | **DOI:** 10.1016/j.apcatb.2012.02.015

Twenty-four different titania-based systems synthesized through the sol–gel process varying the precursor (titanium isopropoxide or tetrachloride) and/or the ageing conditions (magnetic stirring, ultrasounds, microwave or reflux) were tested for liquid-phase selective photooxidation of 2-butenol (crotyl alcohol) to 2-butenal (crotonaldehyde) and gas-phase selective photooxidation of 2-propanol to acetone. To the best of our knowledge, the former process is suggested for the first time as test reaction for photocatalytic activity. Interestingly, both test reactions (despite having very different reactant/catalyst ratio and contact times) showed quite similar results in terms of influence of the precursor (titanium isopropoxide leading to better results than titanium tetrachloride) and the metals (the presence of iron, palladium or zinc being detrimental to activity whereas zirconium and especially gold improved the results as compared to pure titania). To our mind, these results give validity to both processes as test reactions for a fast screening of catalysts for photocatalytic tranformations. Finally, some gold-containing solids even improved photocatalytic activity of Degussa P25.

Cu-modified cryptomelane oxide as active catalyst for CO oxidation reactions

Hernandez, Willinton Y.; Centeno, Miguel A.; Ivanova, Svetlana; Eloy, Pierre; Gaigneaux, Eric M.; Odriozola, Jose A.
Applied Catalysis B-Environmental, **123-124** (2012) 27-35
July, 2012 | **DOI:** 10.1016/j.apcatb.2012.04.024

Manganese oxide octahedral molecular sieves (cryptomelane structure) were synthesized by a solvent-free method and tested in the total oxidation of CO (TOX), and preferential oxidation of CO in presence of hydrogen (PROX). The influence of Cu in the cryptomelane structure was evaluated by several characterization techniques such as: X-ray fluorescence (XRF), thermogravimetric analysis (TGA), hydrogen temperature programmed reduction (TPR-H_2) and X-ray photoelectron spectroscopy (XPS). The Cu-modified manganese oxide material (OMS-Cu) showed very high catalytic activity for CO oxidation in comparison to the bare manganese oxide octahedral molecular sieve (OMS). The improved catalytic activity observed in OMS-Cu catalyst was associated to a high lattice oxygen mobility and availability due to the formation of Cusingle bondMnsingle bondO bridges. In addition, under PROX reaction conditions the catalytic activity considerably decreases in the presence of 10% (v/v) CO_2 in the feed while the same amount of water provokes an improvement in the CO conversion and O_2 selectivity.

Influence of the shape of Ni catalysts in the glycerol steam reforming

Bobadilla, L. F.; Alvarez, A.; Dominguez, M. I.; Romero-Sarria, F.; Centeno, M. A.; Montes, M.; Odriozola, J. A.

Applied Catalysis B-Environmental, **123-124** (2012) 379-390

July, 2012 | **DOI:** 10.1016/j.apcatb.2012.05.004

Biomass is an alternative to replace the use of fossil fuels. Glycerol, a byproduct in the biodiesel production, can be used for obtaining hydrogen. The most efficient method for obtaining hydrogen from glycerol is the steam reforming (SR). So far all the published papers report the use of conventional catalyst. In this paper, a structured catalyst has been prepared and compared with the conventional ones (powder and spherical pellets). Results show that the structured catalyst (monolith) is more stable as formation of coke was not observed.

$LaNiO_3$ as a precursor of Ni/La_2O_3 for CO_2 reforming of CH_4: Effect of the presence of an amorphous NiO phase

Rosa Pereñiguez , Victor M. Gonzalez-delaCruz, Alfonso Caballero, Juan P. Holgado

Applied Catalysis B-Environmental, **123-124** (2012) 324-32

July, 2012 | **DOI:** 10.1016/j.apcatb.2012.04.044

The objective of the present work has been the study of the physico-chemical and catalytic properties of Ni/La_2O_3 catalysts obtained by reduction of four $LaNiO_3$ samples prepared by different methods. The $LaNiO_3$ precursors as well as the resulting Ni/La_2O_3 catalysts, were characterized by scanning electron microscopy (SEM), X-ray diffraction (XRD), X-ray absorption spectroscopy (XAS), temperature programmed reduction and oxidation (TPR, TPO). The catalytic performances of these systems for dry reforming of methane (DRM) were also tested. These samples show different physico-chemical properties resulting from the synthesis method used. The XAS and TPR measurements show that in all four $LaNiO_3$ samples there is, in addition of the crystalline $LaNiO_3$ rhombohedrical phase, a significant amount of an amorphous NiO phase, not detectable by XRD but evidenced by XAS. The amount of this NiO amorphous phase seems to play, together with some other microstructural parameters, an important role in the performance of the Ni/La_2O_3 samples for the DRM reaction.

Influence of Vanadium or Cobalt Oxides on the CO Oxidation Behavior of Au/MOx/CeO_2–Al_2O_3 Systems
Reina, TR; Moreno, AA; Ivanova, S; Odriozola, JA; Centeno, MA
ChemCatChem, **4** (2012) 512-520
April, 2012 | **DOI:** 10.1002/cctc.201100373

A series of V_2O_5- and Co_3O_4-modified ceria/alumina supports and their corresponding gold catalysts were synthesized and their catalytic activities evaluated in the CO oxidation reaction. V_2O_5-doped solids demonstrated a poor capacity to abate CO, even lower than that of the original ceria/alumina support, owing to the formation of $CeVO_4$. XRD, Raman spectroscopy, and H_2-temperature programmed reduction studies confirmed the presence of this stoichiometric compound, in which cerium was present as Ce^{3+} and its redox properties were avoided. Co_3O_4-doped supports showed a high activity in CO oxidation at subambient temperatures. The vanadium oxide-doped gold catalysts were not efficient because of gold particle agglomeration and $CeVO_4$ formation. However, the gold–cobalt oxide–ceria/alumina catalysts demonstrated a high capacity to abate CO at and below room temperature. Total conversion was achieved at −70 °C. The calculated apparent activation energy values revealed a theoretical optimum loading of a half-monolayer.

CO-Induced Morphology Changes in Zn-Modified Ceria: A FTIR Spectroscopic Study
Penkova, A; Laguna, OH; Centeno, MA; Odriozola, JA
Journal of Physical Chemistry C, **116** (2012) 5747-5756
March, 2012 | **DOI:** 10.1021/jp210996b

A FTIR study of the CO adsorption on a Zn-modified ceria is presented. The results indicate that at lower activation temperatures only Ce^{4+} carbonyls were detected, which were reduced with the increase of the activation temperature. At higher activation temperatures, up to three Zn^{2+} carbonyls were also identified according to the arrangement of the Zn^{2+} cations. The consecutive CO adsorptions demonstrated an irreversible modification of the surface, resulting in an agglomeration of the zinc cations. A stepwise conversion of the isolated Zn^{2+} carbonyls into carbonyls of the closely situated zinc cations followed by formation of bigger zinc oxide clusters was observed. The carbon monoxide coordinated on the isolated Zn^{2+} cations at the interface with ceria reacts with the lattice oxygen leading to formation of oxygen vacancies. An insight into the origin of the activation during the CO oxidation process is proposed.

In Situ XAS Study of Synergic Effects on Ni-Co/ZrO_2 Methane Reforming Catalysts
Gonzalez-delaCruz, VM; Pereniguez, R; Ternero, F; Holgado, JP; Caballero, A
Journal of Physical Chemistry C, **116** (2012) 2919-2926
February, 2012 | **DOI:** 10.1021/jp2092048

Four different mono and bimetallic Ni–Co/ZrO_2 catalysts have been studied by means of in situ XAS, X-ray diffraction, TPR, and measurements of the catalytic activity in the dry reforming reaction of methane (DRM). Even though the cobalt monometallic system has no activity for the methane reforming reaction, both bimetallic catalysts (with 1:1 and 1:2 Ni/Co ratio, respectively), showed a better activity and stability than the nickel monometallic system. The XRD data indicate that a mixed cobalt–nickel spinel is formed by calcination of the precursor solids, leading to the formation of an alloy of both metals after reduction in hydrogen. In situ XAS experiments showed a much better resistance of metals in the bimetallic systems to be oxidized under reaction conditions at temperatures until 750 °C. After these results, we proposed the formation in the bimetallic systems of a more reducible nickel–cobalt alloy phase, which remains completely metallic in contact with the CO_2/CH_4 reaction mixture at any temperature. The presence of adjacent nickel and cobalt sites seems to avoid the deactivation of cobalt in the DRM reaction. In the case of cobalt sites, the presence of adjacent nickel atoms seems to prevent the deposition of carbon over the cobalt sites, now showing its higher activity in the dry reforming reaction. Simultaneously, this higher activity of the cobalt sites in the bimetallic system produces more hydrogen as a product, maintaining the nickel atoms completely reduced under reaction conditions. This synergic effect accounts for the better performance of the bimetallic systems and points at both, the oxidation state of nickel particles under reaction conditions and the carbon deposition processes, as important factors responsible for differences in catalytic activities and stabilities in this hydrocarbon reaction.

Making Photo-selective TiO_2 Materials by Cation–Anion Codoping: From Structure and Electronic Properties to Photoactivity

Marquez, AM; Plata, JJ; Ortega, Y; Sanz, JF; Colon, G; Kubacka, A; Fernandez-Garcia, M
Journal of Physical Chemistry C, **116** (2012) 18759-18767
September, 2012 | **DOI:** 10.1021/jp3045143

Photoselective oxidation yielding high-added value chemicals appears as a green novel process with potential to be explored. In this study we combine spectroscopic XPS (N 1s and O 1s) and multiwavelength Raman data with density functional theory calculations to explore the structural and electronic properties of W,N-codoped TiO_2 anatase surfaces and interpret the outstanding photocatalytic properties of such a system in partial oxidation reactions. Theoretical calculations allow us to examine several substitutional and N-interstitial configurations at different concentrations of the W,N dopants (similar to those experimentally found), as well as their interaction with structural point defects: Ti cation vacant sites and surface wolframyl species that are required to compensate the extra charge of the W^{6+} and N-containing anions. The joint use of theoretical and experimental XPS and Raman tools renders key structural information of W,N-codoped microcrystalline TiO_2 solids. The incorporation of N at substitutional positions of anatase with the concomitant presence of W═O species introduces localized states in the band gap, a result that is critical in interpreting the chemical behavior of the solids. The combination of the electronic and geometric structural information leads to a simple mechanism that rationalizes the experimentally observed photoactivity and selectivity in partial oxidation reactions.

Sub-ambient CO oxidation over mesoporous Co_3O_4: Effect of morphology on its reduction behavior and catalytic performance

Alvarez, A; Ivanova, S; Centeno, MA; Odriozola, JA
Applied Catalysis A-General, **431** (2012) 9-17
July, 2012 | **DOI:** 10.1016/j.apcata.2012.04.006

The influence of the Co_3O_4 morphology on its redox behavior and catalytic performance in the CO oxidation reaction is studied. Three different Co_3O_4 morphologies were synthesized by precipitation and hydrothermal methods. TEM and SEM observations clearly show the different obtained morphologies: rods, wires and a mixture of plates and cubes. The textural properties depend on the morphology and the redox ones on the particle size. XRD analysis reveals a spinel structure in all solids with a preferential exposition of the [1 1 0] plane in the Co_3O_4 rods sample. This preferential exposition, along with its higher specific surface area provides the rods with more efficient oxygen storage capacity resulting in an excellent catalytic performance compared to the other two morphologies.

Photocatalytic activity of single and mixed nanosheet-like Bi_2WO_6 and TiO_2 for Rhodamine B degradation under sunlike and visible illumination

Murcia-Lopez, S; Hidalgo, MC; Navio, JA

Applied Catalysis A-General, **423-424** (2012) 34-41

May, 2012 | **DOI:** 10.1016/j.apcata.2012.02.016

The photocatalytic activity, under sunlike illumination, for Rhodamine B (RhB) degradation using Bi_2WO_6-TiO_2 samples, is reported. Two different kinds of Bi_2WO_6-TiO_2 samples were studied, obtained by distinct methods: first, a mechanical mixing, by adding to synthesized nanosheet-like Bi_2WO_6 powder the corresponding amount of TiO_2 nanoparticles (P25) in order to obtain physical mixtures of both catalysts with different percentages of TiO_2 (5, 10 and 50 wt%); second, a single Bi_2WO_6-TiO_2 heterostructure was prepared by adding commercial TiO_2-P25 to the Bi_2WO_6 precursors (50 wt%) prior to the hydrothermal treatment, thus obtaining a sample with "in situ" TiO_2 incorporation. Comparisons between the photocatalytic behaviour of these samples and those exhibited by the single materials Bi_2WO_6 and TiO_2 (P25) were carried out, in order to establish the effect not only of the TiO_2 addition but also of the way in which TiO_2 (P25) is incorporated. The role of each single photocatalyst in the mixtures in the RhB degradation and mineralization under sunlike and just visible illumination was also studied.

Gold supported cryptomelane-type manganese dioxide OMS-2 nanomaterials deposited on AISI 304 stainless steels monoliths for CO oxidation

Martinez, LM; Romero-Sarria, F; Hernandez, WY; Centeno, MA; Odriozola, JA

Applied Catalysis A-General, **423** (2012) 137-145

May, 2012 | **DOI:** 10.1016/j.apcata.2012.02.026

Gold supported on cryptomelane-type OMS-2 catalysts deposited on AISI 304 stainless steels monoliths have been prepared for the first time, characterised and tested in the CO oxidation reaction. An easy and non-conventional method of incorporation of gold to the cryptomelane solid is used. This method allows the preparation of the monolithic catalysts without altering the structural and textural characteristics of the parent OMS-2 material. Although these catalysts do not show an optimal performance for the oxidation of CO, the presence of small gold particles enhances the catalytic performances of the cryptomelane producing promissory CO oxidation catalysts. The non-conventional gold deposition favours a partial loss of K + into the channels, resulting in an increment of the average oxidation state of manganese which favours the catalytic behaviour of these kinds of materials. This study can be taken as a starting point to obtain very active gold catalysts supported on OMS-2 materials through the optimisation of the gold-support interaction and the decrease in the gold particle size.

Sub-ambient CO oxidation over Au/MOx/CeO_2-Al_2O_3 (M = Zn or Fe)

Reina, TR; Ivanova, S; Dominguez, MI; Centeno, MA; Odriozola, JA
Applied Catalysis A-General, **419-420** (2012) 58-66
March, 2012 | **DOI:** 10.1016/j.apcata.2012.01.012

A series of ZnO and Fe_2O_3 modified ceria/alumina supports and their corresponding gold catalyst were prepared and studied in the CO oxidation reaction. ZnO-doped solids show a superior catalytic activity compared to the bare CeO_2-Al_2O_3, which is attributed to the intimate contact of the ZnO and CeO_2 phases, since an exchange of the lattice oxygen occurs at the interface. In a similar way, Fe_2O_3-modified supports increase the ability of the CeO_2-Al_2O_3 solids to eliminate CO caused by both the existence of Ce-Fe contact surface and the Fe_2O_3 intrinsic activity. All of the gold catalysts were very efficient in oxidising CO irrespective of the doping metal oxide or loading, with the ZnO containing systems better than the others. The majority of the systems reached total CO conversion below room temperature with the ZnO and Fe_2O_3 monolayer loaded systems the most efficient within the series.

Mechanism of complete n-hexane oxidation on silica supported cobalt and manganese catalysts

Todorova, S; Naydenov, A; Kolev, H; Holgado, JP; Ivanov, G; Kadinov, G; Caballero, A
Applied Catalysis A-General, **413-414** (2012) 43-51
January, 2012 | **DOI:** 10.1016/j.apcata.2011.10.041

Mono- and bi-component cobalt and manganese samples were prepared by impregnation of silica with aqueous solutions of $Co(NO_3)_2{\cdot}6H_2O$ and/or $Mn(NO_3)_2{\cdot}6H_2O$. The bi-component samples were obtained by a common solution of Co- and Mn nitrates (CoMn-MS) or by deposition of cobalt on calcined Mn sample (Co + Mn). The catalysts were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), temperature programmed reduction (TPR), Fourier transformed infrared (FTIR) spectroscopy, X-ray photoelectron spectroscopy (XPS), elemental analysis and tested in reaction of complete n-hexane oxidation. It was observed that the well crystalline cobalt oxide partially covers poorly crystalline manganese oxide in the Co + Mn catalysts, while finely divided oxides (MnO_2 and Mn_2O_3, Co_3O_4) are present on the surface of the (CoMn-MS) sample. Four Langmuir–Hinshelwood and two Mars–van Krevelen models were fitted with the experimental data from the catalytic tests. According to the model calculations and results from instrumental methods, the reaction pathway over single component manganese and bi-component Co-Mn catalysts proceeds through Mars–van Krevelen mechanism (the oxidation of the catalyst surface being the rate determining step), while Langmuir–Hinshelwood mechanism is more probable for the Co sample. A considerable increase in activity for the sample obtained from a mixed solution is explained by low crystallinity, simultaneous presence of Mn^{4+}–Mn^{3+} and enrichment of the surface in oxygen species.

Effect of hydrothermal treatment on structural and photocatalytic properties of TiO_2 synthesized by sol-gel method

Melian, EP; Diaz, OG; Rodriguez, JMD; Colon, G; Navio, JA; Pena, JP
Applied Catalysis A-General, **411** (2012) 153-159
January, 2012 | **DOI:** 10.1016/j.apcata.2011.10.033

TiO_2 nanoparticles have been prepared by sol-gel precipitation and further hydrothermal treatment. In this way, the effect of the hydrothermal treatment on the structural properties and photocatalytic activity of sol-gel synthesized catalysts has been investigated. These catalysts have been produced by hydrolysis of a mixture of isopropanol-titanium tetraisopropoxide (iPrOH-TiiP). The prepared photocatalysts were characterized by means of X-ray diffraction (XRD), surface area analysis (BET), transmission microscopy (TEM), thermogravimetric analysis (TG), scanning electron microscopy (SEM) analysis, diffuse reflectance, sedimentability analysis and aggregate size study. Besides, the structural evolution with the temperature of the photocatalysts treated or not hydrothermally was studied. It was observed that the calcination produces approaching between the characteristics of both sets of photocatalysts. The photocatalytic activity of the obtained photocatalysts was investigated, using phenol as a model pollutant. The calcination temperature is the most remarkable factor that can affect the ultimate photocatalytic activity of the prepared photocatalysts. However, the hydrothermal treatment previous to calcination led to obtain photocatalysts which exhibit larger photocatalytic activity than their homologous photocatalysts without hydrothermal treatment. The obtained photocatalyst TiO_2ht600 exhibits the same photocatalytic activity per surface area than the commercial TiO_2 Degussa P25 but with much faster sedimentability.

Operando DRIFTS study of the redox and catalytic properties of $CuO/Ce_{1-x}Tb_xO_{2-\delta}$ (x = 0–0.5) catalysts: evidence of an induction step during CO oxidation

Martinez-Arias, A.; Hungria, A. B.; Fernandez-Garcia, M.; Iglesias-Juez, A.; Soria, J.; Conesa, J. C.; Anderson, J. A.; Munuera, G.
Physical Chemistry Chemical Physics, **14** (2012) 2144-2151
February, 2012 | **DOI:** 10.1039/C1CP23298C

Catalysts of 1 wt% copper oxide supported on cerium oxide or cerium–terbium mixed oxides are comparatively examined with respect to their redox and catalytic properties for CO oxidation. Characterization of the catalysts had shown that they contain highly dispersed CuO-type entities on the corresponding nanostructured fluorite supports with copper dispersion increasing with increasing amounts of terbium in the support. In contrast, the CO oxidation catalytic activity decreases with increasing amounts of terbium in the support. On the basis of operando-DRIFTS experiments, one of the factors that could explain such behaviour is related to the greater difficulty in generating reduced copper sites active for the reaction in the presence of terbium, which in turn is evidenced to constitute an induction stage. Analysis of the redox properties is complemented by XPS which confirms the greater resistance to copper reduction in the presence of terbium.

Preferential oxidation of CO (CO-PROX) over CuOx/CeO_2 coated microchannel reactor

Laguna, OH; Ngassa, EM; Oraa, S; Alvarez, A; Dominguez, MI; Romero-Sarria, F; Arzamendi, G; Gandia, LM; Centeno, MA; Odriozola, JA
Catalysis Today, **180** (2012) 105-110
January, 2012 | **DOI:** 10.1016/j.cattod.2011.03.024

The general aspects of the synthesis and characterization results of a CuO_x/CeO_2 catalyst were presented. In addition the principal steps for manufacturing a microchannel reactor and for the coating of the CuO_x/CeO_2 catalyst onto the microchannels walls, were also summarized. The catalytic activity of this microchannel reactor during the preferential oxidation of CO (CO-PROX) was evaluated employing a feed-stream that simulates a reformate off-gas after the WGS unit. Two activation atmospheres were studied (H_2/N_2 and O_2/N_2). The reducing pretreatment improved the resistance to deactivation by formation of carbonaceous species over the catalyst surface at high temperatures. The presence of H_2O and CO_2 in the feed-stream was also analyzed indicating that the adsorption of CO_2 inhibited the conversion of CO at lower temperatures because these compounds modified the active sites through the formation of carbonaceous species on the catalyst surface. Finally, the experimental results of the microreactor performance were compared with CFD simulations that were carried out using a kinetic for the CuO_x/CeO_2 powder catalyst. The experimental results were reasonably well described by the model, thus confirming its validity.

Ethanol partial photoxidation on Pt/TiO_2 catalysts as green route for acetaldehyde synthesis

Murcia, JJ; Hidalgo, MC; Navio, JA; Vaiano, V; Ciambelli, P; Sannino, D
Catalysis Today, **196** (2012) 101-109
November, 2012 | **DOI:** 10.1016/j.cattod.2012.02.033

Heterogeneous photocatalytic partial oxidation of ethanol was studied over different Pt/TiO_2 as an alternative green process for acetaldehyde production.

The catalysts were synthesized through the photodeposition of Pt over sol–gel TiO_2 with platinum loads of 0.5 and 1 wt.%. The effect of some experimental conditions during photodeposition, such as deposition time and Pt loading, was investigated. A short deposition time at 0.5 wt.% Pt nominal loading led to small average particle size of platinum (2–3 nm) homogeneously distributed all over the TiO_2 surface.

Ethanol partial oxidation was tested in a gas–solid photocatalytic fluidized bed reactor at high illumination efficiency, using different reaction temperatures. Activity results have been correlated with characterization results of the different samples. Platinized samples prepared with short deposition times showed high conversion levels and high selectivity to acetaldehyde. Materials prepared at longer times, 120 min, showed selectivities >98%, although with lower ethanol conversion.

Sample with 1 wt.% Pt loading prepared with 15 min deposition time combined a good compromise between a relevant ethanol conversion and a very high selectivity to acetaldehyde at a selected reaction temperature of 80 °C, with an acetaldehyde yield higher than 80%, which make of this catalyst a good candidate for acetaldehyde production by photocatalysis.

Redox and catalytic properties of CuO/CeO2 under CO + O2 + NO: Promoting effect of NO on CO oxidation

Martinez-Arias, A.; Hungria, A. B.; Iglesias-Juez, A.; Fernandez-Garcia, M.; Anderson, J. A.; Conesa, J. C.; Munuera, G.; Soria, J.
Catalysis Today, **180** (2012) 81-87
January, 2012 | **DOI:** 10.1016/j.cattod.2011.02.014

A CuO/CeO_2 catalyst has been studied with respect to its catalytic activity for CO oxidation under stoichiometric conditions employing either O_2 or O_2–NO mixture as oxidants. The obtained results are rationalised on the basis of analysis of redox properties upon interaction with CO and O_2–NO by EPR as well as by redox/catalytic analysis by operando-DRIFTS. These provide useful insight into the processes involved during NO reduction, for which two well differentiated steps associated to a change in the type of active centres during the course of the reaction are evidenced. Nevertheless, the most interesting result is related to observation of a novel promoting effect of NO on CO oxidation. This is explained mainly on the basis of DRIFTS results and appears to be associated with phenomena of adsorption/desorption of NOx species at interfacial positions which apparently activate such interfacial region allowing formation of greater amounts of active reduced copper centres in the presence of NO.

DRIFTS study of methanol adsorption on Mg-Al hydrotalcite catalysts for the transesterification of vegetable oils

Navajas, A; Arzamendi, G; Romero-Sarria, F; Centeno, MA; Odriozola, JA; Gandia, LM
Catalysis Communications, **7** (2012) 189-193
January, 2012 | **DOI:** 10.1016/j.catcom.2011.11.005

Mg-Al hydrotalcites rehydrated after calcination are promising catalysts for the methanolysis of vegetable oils. To gain insight into the basis of their catalytic action, the adsorption of methanol over some commercial Mg-Al hydrotalcites was studied by diffuse reflectance infrared Fourier transform spectroscopy (DRIFTS). Different species formed after methanol adsorption were identified, being the total quantity of methoxy species related to the basic character of the sample. A linear correlation between the amount of adsorbed monodentate methoxy species and the catalytic activity in the biodiesel production was found. Therefore, it is proposed that these species are the mainly involved in the transesterification reaction.

Rapid microwave-assisted synthesis of one-dimensional silver–$H_2Ti_3O_7$ nanotubes

Rodriguez-Gonzalez, V; Obregon-Alfaro, S; Lozano-Sanchez, LM; Lee, SW
Journal of Molecular Catalysis A-Chemical, **353** (2012) 163-170
February, 2012 | **DOI:** 10.1016/j.molcata.2011.11.020

The formation of silver hydrogen trititanate nanotubes, based on the controllable microwave-assisted hydrothermal nanocrystalline TiO_2 transition, was investigated by means of XRD, UV–vis–DRS, Raman, FESEM and HRTEM. The results show that the rapid formation of H-trititanate nanotubes is achieved by self-assemblage of silver nanoparticles in which the lamellar intermediates react with NaOH in hydrothermal conditions. The presence of Ag° nanoparticles in the precursor promotes rapid and more complete formation of layered $H_2Ti_3O_7$ nanotubes.

After reacting for 4 h without subsequent thermal treatment, the inner diameters of the cylinder-like nanotubes are in the range of 3.6–4.0 nm, while their outer diameters are in the range of 7.6–8 nm. In addition, some straight nanotubes form bundles which are hundreds of nanometers in length. As-synthesized ultrathin nanotubes and crystalline precursors were evaluated by methyl orange dye (MOD) UV photo-oxidation. The complete degradation of MOD is achieved after 3.5 h of UV irradiation in the presence of silver–TiO_2 nanocomposites, resulting in 50% of dye mineralization.

Hydrogenation of 2,2,2-trifluoroacetophenone: Molecular insight into the role of solvent in enantioselection

Rosa Pereñiguez; Gianluca Santarossa; Tamas Mallat; Alfons Baiker
Journal of Molecular Catalysis A: Chemical, **365** (2012) 39-49
December, 2012 | **DOI:** 10.1016/j.molcata.2012.08.006

The unique solvent effect in the enantioselective hydrogenation of α-fluorinated ketones has been investigated in ten different solvents using the hydrogenation of 2,2,2-trifluoroacetophenone (1) on cinchonine (CN)-modified Pt/Al_2O_3 as a model reaction. Application of strongly basic solvents – but also increasing hydrogen pressure or conversion – inverted the sense of enantiodifferentiation from (S)-alcohol (expected enantiomer based on the stereochemistry of CN) to (R)-alcohol. The known formation of hemiketals was the origin of the inversion in alcohols. Considering only the non-reacting solvents and low conversions at low pressures, the best correlation was established between the enantiomeric excess and the solvent basicity represented by the H-bond acceptor ability (β). In contrast to former proposals, solvent acidity (α) did not play a significant role. The experimental results are validated by theoretical calculations. The docking of 1 to CN has been investigated in the absence of solvent and also in the presence of toluene and dimethyl formamide. Several competing docking complexes have been isolated that can coexist on the metal surface. Detailed analyses of these complexes show that their stabilities depend on the formation of enantiospecific local interactions between 1, CN, and the platinum surface. The presence of solvent interferes with these interactions, affecting the relative stability of the docking complexes. A correlation between the solvent-induced interactions at molecular level and changes in enantioselectivity is suggested.

Gold supported on pillared clays for CO oxidation reaction: Effect of the clay aggregate size

Alvarez, A; Moreno, S; Molina, R; Ivanova, S; Centeno, MA; Odriozola, JA
Applied Clay Science, **69** (2012) 22-29
November, 2012 | **DOI:** 10.1016/j.clay.2012.07.008

A series of 1% m/m gold particles supported on Fe, Ce and Al pillared bentonite (from Valle del Cauca, Colombia) and clay “M64” (from Tolima, Colombia) using three different fractions of aggregate sizes (≤ 2 μm, ≤ 50 μm, and ≤ 150 μm) were characterized by particle size measurements, X-ray diffraction, transmission electronic microscopy (TEM), SBET and X-ray fluorescence spectrometry (XRF) techniques. The materials tested with CO oxidation. The separation yield for each fraction depended on the type of clay. Whatever the clay or the aggregate size, the pillaring process was successfully carried out, introducing Fe, Ce and Al pillars and increasing the microporosity and the specific surface area of the material. Gold particles presented a homogenous distribution of 2–3 nm on the pillared bentonite, and of about 10 nm on the pillared clay M64. The aggregate size slightly influenced the amount of deposited gold particles and their size. All gold catalysts were active in CO oxidation, the activity depending on the nature of the clay as well as the gold loading and average gold particle size but not on the aggregate size.

Nanostructured Spark Plasma Sintered Ce-TZP Ceramics
Cruz, SA; Poyato, R; Cumbrera, FL; Odriozola, JA
Journal of the American Ceramic Society, **95** (2012) 901-906
March, 2012 | **DOI:** 10.1111/j.1551-2916.2011.04978.x

In this work, spark plasma sintering (SPS) of 10 mol% CeO_2-doped ZrO_2 nanocrystalline powders, obtained by a two-step synthesis procedure, allows the preparation of fully densified nanostructured ceramics. The CeO_2-ZrO_2 powders with particle size below 100 nm are obtained after CeO_2 deposition on hydrothermally synthesized ZrO_2 particles by the impregnation method. Tetragonal CeO_2-ZrO_2 ceramics are obtained when sintering at 1200°C without holding time. A graded material containing tetragonal, monoclinic, and pyrochlore phases are obtained when sintering at 1200°C and for 5 min holding time. This is explained in terms of the gradual reduction of Ce^{4+} to Ce^{3+} species by carbon in the graphite environment during SPS. With the successful combination of the stabilizer coating technique and SPS, we achieve not only the stabilization of the tetragonal phase in the ceramics, but also good control of the grain size, by producing nanostructured ceramics with 40-70 nm grain sizes.

A CFD study on the effect of the characteristic dimension of catalytic wall microreactors
Arzamendi, G; Uriz, I; Navajas, A; Dieguez, PM; Gandia, LM; Montes, M; Centeno, MA; Odriozola, JA
AlChE Journal, **58** (2012) 2785-2797
September, 2012 | **DOI:** 10.1002/aic.12790

A three-dimensional computational fluid dynamics study of the steam methane reforming (SMR) in microreactors is presented. Emphasis has been made on investigating the effects of the characteristic dimension (d: 0.35, 0.70, 1.40, and 2.80 mm) on the performance of two microreactor geometries: square microchannels and microslits. Results have shown that for both geometries the SMR conversion decreases markedly as d increases. Conversely, the microchannels provide a methane conversion slightly higher than that of the microslits. The different performance of the microreactors is only partially due to the different surface-to-volume ratio. Pronounced transverse temperature and concentration gradients develop as the characteristic dimension increases especially for microslits in the first half of the reactor. Therefore, external transport limitations can affect the performance of microreactors for SMR, although the characteristic dimensions are of the order of very few millimeters.

Study of Oxygen Reactivity in La_{1-x} Sr(x) CoO_3-delta Perovskites for Total Oxidation of Toluene
Pereniguez, R; Hueso, JL; Gaillard, F; Holgado, JP; Caballero, A
Catalysis Letters, **142** (2012) 408-416
April, 2012 | **DOI:** 10.1007/s10562-012-0799-z

The total oxidation of toluene is studied over catalytic systems based on perovskite with general formula $AA'CoO_{3-\delta}$ (A = La, A' = Sr). The systematic and progressive substitution of La^{3+} by Sr^{2+} cations in the series ($La_{1-x}Sr_xCoO_{3-\delta}$ system) of the perovskites have been studied to determine their influence in the final properties of these mixed oxides and their corresponding reactivity performance for the total oxidation of toluene as a model volatile organic compound with detrimental effects for health and environment. The structure and morphology of the samples before and after reaction have been characterized by XRD, BET and FE-SEM techniques. Additional experiments of temperature programmed desorption of O_2 in vacuum and reduction in H_2 were also performed to identify the main surface oxygen species and the reducibility of the different perovskites. It is remarkable that the $La_{1-x}Sr_xCoO_{3-\delta}$ series presents better catalytic performance for the oxidation of toluene, with lower values for the T 50 (temperature of 50 % toluene conversion) than the previously studied $LaNi_{1-y}Co_yO_3$ series.

Influence of PVP in magnetic properties of NiSn nanoparticles prepared by polyol method
Bobadilla, LF; Garcia, C; Delgado, JJ; Sanz, O; Romero-Sarria, F; Centeno, MA; Odriozola, JA
Journal of Magnetism and Magnetic Materials, **324** (2012) 4011-4018
November, 2012 | **DOI:** 10.1016/j.jmmm.2012.07.005

The influence of PVP on the magnetic properties of NiSn nanoparticles prepared by polyol method has been studied. NiSn nanoparticles exhibit superparamagnetic behavior although there is a ferromagnetic contribution due to particles agglomerated below the blocking temperature. The particle size is controlled by the addiction of PVP in varying amounts. The addition of PVP also favours the particles isolation, narrow the particle size distribution and decrease the interparticle interaction strength increasing the superparamagnetic contribution.

Photocatalytic Ethanol Oxidative Dehydrogenation over Pt/TiO_2: Effect of the Addition of Blue Phosphors
Murcia, JJ; Hidalgo, MC; Navio, JA; Vaiano, V; Ciambelli, P; Sannino, D
International Journal of Photoenergy, **2012** (2012) 687262
January, 2012 | **DOI:** 10.1155/2012/687262

Ethanol oxidative dehydrogenation over Pt/TiO_2 photocatalyst, in the presence and absence of blue phosphors, was performed. The catalyst was prepared by photodeposition of Pt on sulphated TiO_2. This material was tested in a gas-solid photocatalytic fluidized bed reactor at high illumination efficiency. The effect of the addition of blue phosphors into the fluidized bed has been evaluated. The synthesized catalysts were extensively characterized by different techniques. Pt/TiO_2 with a loading of 0.5 wt% of Pt appeared to be an active photocatalyst in the selective partial oxidation of ethanol to acetaldehyde improving its activity and selectivity compared to pure TiO_2. In the same way, a notable enhancement of ethanol conversion in the presence of the blue phosphors has been obtained. The blue phosphors produced an increase in the level of ethanol conversion over the Pt/TiO_2 catalyst, keeping at the same time the high selectivity to acetaldehyde.

Preparation of nanostructured nickel aluminate spinel powder from spent NiO/Al_2O_3 catalyst by mechano-chemical synthesis

Nazemi, M. K.; Sheibani, S.; Rashchi, F.; Gonzalez-DelaCruz, V. M.; Caballero, A.
Advanced Powder Technology, **23** (2012) 833-838
November, 2012 | **DOI:** 10.1016/j.apt.2011.11.004

In this paper, the possibility of mechano-chemical synthesis, as a single step process for preparation of nanostructured nickel aluminate spinel powder from NiO/Al_2O_3 spent catalyst was investigated. Powder samples were characterized in terms of composition, morphology, structure, particle size and surface area using complementary techniques such as X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), differential thermal analysis (DTA) and volumetric adsorption of nitrogen. It was found that formation of spinel was possible after 60 h of milling with no heat treatment. Additionally, influence of mechanical activation on the heat treatment temperature was discussed. It was observed that heat treatment of 15 h milled sample at 1100 °C is enough to produce nickel aluminate spinel. A product of direct mechanical milling showed higher value of surface area (42.3 m^2/g) and smaller crystallite size (12 nm) as compared to the heat treated product.

Oxidative Dehydrogenation of Ethanol over Au/TiO_2 Photocatalysts

Sannino, Diana; Vaiano, Vincenzo; Ciambelli, Paolo; Carmen Hidalgo, M.; Murcia, Julie J.; Antonio Navio, J.
Journal of Advanced Oxidation Technologies, **15** (2012) 284-293
July, 2012

Au/TiO2 photocatalysts were used in ethanol oxidative dehydrogenation. Catalysts at gold loading ranging between 0.5-2 wt.% were synthesized by photodeposition (using different deposition times: 15 and 120 min) over an own-prepared TiO2 by sol-gel method. For reference purposes, a commercial 1 wt.% Au/TiO2 catalyst (AUROlite (TM), Strem Chemicals) was also tested. Photocatalytic reactions were carried out in a gas-solid photocatalytic fluidized bed reactor. Catalytic activity depends strongly both on Au loading and on the material properties, such as particle size and distribution of metal on titania surface. Acetaldehyde was the main reaction product, with ethylene, crotonaldehyde and CO2 as by-products. An important improvement of TiO2 photoactivity was achieved for the catalyst with 0.5 wt.% gold prepared with 120 min deposition time. For an ethanol inlet concentration of 0.2 vol% at 60 degrees C, the maximum conversion and acetaldehyde selectivity were 82% and 95%, respectively. These values are considerably higher than those obtained over pristine TiO2 and over the commercial catalyst.

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

COMUNICACIONES EN CONGRESOS Y REUNIONES / COMMUNICATIONS IN CONGRESS AND MEETINGS

The International Conference on Microreaction Technology, IMRET12

20 – 22 febrero [Lyon, Francia]

Effect of the catalyst layer thickness on CuOx/CeO_2-coated microreactor for the PROX reaction
O.H. Laguna; S. Oraá; D. Marecar; F. J. Echave; O. Sanz; G. Arzamendi; L.M. Gandía; M.A. Centeno; M. Montes; J.A. Odriozola
Poster

Methanol Steam Reforming in Catalytic Wall (PdZn) Microchannels: A CFD Simulation Study
G. Arzamendi; I. Uriz; P.M. Diéguez; F.J. Echave; O. Sanz; O.H. Laguna; M.A. Centeno; M. Montes; J.A. Odriozola; L.M. Gandía
Poster

Réunion Plénière du GdR OR NANO

19 – 21 marzo [Poitiers, Francia]

Gold nanoparticles in heterogeneous oxidation catalysis: structured catalysts
M.A. Centeno
Conferencia Invitada

4th International Congress on Operando Spectroscopy

29 abril – 3 mayo [Nueva York, Estados Unidos de América]

Operando characterization of Au/FeO_x-CeO_2/Al_2O_3 catalysts during water gas shift reaction
T.R. Reina; W. Xu; S. Ivanova; M. A. Centeno; R. S. Jonathan Hanson; J. A. Odriozola; J. A. Rodriguez
Comunicación Oral

7th European Meeting on Solar Chemistry and Photocatalysis:Environmental Applications (SPEA-7)

17 – 20 junio [Oporto, Portugal]

Cyclohexane Photodegradation on Pt/TiO_2 Catalysts
J.J.Murcia; M.C.Hidalgo; J.A.Navio; V.Vaiano; D. Sannino; P. Ciambelli
Poster

Insights Towards the Influence of Pt Features on the Photocatalytic Activity Improvement of TiO_2 by Platinisation
J.J.Murcia; M.C.Hidalgo; J.A.Navio
Poster

Photocatalytic Activity of Single and Mixed Bi_2WO_6 and TiO_2 for Rhodamine B Degradation under Sun-like and Visible Illumination
M.C.Hidalgo; S. Murcia-López; J.A.Navio
Poster

Photocatalytic Water Splitting for H_2 production over Pt-TiO_2 in the presence of metahanol
C.Rodriguez-López; E. Pulido Melián; J.A. Ortega Méndez; J.A. Doña Rodriguez; O.González Diaz; J. Pérez Peña; J.A. Navio; M.Macias
Poster

Synthesis, Characterisation and Photocatalytic Evaluation of Activated Carbon-Bi_2WO_6 systems
M.C.Hidalgo; S. Murcia-López; J.A.Navio
Poster

Hydrogen Production by Photocatalysis Using Bare TiO_2 Photocatalysts
E. Pulido Melián; A. Ortega Méndez; Cristina R. López; O. González Díaz; J.M. Doña Rodriguez; J.A.Navio; D. Fernández Hevía; M.Macias; J. Pérez Peña
Comunicación Oral

EMR 2012 : The Energy and Materials Research Conference

20 – 22 junio [Torremolinos, Málaga, España]

H_2 production by glycerol steam reforming using CoRh/mixed oxide catalysts
L.M. Martínez T.; M. Araque; J.C. Vargas; M.A. Centeno; A. C. Roger
Comunicación oral

15th International Congress on Catalysis

1 – 6 julio [Munich, Alemania]

Economically viable highly active gold based catalyst for WGSR
T.R. Reina; S. Ivanova; V. Idakiev; I. Ivanov; T. Tabakova; M.A. Centeno; J.A. Odriozola
Poster

Highly active Au-TiO_2 systems for low temperature CO oxidation obtained by photodeposition method
A. Caballero; V.M. González-delaCruz; S. Obregón; J.P. Merckl; G.Colón
Poster

Structural and morphological influence on the photocatalytic activity of $BiVO_4$
S. Obregón; G. Colón
Poster

22 International Symposium on Chemical Reaction Engineering, ISCRE22
2 – 5 septiembre [Maastricht, Holanda]

Gas-phase photocatalytic partial oxidation of cyclohexane to cyclohexanol and cyclohexanone on Au/TiO_2 photocatalysts
D. Sannino, V. Vaiano, P. Ciambelli, J.J. Murcia, M.C. Hidalgo, J.A. Navío
Comunicación oral

XXIII Congreso Iberoamericano de Catálisis, CICAT2012
2 – 7 septiembre [Santa Fé, Argentina]

Caracterización in-situ de sistemas Au/CeO_2-Fe_2O_3/Al_2O_3 durante la reacción de WGS
T. R. Reina; W. Xu; S. Ivanova; M. A. Centeno; J. Hanson; J. A. Rodríguez; J. A. Odriozola
Comunicación oral

Desarrollo de reactores de microcanales para la reacción de oxidación preferente de CO en presencia de H_2 (PROX)
O.H. Laguna; F.J. Echave; O. Sanz; G. Arzamendi; L.M. Gandía; M.A. Centeno; M. Montes; J.A. Odriozola
Comunicación oral

Efecto de la temperatura de calcinación en catalizadores Ru/TiO_2 en la reacción de metanación selectiva de CO
A. Muñoz Murillo; A. Pérez; S. Ivanova; M. Á. Centeno; J. A. Odriozola
Comunicación oral

Efecto del Oro en la perovskita de $NiLaO_3$ en la reacción de reformado con vapor de metano
A. Muñoz Murillo; A. Pérez; S. Ivanova; M. Á. Centeno; J. A. Odriozola
Comunicación oral

Influencia de la aleación del reactor microestructurado en el reformado de metanol
F.J. Echave; I. Velasco; O. Sanz; J. A. Odriozola; M. Montes
Comunicación oral

Efecto catalítico y térmico sobre el mecanismo de reacción en el reformado catalítico de glicerol con vapor de agua con catalizadores CeZr(Co/Rh)
S. Palma; L.F. Bobadilla; B. Lorenzatti; S. Ivanova; F. Romero-Sarría; M.A. Centeno; J. A. Odriozola
Poster

Influencia de la forma catalítica en el reformado con vapor de glicerol
L.F. Bobadilla; A. Alvarez; M.I. Domínguez; F. Romero-Sarria; M.A. Centeno; M. Montes; J.A. Odriozola
Poster

Reformado con vapor de agua de la mezcla glicerina-metano
A. Alvarez; L. F. Bobadilla; M. A. Centeno; J. A. Odriozola
Poster

Síntesis y caracterización de catalizadores Au/MnOx depositados sobre monolitos metálicos para la oxidación de CO
L. M. Martínez T; F. Romero Sarria; W. Y. Hernández; M. A. Centeno; J. A. Odriozola
Poster

1st Italian-Spanish School on Catalysis “Recent Advances and New Trends in Catalysis”

11 – 13 septiembre [Verbania, Italia]

A new integrate unit for production and purification of hydrogen
S. Palma; A. Pérez; F. Romero-Sarría; M.A. Centeno; J.A. Odriozola
Poster

Influence of calcination temperature in Ru supported catalyst in selective CO methanation
Muñoz-Murillo; A. Pérez; M.A. Centeno; J.A. Odriozola
Poster

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

CONGRESOS Y REUNIONES NACIONALES / NATIONAL CONGRESSES AND MEETINGS

COMUNICACIONES EN CONGRESOS Y REUNIONES / COMMUNICATIONS IN CONGRESS AND MEETINGS

XII Congreso Nacional de Materiales/XII Congreso Iberoamericano de Materiales

30 mayo – 1 junio [Alicante, España]

Producción de hidrógeno por vía fotocatalítica
E. Pulido Melián, J.A. Ortega Méndez, Cristina R. López, M. Nereida Suárez, O. González Díaz, J. M. Doña Rodriguez, J. A. Navio, M. Macias, J. Pérez Peña, D. Fernández Hevía
Comunicación Oral

Materiales compuestos Bi_2WO_6/TiO_2. Actividad fotocatalítica en la degradación de Rodamina B bajo iluminación solar simulada y visible
S. Murcia-López; J.A. Navio; M.C. Hidalgo
Poster

Preparación y Caracterización de Fotocatalizadores Pt/TiO2: Efecto de la adición de Fosforescentes en al fotooxidación selectiva de Etanol
J.A. Navio; J.J. Murcia; M.C. Hidalgo; V. Vaiano; D. Sannino; P. Ciambelli
Poster

XXX Reunión del grupo especializado en Química Organometálica (GEQO)
12 - 14 junio [Castellón, España]

Epoxidación de olefinas en líquidos iónicos soportados catalizada por complejos de Molibdeno
F. Montilla, C.J. Carrasco, A. Galindo, L. Bobadilla, J.A. Odriozola
Poster

FORMACION / TRAINING

FORMACIÓN DE GRADUADOS / MASTER DEGREE THESIS

Título:	**Development of photocatalysts base don TiO_2 for environmental applications**
Autor:	Laura Abis
Directores:	M. Carmen Hidalgo López y José Antonio Navío Santos
Grado:	(Corso di Laurea in Chimica-Proyecto Fin de Carrera en el ámbito del programa ERASMUS) entre la Universidad de Sevilla y la Universidad de Cagliari (Italia)
Centro:	Instituto de Ciencia de Materiales de Sevilla (Centro Mixto CSIC-Universidad de Sevilla) y Facoltá di Scienze Matematiche Fisiche e Naturali- Universitá Degli Studi di Cagliari (Italy)
Año Académico:	2012

Título: **Estudio de la reacción de oxidación preferencial de CO en presencia de H_2 (PROX) usando microrreactores**
Autor: Miriam González Castaño
Directores: José Antonio Odriozola Gordón, Oscar Hernando Laguna
Grado: Trabajo Fin del Máster "Ciencia y Tecnología de Materiales"
Centro: Facultad de Química. Universidad de Sevilla
Año Académico: 2011-2012

Título: **Evaluación de la eficacia de adsorción de contaminantes orgánicos e inorgánicos por micas sintéticas de alta carga**
Autor: Pilar Fernández Poyatos
Directores: María Dolores Alba Carranza y Mery Carolina Pazos Zarama
Grado: Trabajo Fin del Máster "Ciencia y Tecnología de Materiales"
Centro: Universidad de Sevilla
Año Académico: 2011-2012

Premios y Reconocimientos / Prizes and Acknowledgements

Premio Extraordinario de Doctorado de la Univesidad de Sevilla a Oscar H. Laguna Espitia por el trabajo de Tesis Doctoral titulado "Oxidación preferencial de CO en presencia de H2: diseño de catalizadores basados en CeO2 y aplicación a microrreactores". Dirigida por el Catedrático de Química Inorgánica de la Universidad de Sevilla José Antonio Odriozola y el Investigador Científico del CSIC Miguel Ángel Centeno, esta investigación trata sobre la eliminación del monóxido de carbono (CO) remanente en las corrientes de hidrógeno producidas al transformar alcoholes. Estas corrientes son las utilizadas para alimentar las celdas de combustible, que, a su vez, producirán electricidad.

Premio del Ayuntamiento de Sevilla a la mejores Tesis Doctorales defendidas en la Universidad de Sevilla y en la Universidad Pablo de Olavide en el curso académico 2010-2011 a Oscar H. Laguna Espitia por el trabajo de Tesis Doctoral titulado "Oxidación preferencial de CO en presencia de H2: diseño de catalizadores basados en CeO2 y aplicación a microrreactores". Dirigida por el Catedrático de Química Inorgánica de la Universidad de Sevilla José Antonio Odriozola y el Investigador Científico del CSIC Miguel Ángel Centeno.

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

Proyectos de Investigación / Research Projects

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MINISTERIO DE ECONOMÍA Y COMPETITIVIDAD

Microestructura y deformación plástica a alta temperatura de óxidos eutécticos basados en Al_2O_3

Código/Code:	MAT2009-13979-C03-01
Periodo/Period:	01-01-2010 / 31-12-2012
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	145.200 €
Investigador responsable/Research head:	Jiménez Melendo, M.
Componentes/Research group:	Martínez Fernández, J., De Arellano-López, A. R., Bravo León, A., Clauss Klamp, C., Varela Feria, F. M., Vaquero Aguilar, C.

RESUMEN / ABSTRACT

Se pretende desarrollar óxidos eutécticos binarios y ternarios basados en la alúmina – en concreto, Al_2O_3/ZrO_2, $Al_2O_3/Y_3Al_5O_{12}$(YAG), Al_2O_3/ZrO_2/YAG y $Al_2O_3/SiO_2/ZrO_2$, con la circona estabilizada con diferentes contenidos de Y_2O_3– con microestructuras controladas en el intervalo micro- a nanométrico, para su empleo en aplicaciones estructurales y térmicas de sistemas de generación y conversión más eficientes de la energía: soportes para pilas de combustible, reactores químicos, reactores enfriados por gas de alta temperatura, barreras térmicas sobre superaleaciones metálicas en turbinas y motores, etc.

Entre las altas prestaciones de estos materiales caben destacar: elevado punto de fusión, baja densidad, conductividad térmica, escasa reactividad química incluso a alta temperatura, y excelentes propiedades mecánicas tanto a bajas como a elevadas temperaturas: resistencia mecánica cercana a 5 GPa a temperatura ambiente, resistencia a la fluencia, al desgaste y a la erosión. Pero lo más novedoso y potencialmente interesante de estos materiales es su comportamiento superplástico, en microestructuras nanométricas, recientemente descubierto por el equipo investigador solicitante.Estos materiales se producirán por técnicas de procesamiento asistido por láser en tres configuraciones distintas: masivos, recubrimientos planos (capas individuales sobre soportes cerámicos y metálicos) y multicapas. En particular, para esta última configuración se diseñarán estructuras con campos de tensiones residuales favorables para unas prestaciones mecánicas y térmicas óptimas en función de las diferentes aplicaciones requeridas. Estas tensiones residuales se investigarán mediante técnicas de espectroscopía Raman y piezoespectroscopía, y se compararán con los resultados de simulaciones numéricas. Por otra parte, las técnicas de tratamiento por láser se emplearán también para alterar la microestructura de los recubrimientos cerámicos convencionales depositados por proyección térmica sobre las superaleaciones metálicas en componentes de motores así como para mecanizar los materiales producidos con objeto de conferirles una determinada geometría funcional, o con el fin de modificar su superficie de forma que mejore el comportamiento frente al desgaste.

Uno de los objetivos fundamentales de este proyecto será estudiar materiales con fases nanométricas que posibiliten un comportamiento superplástico a altas temperaturas (que contrasta con la extraordinaria resistencia a la deformación de los materiales con fases micrométricas), de forma que abra la posibilidad de la unión y el conformado fácil con forma casi final de piezas complejas, recuperando su resistencia característica tras tratamientos térmicos.

Las propiedades mecánicas (resistencia a compresión y a flexión, módulo de elasticidad, dureza, tenacidad a fractura y desgaste) de los materiales se evaluarán desde temperatura ambiente hasta 1950 K mediante diferentes técnicas en aire, así como en otras atmósferas para determinar su influencia en el comportamiento mecánico o la degradación del material.

Una parte complementaria, pero fundamental, del proyecto es la caracterización estructural y microestructural de los materiales fabricados, así como su evolución con los ensayos mecánicos. De esta forma se podrán establecer las relaciones entre el comportamiento mecánico observado (propiedades necesarias para el diseño ingenieril) y sus parámetros microestructurales característicos. Con esta finalidad, se utilizarán técnicas de microscopía óptica convencional y confocal, electrónica con microanálisis y difracción, de fuerzas atómicas, así como difracción con textura por rayos X.

Del análisis de los resultados de los ensayos termo-mecánicos y de la caracterización microestructural se pretende llegar al entendimiento básico de la relación entre microestructura y propiedades. Esto permitirá retroalimentar el proceso de producción de los materiales eutécticos, para lograr microestructuras específicas para las diferentes aplicaciones con propiedades macroscópicas optimizadas.

This research addresses to produce binary and ternary oxide eutectics –specifically, Al_2O_3/ZrO_2, $Al_2O_3/Y_3Al_5O_{12}$(YAG), Al_2O_3/ZrO_2/YAG and $Al_2O_3/SiO_2/ZrO_2$, zirconia being stabilized with different amounts of Y_2O_3– with well-controlled microstructures in the micro- to nanometric range for structural and thermal applications in efficient-enhanced power generation and conversion systems: fuel cells, chemical and high-temperature gas cooled reactors, thermal barriers of steels and super alloys in gas turbines and diesel engine components, etc.

These materials are very attractive because of their excellent properties: high melting point, low density, thermal conductivity and chemical reactivity, and superior mechanical performance at both low and high temperature: mechanical strength close to 5 GPa at room temperature, and high creep, wear and erosion resistance. Very recently, superplasticity has been discovered in nanosized materials by the applicant team.

Oxide eutectics will be produced by laser-assisted processing techniques in three configurations: bulk, plates (on ceramic and metallic substrates) and multilaminates. For the later configuration, microarquitectures with optimized residual stresses will be designed for enhanced mechanical and thermal performance. The residual stresses will be investigated by using piezo- and Raman spectroscopy, and the data compared to numerical predictions. Laser techniques will be also used to modify the microstructure of conventional ceramic coatings deposited on metallic engine components by Air Plasma Spray, and for machining of ceramic components to obtain a given functional geometry or to modify the external surfaces for improved wear behavior.

One of the main goals of this Project is to produce materials with nanosized phases in order to achieve superplasticity (which contrasts with the superior creep resistance of microsized materials). This capability opens the possibility of using superplastic joining and forming as processing methods for complex pieces with near net shape, recovering back its characteristic resistance after thermal treatments.

The mechanical properties (flexural and compression resistance, elastic modulus, hardness, toughness and wear) will be evaluated from room temperature up to 1950 K in air as

well as under other different environmental atmospheres in order to investigate their effect in the mechanical behavior or material degradation.

A significant part of the Project is the structural and microstructural characterization of the as-received materials, and their evolution during mechanical tests. Such an investigation is critical to establish relationships between the experimental mechanical behavior (necessary for engineering designs) and the microstructural and processing parameters. To this end, techniques of optical (particularly confocal), electron (image, microanalysis and diffraction) and atomic force microscopy, and X-ray diffraction with texture facilities will be used.

Mechanical and microstructural data will feedback the fabrication process in order to obtain materials with tailored properties for specific applications.

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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 concre-

to 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 primer 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 electro-

químico 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.

TRANSFERENCIA / TECHNOLOGICAL TRANSFER

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 / 31-03-2013
Organismo Financiador/Financial source:	ENRESA
importe total/Total amount:	113.575,00 €
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

PUBLICACIONES / PUBLICATIONS

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

Synthetic high-charge organomica: Effect of the layer charge and alkyl chain length on the structure of the adsorbed surfactants
Pazos, MC; Castro, MA; Orta, MM; Pavon, E; Rios, JSV; Alba, MD
Langmuir, **28** (2012) 7325-7332
May, 2012 | **DOI:** 10.1021/la300153e

A family of organomicas was synthesized using synthetic swelling micas with high layer charge ($Na_nSi_{8-n}Al_nMg_6F_4O_20$ center dot XH_2O, where n = 2, 3, and 4) exchanged with dodecylammonium and octadecylammonium cations. The molecular arrangement of the surfactant was elucidated on the basis on XRD patterns and DTA. The ordering conformation of the surfactant molecules into the interlayer space of micas was investigated by C-13, Al-27, and Si-29 MAS NMR The arrangement of alkylammonium ions in these high-charge synthetic micas depends on the combined effects of the layer charge of the mica and the chain length of the cation. In the organomicas with dodecylammonium, a transition from a parallel layer to a bilayer-paraffin arrangement is observed when the layer charge of the mica increases. However, when octadecylammonium is the interlayer cation, the molecular arrangement of the surfactant was found to follow the bilayer-paraffin model for all values of layer charge. The amount of ordered conformation all-trans is directly proportional of layer charge.

Remediation of metal-contaminated soils with the addition of materials - Part II: Leaching tests to evaluate the efficiency of materials in the remediation of contaminated soils
Gonzalez-Nunez, R; Alba, MD; Orta, MM; Vidal, M; Rigol, A
Chemosphere, **87** (2012) 829-837
May, 2012 | **DOI:** 10.1016/j.chemosphere.2012.01.015

The effect of the addition of materials on the leaching pattern of As and metals (Cu, Zn, Ni, Pb, and Cd) in two contaminated soils was investigated. The examined materials included bentonites, silicates and industrial wastes, such as sugar foam, fly ashes and a material originated from the zeolitization of fly ash. Soil + material mixtures were prepared at 10% doses. Changes in the acid neutralization capacity, crystalline phases and contaminant leaching over a wide range of pHs were examined by using pHstat leaching tests. Sugar foam, the zeolitic material and MX-80 bentonite produced the greatest decrease in the leaching of pollutants due to an increase in the pH and/or the sorption capacity in the resulting mixture. This finding suggests that soil remediation may be a feasible option for the reuse of non-hazardous wastes.

Effect of ytterbium doping on the microstructure and plastic deformation of $BaCeO_3$ perovskite oxide
Jimenez-Melendo, M; Vaquero-Aguilar, C; Huaman-Mamani, FA
Fuel Processing Technology, **103** (2012) 146-150

November, 2012 | **DOI:** 10.1016/j.fuproc.2011.10.005

Trivalent cation-doped barium cerate perovskites are attractive materials for clean-energy applications, in particular solid oxide fuel cells, due to their singular proton conductivity in wet environments. Furthermore, these devices operate at high temperatures, where creep and other deformation processes determine the lifetime and overall performance. In this work, the structural and microstructural characteristics of undoped and ytterbium-doped (1 to 10 at.%) $BaCeO_3$ polycrystals produced by solid state reaction have been investigated. A single orthorhombic perovskite phase was found after sintering in air at 1500 °C for 10 h. The microstructure shows a complex evolution with doping: the average grain size firstly decreases with increasing Yb content up to 5 at.%, and then increases with further Yb additions. The high-temperature mechanical properties have been studied in compression between 1100 and 1250 °C in air at constant initial strain rate. The creep strength increases with increasing Yb content. Extended steady states of deformation were attained at lower strain rates and higher temperatures when increasing doping amount.

Microstructural and high-temperature mechanical characteristics of nickel oxide/zirconia composites for solid oxide fuel cells
Oliva-Ramirez, M.; Huaman-Mamani, F. A.; Jimenez-Melendo, M.
Fuel Processing Technology, **103** (2012) 45-50
November, 2012 | **DOI:** 10.1016/j.fuproc.2011.09.013

NiO/8YSZ (8 mol% Y_2O_3-stabilized ZrO_2) composites with different NiO contents (10, 20 and 40 mol%) have been fabricated by a conventional route of mechanical mixing of NiO and 8YSZ powders and sintering at 1500 °C for 10 h in air. The resulting microstructures have been characterized by electron microscopy. In 10 and 20 mol% NiO/8YSZ, the composite is formed by isolated NiO particles surrounded by zirconia matrix grains; this phase is interconnected in the 40 mol% NiO/8YSZ composite. Mechanical tests at constant strain rate and at constant load were conducted on these materials at temperatures of up to 1350 °C. Different behaviors were found depending on the percolation of the NiO phase. Microstructural observations after deformation are essential to understand the overall mechanical behavior of the composites.

Creep strength of nickel oxide/zirconia composites under different environmental atmospheres
M. Jiménez-Melendo; F.A. Huamán-Mamani
Solid State Ionics, **225** (2012) 471-475
October, 2012 | **DOI:** 10.1016/j.ssi.2012.02.011

NiO/8YSZ (8 mol% Y_2O_3-stabilized cubic ZrO_2) and NiO/3YTZP (3 mol% Y_2O_3-stabilized tetragonal ZrO_2) composites with different NiO contents have been fabricated by a conventional route of mechanical mixing of NiO and zirconia powders and sintering at 1500 °C for 10 h in air. The resulting microstructures have been characterized by electron microscopy. The composites show a duplex microstructure formed by equiaxed grains of NiO and ZrO_2, without any intermediate phase. Compressive mechanical tests at constant strain rate were carried out at temperatures between 1150 and 1350 °C under different environments: air, inert (Ar) and reducing (5% H2/95% Ar) atmospheres. The overall creep behavior of the composites is essentially controlled by the zirconia matrix, due to the softness of the NiO phase in the experimental conditions used in this study. The creep strength is not affected by oxygen partial pressure. However, a large decrease in creep resistance under reducing conditions was observed in samples submitted to in situ redox cycling.

High-temperature plastic deformation mechanisms of ytterbium-doped barium cerate proton conductor
M. Jiménez-Melendo
Solid State Ionics, **225** (2012) 286-290
October, 2012 | **DOI:** 10.1016/j.ssi.2012.03.031

The enhanced proton conductivity exhibited by trivalent cation-doped barium cerate perovskites makes these materials excellent candidates for electrochemical applications, in particular as electrolytes for solid oxide fuel cells. These devices operate at elevated temperatures, where creep and other deformation processes influence the overall efficiency and lifetime. In this work, the high-temperature plastic deformation mechanisms of fine-grained 5 at.% Yb-doped $BaCeO_3$ polycrystals produced by conventional solid-state reaction has been investigated by means of compressive tests at constant load between 1150 and 1250 °C in air. The creep curves show an unusual sigmoidal behavior, followed by extended steady states of deformation. Grain boundary sliding is the main deformation mechanism, characterized by a stress exponent n of 2, as found in other fine-grained superplastic ceramics and metals.

Effects of the presence of Fe(0) on the sorption of lanthanum and lutetium mixtures in smectites
Galunin, E; Alba, MD; Santos, MJ; Vidal, M
Applied Clay Science, **65-66** (2012) 162-172
September, 2012 | **DOI:** 10.1016/j.clay.2012.06.011

The sorption of La and Lu mixtures was examined in two bentonites after incubation for three months at 20 and 80 °C with Fe(0), as a laboratory approach to evaluate the effects of waste canister corrosion in a deep repository on the performance of clay engineered barriers. The sorption/desorption parameters were determined from batch tests in two ionic media: deionized water and, to consider the additional effect of cement leachates, 0.02 mol L− 1 Ca. Results from XRD analyses showed the formation of crystalline FeO(OH), goethite, in a few samples and the degradation of the bentonites due to Fe(0) oxidation during incubation. Moreover, the EDX spectra showed that the lanthanides were sorbed primarily at smectite sites, although sorption onto goethite was also observed, whereas Fe(0) particles did not contribute to lanthanide sorption. The formation of goethite could explain the high Kd values measured in a few scenarios (e.g., those with single solutions or mixtures with the lowest initial concentration of the competitive lanthanide in which high affinity sites governed sorption), with up to 3-fold increases over the values obtained without Fe incubation. However, at higher lanthanide concentration, Kd values decreased or remained constant compared to the samples without Fe incubation, which could be explained by bentonite degradation. In the Ca medium, as much as 5 times lower Kd values were obtained, because of the competitive effect of the Ca ions, especially for Lu in the MX80 bentonite. This indicated that the small number of high affinity sites had been diminished. The sorption data were satisfactorily fitted to a two-solute Langmuir model. In addition, Kd values correlated well with desorption data, which showed that the larger the decrease in Kd, the larger the increase in sorption reversibility. It is suggested that corrosion products from the metal canister might compromise the long-term radionuclide retention of the clay-engineered barriers.

High-temperature mechanical characteristics of NiO/3YTZP composites
Jimenez-Melendo, M; Oliva-Ramirez, M; Huaman-Mamani, FA
Journal of Alloys and Compounds, **536** (2012) S472-S476
September, 2012 | **DOI:** 10.1016/j.jallcom.2011.11.040

NiO/3YTZP (3 mol% Y2O3-stabilized tetragonal ZrO_2) composites with 40 mol% NiO (26 vol% NiO) have been fabricated by mechanical mixing of NiO and 3YTZP powders and sintering at 1500 °C for 10 h in air. The resulting microstructures have been characterized by electron microscopy. Compressive mechanical tests at constant strain rate were conducted on these materials at temperatures between 1150 and 1350 °C in air. The σ–ε curves display extended secondary creep regimes without signals of macroscopic failure. The composite creep behavior is characterized by a stress exponent $n = 2$ and an activation energy for flow $Q = 490$ kJ/mol. The overall creep behavior of the composites is essentially controlled by the zirconia matrix, due to the softness of the NiO phase in the experimental conditions used in this study.

Microstructure and high-temperature mechanical behavior of melt-growth $Al_2O_3/Er_3Al_5O_{12}/ZrO_2$ ternary eutectic composites
Huaman-Mamani, FA; Jimenez-Melendo, M; Mesa, MC; Oliete, PB
Journal of Alloys and Compounds, **536** (2012) S527-S531
September, 2012 | **DOI:** 10.1016/j.jallcom.2012.01.105

The microstructural and high-temperature mechanical characteristics of directionally solidified rods of Al_2O_3–$Er_3Al_5O_{12}$–ZrO_2 ternary eutectic oxides processed by the laser-heated floating zone method at different growth rates have been investigated. The eutectic microstructure displayed an entangled three-dimensional network of Al_2O_3 and $Er_3Al_5O_{12}$ phases of similar sizes, elongated along the growth direction; the minority zirconia phase formed small fibers into the alumina phase. The interphase spacing is reduced with increasing solidification rate, changing about 2 µm down to 200 nm. These microstructural features are essentially the same exhibited by Al_2O_3–$Y_3Al_5O_{12}$–ZrO_2 composites processed by the same technique. Compressive deformation tests performed at 1400 °C at constant strain rate showed that the creep resistance decreased when increasing the growth rate due to the refinement of the microstructure.

Effect of oxidation on the compressive strength of sintered SiC-fiber bonded ceramics
Ramirez-Rico, J; Martinez-Fernandez, J; Singh, M
Materials Science and Engineering A, **534** (2012) 394-399
February, 2012 | **DOI:** 10.1016/j.msea.2011.11.085

The compressive strength of SiC-fiber bonded ceramics obtained from hot-pressed amorphous Si-Al-C-O fibers and its degradation by high temperature exposure to an oxidizing environment was studied. Compressive strength was measured at room temperature as a function of strain rate, orientation, and oxidation temperature. Weight loss was monitored as a function of exposure time in atmospheric air at temperatures ranging from 800 to 1600°C, for times ranging from 0.5 to 5. h. Room-temperature compressive strength had a moderate decrease after exposures at 800°C associated to carbon burnout; increased for exposures in the range 1000-1500°C due to a defect-blunting action of the silica scale; and decreased significantly at 1600°C due to extensive surface recession.

Residual stresses in Al_2O_3-ZrO_2 (3mol.% Y_2O_3) directionally solidified eutectic ceramics as a function of temperature
Ramirez-Rico, J; Martinez-Fernandez, J; Pena, JI; Singh, D; Routbort, J
Materials Science and Engineering A, **541** (2012) 61-66
April, 2012 | **DOI:** 10.1016/j.msea.2012.02.001

Directionally solidified eutectics are in situ composites grown from the melt. Due to the differences in the thermoelastic properties of the different phases present in the material, these composites often exhibit residual stresses that can affect their mechanical properties. In this work we use neutron diffraction to investigate residual stresses in Al_2O_3-ZrO_2 eutectic composites as a function of temperature, for samples processed at two different growth rates, 10mm/h and 750mm/h. Our results show that the stress-free temperature is in the range of 1200±200°C. We explain the experimental observations based on the thermoelastic properties of the phases in the material and confirm our measurements using a simple, self-consistent model.

Interfacial characterization of silicon nitride/silicon nitride joints brazed using Cu-base active metal interlayers
Singh, M; Fernandez, JM; Asthana, R; Rico, JR
Ceramics Intenational, **38** (2012) 2793-2802
May, 2012 | **DOI:** 10.1016/j.ceramint.2011.11.050

Silicon nitride/silicon nitride joints were vacuum brazed at 1317 K for 5 min and 30 min using ductile Cu-base active metal interlayers. The joints were characterized using scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), electron back scattered diffraction (EBSD), and transmission electron microscopy (TEM). An inhomogeneous Ti-rich reaction layer (similar to 2-3 mu m thick) formed in 5 min at the Si3N4/braze interface. The inhomogeneity disappeared after brazing for 30 min and was replaced with a compact and featureless reaction zone. TEM studies revealed fine grains in the reaction layer, and larger grains in the inner part of the joint interfaces. The joints were crack-free and presented features associated with plastic deformation, which indicated accommodation of strain associated with CTE mismatch. Electron Backscatter diffraction (EBSD) revealed a highly textured braze alloy interlayer and its crystallographic orientation was determined. The formation of additional phases at the joint interface during brazing is discussed.

Electrical resistivity and thermal conductivity of SiC/Si ecoceramics prepared from sapele wood biocarbon

Parfen'eva, LS; Orlova, TS; Smirnov, BI; Smirnov, IA; Misiorek, H; Mucha, J; Jezowski, A; Pardo, AG; Rico, JR

Physics of the Solid State, **54** (2012) 2132-2141

October, 2012 | **DOI:** 10.1134/S1063783412100289

Samples of β-SiC/Si ecoceramics with a silicon concentration of ∼21 vol % have been prepared using a series of consecutive procedures (carbonization of sapele wood biocarbon, synthesis of high-porosity biocarbon with channel-type pores, infiltration of molten silicon into empty channels of the biocarbon, formation of β-SiC, and retention of residual silicon in channels of β-SiC). The electrical resistivity ρ and thermal conductivity κ of the β-SiC/Si ecoceramic samples have been measured in the temperature range 5–300 K. The values of ρ Si chan(T) and κ Si chan(T) have been determined for silicon Sichan located in β-SiC channels of the synthesized β-SiC/Si ecoceramics. Based on the performed analysis of the obtained results, the concentration of charge carriers (holes) in Sichan has been estimated as $p \sim 1019$ cm^{-3}. The factors that can be responsible for such a high value of p have been discussed. The prospects for practical application of β-SiC/Si ecoceramics have been considered.

Thermal conductivity of high-porosity heavily doped biomorphic silicon carbide prepared from sapele wood biocarbon

Parfen'eva, LS; Orlova, TS; Smirnov, BI; Smirnov, IA; Misiorek, H; Mucha, J; Jezowski, A; Cabezas-Rodriguez, R; Ramirez-Rico, J

Physics of the Solid State, **54** (2012) 1732-1739

August, 2012 | **DOI:** 10.1134/S1063783412080240

The electrical resistivity and thermal conductivity of high-porosity (~52 vol %, channel-type pores) bio-SiC samples prepared from sapele wood biocarbon templates have been measured in the temperature range 5-300 K. An analysis has been made of the obtained results in comparison with the data for bio-SiC samples based on beech and eucalyptus, as well as for polycrystalline β-SiC. The conclusion has been drawn that the electrical resistivity and thermal conductivity of bio-SiC samples based on natural wood are typical of heavily doped polycrystalline β-SiC.

Biomimetic mineralization of calcium phosphate on a functionalized porous silicon carbide biomaterial

Dey, A; van den Hoogen, CJ; Rosso, M; Lousberg, N; Hendrix, MMRM; Friedrich, H; Ramirez-Rico, J; Zuilhof, H; de With, G; Sommerdijk, NAJM

ChemPlusChem, **77** (2012) 694-699

August, 2012 | **DOI:** 10.1002/cplu.201200118

Porous biomorphic silicon carbide (bioSiC) is a structurally realistic, high-strength, and biocompatible material which is promising for application in load-bearing implants. The deposition of an osteoconductive coating is essential for further improvement of its integration with the surrounding tissue. A new strategy towards biomimetic calcium phosphate coatings on bioSiC is described. X-ray photoelectron spectroscopy (XPS) analysis shows that using 10-undecenoic acid methyl ester a covalently bound monolayer can be synthesized on the surface of the bioSiC. After hydrolysis it exposes carboxylic acid groups that promote the selective nucleation and growth of a very well-defined crystalline layer of calcium phosphate. The resulting calcium phosphate coating is characterized by X-ray diffraction and electron microscopy techniques. Further, ion beam imaging is employed to quantify the mineral deposition meanwhile, three-dimensional dual-beam imaging (FIB/SEM) is used to visualize the bioSiC/mineral interface. The monolayer is show to actively induce the nucleation of a well-defined and highly crystalline mixed octacalcium phosphate/hydroxyapatite (OCP/HAP) coating on implantable bioSiC substrates with complex geometry. The mild biomimetic procedure, in principle, allows for the inclusion of bioactive compounds that aid in tissue regeneration. Moreover, the mixed OCP/HAP phase will have a higher solubility compared to HAP, which, in combination with its porous structure, is expected to render the coating more reabsorbable than standard HAP coatings.

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

COMUNICACIONES EN CONGRESOS Y REUNIONES / COMMUNICATIONS IN CONGRESS AND MEETINGS

Ultra-High Temperature Ceramics: Materials for Extreme Environment Applications II

13-18 mayo [Hernstein, Austria]

Microstructure, tribological response, and mechanical properties of fiber bonded silicon carbide ceramics

M. C. Vera; J. Ramírez-Rico; J. Martínez-Fernández; M. Singh

Poster

1st Al-Nanofunc Workshop: Advanced Microstructural Characterization of Nanomaterials

5-6 julio [Sevilla, España]

Comparative oxidation resistance of CrAlN, CrAlYN and CrAlZrN films by electron microscopies and EELS techniques

T.C. Rojas; S. Domínguez-Meister; S. El Mrabet; M. Brizuela; A. García-Luis; J.C. Sánchez-López

Poster

5th International meeting of Clays in Natural and Enginered Barriers for Radioactive Waste Confinement

20-22 octubre [Montpellier, Francia]

Oxidative dissolution of rare-earth disilicates in H2O2 containing aqueous solutions
R. Gonzalez-Nuñez; M.D. Alba; M. Vidal Espinar
Poster

Competitive effect of metallic canister and clay barrier on the sorption of Eu^{3+} under subcritical conditions
S. Elmrabet; J. Astudillo; M.A. Castro; S. Hurtado; M.M. Orta; M.C. Pazos; S. Rueda; M. Villa; M.D. Alba
Poster

Stability of rare earth disilicate REE$_2$SI$_2$O$_7$ (REE=Sc, Y, Lu) under simulated degradated solutions caused by canister corrosion products
M.C. Pazos; J. Astudillo; M.A. Castro; S. Hurtado; S. EL Mrabet; M.M. Orta; S. Rueda; M. Villa; M.D. Alba
Poster

FORMACION / TRAINING

TESIS DOCTORALES/ DOCTOR DEGREE THESIS

Título: **Influencia de los grupos hidroxilos como grupo funcional primario o secundario en la adsorción de moléculas orgánicas sobre grafito**
Autor: Santiago Medina Carrasco
Directores: Miguel Angel Castro Arroyo y María Dolores Alba Carranza
Calificación: Apto “Cum Laude”
Centro: Universidad de Sevilla

FORMACIÓN DE GRADUADOS / MASTER DEGREE THESIS

Título: **Caracterización de la microestructura de módulos fotovoltáicos de lámina delgada de Tecnología CIGS**
Autor: José María Delgado Sánchez
Directores: María Dolores Alba Carranza y Said El Mrabet
Grado: Trabajo Fin del Carrera “Ingeniería de Materiales”
Centro: Facultad de Física. Universidad de Sevilla

Año Académico: 2011-2012

Título: **Evaluación de la capacidad inmovilizadora de residuos radioactivos trivalentes por mica de alta carga**
Autor: María José García Jiménez
Directores: María Dolores Alba Carranza
Grado: Trabajo Fin del Máster "Ciencia y Tecnología de Materiales"
Centro: Universidad de Sevilla
Año Académico: 2011-2012

Título: **Evaluación de la eficacia de adsorción de contaminantes orgánicos e inorgánicos por micas sintéticas de alta carga**
Autor: Pilar Fernández Poyatos
Directores: María Dolores Alba Carranza y Mery Carolina Pazos Zarama
Grado: Trabajo Fin del Máster "Ciencia y Tecnología de Materiales"
Centro: Universidad de Sevilla
Año Académico: 2011-2012

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

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:	2010 / 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, M. 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 Economía y Competitividad
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 alcan-

zar 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 Ciencia y Competitividad

Importe total/Total amount:	66.550 €
Investigador responsable/Research head:	Luis A. Pérez Maqueda
Componentes/Research group:	Maria Jesús Diánez Millán, José Manuel Criado Luque, 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.

TRANSFERENCIA / TECHNOLOGICAL TRANSFER

PATENTES / PATENTS

Método de determinación de estado de materiales de cubierta de invernaderos

Inventores: Sánchez Soto, P.J.; Garzón Garzón, E.; García Rodríguez, I.G. y Morales Hernández, L.
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Entidad/es Titular/es: CONSEJO SUPERIOR DE INVESTIGACIONES CIENTÍFICAS (CSIC) y UNIVERSIDAD DE ALMERÍA

CONVENIOS Y CONTRATOS / CONTRACTS AND AGREEMENTS

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

Periodo/Period:	25-11-2011 / 24-11-2014
Organismo Financiador/Financial source:	Proyectos e Implantación de Tuberias, SA
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

COOPERACIÓN INTERNACIONAL Y OTROS INTERNATIONAL COOPERATION AND OTHERS

Phase transformations and strengthening during aging of Cu-Ni-X alloys

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Entidad Financiadora/Financial source:	FONDECYT/CONYCIT. Ministerio de Tecnología Chileno

Investigador responsable/Research head:	E. Donoso Catalán
Componentes/Research group:	José Manuel Criado Luque, Maria Jesús Diánez Millán

PUBLICACIONES / PUBLICATIONS

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

Enhancement of Fast CO_2 Capture by a Nano-SiO_2/CaO Composite at Ca-Looping Conditions
Valverde, JM; Perejon, A; Perez-Maqueda, LA
Environmental Science and Technology, **46** (2012) 6401-6408
June, 2012 | **DOI:** 10.1021/es3002426

In this paper we show the performance of a new CO_2 sorbent consisting of a dry physical mixture of a Ca-based sorbent and a SiO_2 nanostructured powder. Thermo-gravimetric analysis (TGA) performed at conditions close to the Ca-looping process demonstrate that the rate of CO 2 capture by the mixture is enhanced during the fast carbonation stage of practical interest in applications. Moreover, the residual capture capacity of the mixture is increased. SEM/EDX, physisorption, and XRD analyses indicate that there is a relevant interaction between the nanostructured SiO_2 skeleton and CaO at high temperatures, which serves to improve the efficiency of the transfer of CO_2 to small reactive pores as well as the stability of the sorbent pore structure.

Nanoclay Nucleation Effect in the Thermal Stabilization of a Polymer Nanocomposite: A Kinetic Mechanism Change
Sanchez-Jimenez, PE; Perez-Maqueda, LA; Perejon, A; Criado, JM
Journal of Physical Chemistry C, **116** (2012) 11797-11807
May, 2012 | **DOI:** 10.1021/jp302466p

The enhanced thermal stability of polymer-clay nanocomposites over the original polymers is one of their most interesting features, and it has been profusely studied within the last decades. Here, a thorough kinetic analysis of polystyrene and a montmorillonite-polystyrene nanocomposite has been performed making use of state-of-the-art kinetic procedures. It has been found that the degradation mechanism changes from a chain scission process for the polymer to a complex two-step nucleation-driven reaction for the nanocomposite. This mechanism change can explain the delayed onset of degradation found in the nanocomposite. Moreover, observation by transmission electron microscopy (TEM) has shown that the clay platelets within the composite could act as nucleation centers for the decomposition.

XRF, μ-XRD and μ-spectroscopic techniques for revealing the composition and structure of paint layers on polychrome sculptures after multiple restorations
Franquelo, ML; Duran, A; Castaing, J; Arquillo, D; Perez-Rodriguez, JL

Talanta, **89** (2012) 462-469
January, 2012 | **DOI:** 10.1016/j.talanta.2011.12.063

This paper presents the novel application of recently developed analytical techniques to the study of paint layers on sculptures that have been restored/repainted several times across centuries. Analyses were performed using portable XRF, µ-XRD and µ-Raman instruments. Other techniques, such as optical microscopy, SEM-EDX and µ-FTIR, were also used. Pigments and other materials including vermilion, minium, red lac, ivory black, lead white, barium white, zinc white (zincite), titanium white (rutile and anatase), lithopone, gold and brass were detected. Pigments from both ancient and modern times were found due to the different restorations/repaintings carried out. µ-Raman was very useful to characterise some pigments that were difficult to determine by µ-XRD. In some cases, pigments identification was only possible by combining results from the different analytical techniques used in this work. This work is the first article devoted to the study of sculpture cross-section samples using laboratory-made µ-XRD systems.

Sonication induced reduction of the Ojen (Andalucia, Spain) vermiculite under air and under nitrogen
Poyato, J; Perez-Rodriguez, JL; Lerf, A; Wagner, FE
Ultrasonics Sonochemistry, **19** (2012) 373-375
March, 2012 | **DOI:** 10.1016/j.ultsonch.2011.07.004

Development of multicomponent-multiphase materials based on (Ti,Ta,Nb)C_xN_{1-x} carbonitride solid solutions
Cordoba, JM; Chicardi, E; Gotor, FJ
Chemical Engineering Journal, **192** (2012) 58-66
June, 2012 | **DOI:** 10.1016/j.cej.2012.03.046

A set of powdered cermets based on (Ti,Ta,Nb)C_xN_{1-x} carbonitride solid solutions were synthesized from mixtures of elemental powders by a mechanically induced self-sustaining reaction (MSR) method and subsequently sintered using a pressureless method. Differing nominal compositions of the hard phase were used, and the nature of the metallic-binder phase (Co, Ni, or Co-Ni) was varied. For comparative purposes, the design of the material was performed using two different synthesis pathways. The composition and microstructure of the ceramic and binder phases before and after sintering were analyzed and related to the microhardness of the material, which was found to increase with increasing contiguity of the hard phase and with decreasing particle size.The samples synthesized in one step (SERIES 2) showed higher microhardness and a more homogeneous microstructure with smaller particle size of the hard phase due to the presence of Ti, Ta, and Nb in the molten binder that hindered ceramic growth during liquid phase sintering.

Microstructural Characterization of Silver Nanoparticles for Bioimaging Applications
Zaderenko, AP; Caro, C; de la Mata, M; Sanchez, JA; Sayagues, MJ
Microscopy and Microanalysis, **18** (2012) 53-54

August, 2012 | **DOI:** 10.1017/S1431927612012925

Silver nanoparticles are emerging as a powerful tool in bioimaging applications owing to their unique plasmonic properties i.e., extremely high molar extinction coefficients, resonant Rayleigh scattering and enhanced local electromagnetic fields. Through the optimization of these properties, by controlling composition, size, shape, and interparticle spacing of nanoparticles and their assemblies, highly enhanced local electromagnetic fields in the vicinity of nanoparticles are achievable giving rise to IR, Raman and fluorescence surface enhanced spectroscopies (SEIRS, SERS and MEF, respectively).

Microstructural Characterization of Silver Nanoparticles for Biomedical Applications
Zaderenko, AP; Castillo, PM; de la Mata, M; Sayagues, MJ; Sanchez, JA
Microscopy and Microanalysis, **18** (2012) 55-56
August, 2012 | **DOI:** 10.1017/S1431927612012937

There is a growing interest in nanoparticles as carriers of chemotherapeutic agents in order to improve their administration and minimize their side effects. Despite the fact that silver nanoparticles can be conjugated to therapeutic agents, offering additionally advantages due their unique and tunable optical properties, few examples have been described yet.

Synthesis of a TiCN–SiC polyhedron and elongated crystals nanopowder at low nitrogen concentration
Engstrom, A; Mouzon, J; Cordoba, JM; Tegman, R; Antti, ML
Materials Letters, **81** (2012) 148-150
August, 2012 | **DOI:** 10.1016/j.matlet.2012.04.071

At room temperature diluted $TiCl_4$ and CCl_4 were reduced by sodium particles and mixed with a polycarbomethylsilane (PCS) solution to yield a precursor. It was dried and subsequently annealed at 1300 °C, 1400 °C and 1450 °C in a tube furnace using argon with 10 ppm N2. After the 1450 °C annealing a nanocrystalline powder of $TiC_{0.5}N_{0.5}$–SiC polyhedron and elongated crystals was obtained. At the low nitrogen concentration during annealing a gradual nitration is proposed. It is promoted by carbon gaseous species, precursor oxidation, a sufficient temperature and a summarised nitrogen surplus compared to the titanium and carbon amount.

In Situ Synthesis of Ceramic Composite Materials in the Ti-B-C-N System by a Mechanically Induced Self-Sustaining Reaction
Aviles, MA; Chicardi, E; Cordoba, JM; Sayagues, MJ; Gotor, FJ
Journal of the American Ceramic Society, **95** (2012) 2133-2139
July, 2012 | **DOI:** 10.1111/j.1551-2916.2012.05174.x

The synthesis of multicomponent ceramic materials in the titanium-diboride-carbide-nitride-carbonitride system by the mechanochemical process known as the mechanically induced self-sustaining reaction (MSR) was investigated. Ceramic composite powders containing TiB_2 and

TiC, TiN or TiC_xN_{1-x} were prepared from a blended mixture of the elements by exploiting the highly exothermic nature of the formation reactions. The synthesis of the composite materials was made possible by the ability of the MSR to simultaneously induce independent self-sustaining reactions, generating a mixture of ceramic phases. The composition of the ceramic composites was designed using the initial atomic ratio of the reactants, and the achieved microstructure was characterized by TiB_2 particles in the micrometric range, surrounded by submicrometric and nanometric TiC, TiN, or TiC_xN_{1-x} crystals.

Room temperature mechanosynthesis of the $La_{1-x}Sr_xMnO_{3\pm\delta}$ (0≤x≤1) system and microstructural study

Sayagues, MJ; Cordoba, JM; Gotor, FJ
Journal of Solid State Chemistry, **188** (2012) 11-16
April, 2012 | **DOI:** 10.1016/j.jssc.2012.01.018

Monophase nanocrystalline powders belonging to the La_{1-x}SrxMn$O_{3\pm\delta}$ system (0≤x≤1) with a perovskite structure have been obtained by mechanochemistry synthesis using a planetary ball milling equipment from La_2O_3, SrO, and Mn_2O_3 mixtures. The solid state reaction was complete after one hour of milling treatment. For all the compositional range, the diffraction domain was very small and the structure appeared as a pseudo cubic perovskite. After annealing at 1100 °C under static air, the symmetry evolution due to the La substitution by Sr was analyzed by X-ray and electron diffraction. Samples with x=0, 0.25, 0.5, and 0.75 were assigned to R-3c space group (1 6 7) in the rhombohedral system and perovskite structure. However, the symmetry of the last term of the system (x=1), $SrMnO_{3\pm\delta}$ sample, changed to P63/mmc space group (1 9 4) in the hexagonal system. The terms with x=0.8, 0.85, and 0.9 presented mainly rhombohedral symmetry.

Bulk TiC_xN_{1-x}–15%Co cermets obtained by direct spark plasma sintering of mechanochemical synthesized powders

Borrell, A; Salvador, MD; Rocha, VG; Fernandez, A; Aviles, MA; Gotor, FJ
Materials Research Bulletin, **47** (2012) 4487-4490
December, 2012 | **DOI:** 10.1016/j.materresbull.2012.09.066

TiC_xN_{1-x}–15 wt.%Co cermets were obtained by a mechanically induced self-sustaining reaction (MSR) and sintered by spark plasma sintering (SPS) technique at different temperatures (1200–1400 °C) for 1 min in vacuum under a uniaxial load of 80 MPa. The evolution of microstructure and mechanical properties was investigated. SPS allowed high densification with limited grain growth at a relatively low temperature. Material sintered at 1300 °C showed a good combination of mechanical properties with Vickers hardness of 17.1 ± 0.5 GPa, fracture toughness of 5.51 ± 0.29 MPa m1/2 and bending strength of 904 ± 12 MPa. Lower sintering temperature resulted in a decrease in bending strength due to poor cohesion between the ceramic and binder phases. An increase in sintering temperature would allow tailoring the cermet microstructure and, therefore, adjusting the Vickers hardness/fracture toughness relation.

Microcalorimetric study of the annealing hardening mechanism of a Cu–2.8Ni–1.4Si (at%) alloy
Donoso, E; Espinoza, R; Dianez, MJ; Criado, JM
Materials Science and Engineering: A, **556** (2012) 612-616
October, 2012 | **DOI:** 10.1016/j.msea.2012.07.035

The precipitation processes in a Cu–2.8 at% Ni–1.4 at% Si alloy were studied using differential scanning calorimetry (DSC), transmission electron microscopy (TEM) and microhardeness measurements. The analysis of the calorimetric curves from room temperature to 900 K shows the presence of one exothermic reaction attributed to the formation of δ-Ni2Si particles in the copper matrix that was confirmed by Transmission Electron Microscopy (TEM) and EDS microanalysis. The activation energies calculated for the precipitation of δ-Ni2Si was lower than the ones corresponding to diffusion of nickel and silicon in copper. A correlation between of microhardness of the alloy and the formation of δ-Ni2Si particles has been found.

Spark plasma sintering of $Ti_yNb_{1-y}C_xN_{1-x}$ monolithic ceramics obtained by mechanically induced self-sustaining reaction
Borrell, A; Salvador, MD; Garcia-Rocha, V; Fernandez, A; Chicardi, E; Gotor, FJ
Materials Science and Engineering A, **543** (2012) 173-179
May, 2012 | **DOI:** 10.1016/j.msea.2012.02.071

Nanometer-sized titanium-niobium carbonitride powders ($Ti_yNb_{1-y}C_xN_{1-x}$) with different Ti/Nb atomic ratios were obtained by a mechanically induced self-sustaining reaction, and sintered by spark plasma sintering technique at 1500°C for 1min in a vacuum atmosphere. Mechanical properties such as hardness and Young's modulus were determined by nanoindentation technique and friction and wear coefficients assessed by ball-on-disk testing using alumina ball in dry sliding conditions. The fracture surface and wear tracks of samples were examined by scanning electron microscopy. Results showed that it is possible to obtain dense monolithic ceramics from the solid solution ($Ti_yNb_{1-y}C_xN_{1-x}$) with good mechanical properties and excellent wear resistance. The optimum values of nanomechanical properties were found for the Ti 0.3Nb 0.7C 0.5N 0.5 ceramic composition, which exhibited a high hardness over 26.0GPa and Young's modulus around 400GPa.

Obituary: Prof. Andrés Ortega
Luque, JMC; Martinez, FJG; Azana, MM; Perez, CR
Thermochimica Acta, **543** (2012) 318-319
September, 2012 | **DOI:** 10.1016/j.tca.2012.06.001

Prof. Andrés Ortega passed away on last January after a painful and long illness. He was Professor of Inorganic Chemistry at the University of Seville (Spain) and was an outstanding researcher in the field of solid state reaction kinetics, an area to which he devoted his entire career since 1983, when he submitted his PhD thesis entitled ‘Critical study of non-isothermal methods for the kinetic analysis of solid-state reactions’. During his post doc stage and collaboration with Prof. Jean Rouquerol, his interest was raised by the Sample Controlled Thermal Analysis (SCTA) technique and its application to the kinetic study of solid state reactions, this latter one developed in Seville along with Prof. José Manuel Criado. A paper from this period should be highlighted: 'Correlation between the shape of controlled-rate thermal analysis curves and the kinetics of solid-state reactions' [Thermochimica Acta 157 (1990) 171], the most cited one in his research career. Most of his scientific production was published in Thermochimica Acta and in the Journal of Thermal Analysis and Calorimetry. A tireless professional, he remained active until a few weeks before dying. Being seriously ill he developed a method for the kinetic analysis of reactions with variable activation energies that notably simplifies the previous one proposed by Vyazovkin. The results were published in Thermochimica Acta under the title 'A simple and precise linear integral method for isoconversional data' [Thermochim. Acta 474 (2008) 81]. The high number of citations of this article – according to the ISI WEB of Knowledge – in spite of the short time elapsed since it was published reveals its impact within the scientific community.

He was also very much involved in teaching duties, developing new subjects and applying new teaching methodologies. He chaired two important academic positions at the University of Seville related to his works on teaching and educational sciences, as Director of the Institute of Educational Science and as Chairman of the Committee on Education of the University.

Though he sometimes appeared to be reserved, Andrés was a kind man, always ready to help in any problem that was presented to him. With a critical attitude and many cultural interests, he had a vast knowledge and a great ability to interpret the most diverse questions, frequently presenting a reasoning alternative to those commonly established. This was a continuous source of enrichment for both his friends and colleagues, who never will forget him.

Characterisation of ternary TixV1-xNy nitride prepared by mechanosynthesis

Roldan, MA; Alcala, MD; Real, C
Ceramics Intenational, **38** (2012) 687-693
January, 2012 | **DOI:** 10.1016/j.ceramint.2011.07.057

In the present manuscript the authors have systematically investigated the composition and microstructure of a series of ternary nitrides ($Ti_xV_{1-x}N_y$) (0.0 <= x <= 1.0) prepared by mechanosynthesis, using XRD, SEM, EELS, XAS and TGA. The ternary titanium-vanadium nitride ($Ti_xV_{1-x}N_y$) has been obtained in all range of compositions by the mechanical treatment of the two metals under nitrogen pressure in a planetary mill with a maximum milling time of 3 h and without any post-heating treatment. The materials' microhardnesses were measured after sinterisation and compared to those reported in the literature for these types of materials. When compared with the previously reported data for bulk samples, these values are similar or higher for compositions within the range x = 0.5 to x = 0.77 (TixV1-xN).

Absence of the core-rim microstructure in $Ti_xTa_{1-x}C_yN_1$-y-based cermets developed from a pre-sintered carbonitride master alloy

Chicardi, E; Cordoba, JM; Sayagues, MJ; Gotor, FJ
International Journal of Refractory Metals & Hard Materials, **33** (2012) 38-43
July, 2012 | **DOI:** 10.1016/j.ijrmhm.2012.02.005

(Ti,Ta)(C,N) solid solution-based cermets with cobalt as the binder phase were synthesised by a two-step milling process. The titanium-tantalum carbonitride solid solution (the ceramic phase) was obtained via a mechanically induced self-sustaining reaction (MSR) process from stoichiometric elemental Ti, Ta, and graphite powder blends in a nitrogen atmosphere. Elemental Co (the binder phase) was added to the ceramic phase, and the mixture was homogenised by mechanical milling (MM). The powdered cermet was then sintered in a tubular furnace at temperatures ranging from 1400°C to 1600°C in an inert atmosphere. The chemical composition and microstructure of the sintered cermets were characterised as ceramic particles grown via a coalescence process and embedded in a complex (Ti,Ta)-Co intermetallic matrix. The absence of the typical core-rim microstructure was confirmed.

Inverse core-rim microstructure in (Ti,Ta)(C,N)-based cermets developed by a mechanically induced self-sustaining reaction
Chicardi, E; Cordoba, JM; Sayagues, MJ; Gotor, FJ
International Journal of Refractory Metals & Hard Materials, **31** (2012) 39-46
March, 2012 | **DOI:** 10.1016/j.ijrmhm.2011.09.003

Cermets with a nominal composition (Tia(0.8)Ta(0.2)C(0.5)N(0.5)-20 wt.% Co) were synthesised by a mechanically induced self-sustaining reaction (MSR) process from stoichiometric elemental powder blends. The MSR allowed the production of a complex (Ti,Ta)(C,N) solid solution, which was the raw material used for the sintering process. The pressureless sintering process was performed at temperatures between 1400 degrees C and 1600 degrees C in an inert atmosphere. The microstructural characterisation showed a complex microstructure composed of a ceramic phase with an unusual inverse core-rim structure and a Ti-Ta-Co intermetallic phase that acted as the binder.

Thermal behaviour of ground and unground acid leached vermiculite
Perez-Maqueda, LA; Maqueda, C; Perez-Rodriguez, JL; Subrt, J; Cerny, Z; Balek, V
Journal of Thermal Analysis and Calorimetry, **107** (2012) 431-438
February, 2012 | **DOI:** 10.1007/s10973-011-1480-2

Acid leaching of vermiculite is an interesting procedure to prepare high surface area porous silica. Thermal behaviour of unground and ground vermiculite leached with HCl solutions has been studied by TG, DTA, ETA and high temperature XRD. Important differences have been observed in the thermal behaviour of unground and ground vermiculite after the acid treatments. Thus, for the acid-treated unground vermiculite, dehydrated vermiculite, enstatite and cristobalite were formed during the heating, while for the acid-treated ground vermiculite only iron oxides and cristobalite phases were observed. Structural modifications due to acid treatment were responsible for changes in the transport properties determined by ETA for the vermiculite samples.

Mechanochemical synthesis and characterization of nanocrystalline BiSe, Bi_2Se_3 semiconductors
Achimovicova, M; Gotor, FJ; Real, C; Daneu, N
Journal of Materials Science: Materials in Electronics, **23** (2012) 1844-1850
October, 2012 | **DOI:** 10.1007/s10854-012-0672-2

Mechanochemical synthesis of bismuth selenides (BiSe, Bi_2Se_3) was performed by high-energy milling of bismuth and selenium powders in a planetary ball mill. The particle size distribution and the specific surface area of Bi/Se and 2Bi/3Se powder mixtures were analysed at increasing milling time. The products were characterized by X-ray diffraction, differential scanning calorimetry and transmission electron microscopy. The presence of bismuth selenide phases was observed after only 1 min of milling and full conversion into hexagonal BiSe phase (nevskite) and rhombohedral Bi 2Se 3 phase (paraguanajuatite) was reached after 10 min of milling. The nanocrystalline nature of both mechanochemically synthesised bismuth selenides was confirmed and their optical band gap energies were obtained on the basis of the recorded absorption spectra in UV-Vis spectral region.

Mechanochemical Synthesis and Characterization of II-VI Nanocrystals: Challenge for Cytotoxicity Issues

Balaz, P; Jardin, R; Dutkova, E; Sayagues, MJ; Balaz, M; Mojzisova, G; Mojzis, J; Turianicova, E; Fabian, M
Acta Physica Polonica A, **122** (2012) 224-229
July, 2012

CdSe@ZnS nanocrystals have been prepared by a two-step solid state mechanochemical synthesis. CdSe prepared from elements in the first step is mixed with ZnS synthesized from zinc acetate and sodium sulfide in the second step. The crystallite size of the new type CdSe@ZnS nanocrystals determined by X-ray diffraction Rietveld refined method was 35 nra and 10 Jam for CdSe and ZnS, respectively. Energy dispersive/transmission electron microscopy/energy dispersive spectroscopy methods show good crystallinity of the nanoparticles and scanning electron microscopy elemental mapping illustrate consistent distribution of Cd, Se, Zn and S elements in the bulk of samples. UV-VIS spectra show an onset at 320 urn with calculated bandgap 3.85 eV. This absorption arises from the vibration modes of Zn-S bonds. The nanocrystals show the blue shift from the bandgap of bulk ZnS (3.66 eV). The synthesized CdSe@ZnS nanocrystals have been tested for dissolution, cytotoxicity and L-cysteine conjugation. The dissolution of Cd was less than 0.05 mu g mL(-1) (in comparison with 0.8 mu g mL(-1) which was evidenced for CdSe alone). The very low cytotoxic activity for selected cancer cell lines has been evidenced. CdSe@ZnS nanocrystals coated with L-cysteine are water-soluble and have a great potential in biomedical engineering as fluorescent labels.

Analysis of the restoration of an historical organ: The case study of the Cavaillé-Coll organ of La Merced Church in Burgos, Spain

Justo-Estebaranz, A; Herrera, LK; Duran, A; Siguenza, B; de Haro, MCJ; Laguna, O; Justo, A
Studies in Conservation, **57** (2012) 21-28
January, 2012 | **DOI:** 10.1179/2047058411Y.0000000001

The restoration of the Cavaille-Coll Romantic organ housed in La Merced Church of Burgos, Spain is described in this paper. The organ was affected by a fire that took place in the church. The effect of the fire on the pipes differed depending on their location within the instrument. A combination of analytical techniques (X-ray diffraction, scanning electron microscopy with energy-dispersive X-ray analysis, particle-induced X-ray emission, metallography, and specific density) allowed the accurate determination of the microstructures and compositions of the alloys used to make the different pipes of the organ, some of which had a high tin content and others which had a high lead content. The most damaged pipes were replaced by reconstructed pipes made out of metallic sheets of the same composition as the originals, to ensure a historically accurate sound.

Degradation of Two Historic Buildings in Northern Spain by Formation of Oxalate and Sulphate-Based Compounds
Duran, A; Robador, MD; Perez-Rodriguez, JL
International Journal of Architectural Heritage, **6** (2012) 342-358
January, 2012 | **DOI:** 10.1080/15583058.2010.551447

This study focused on the degradation processes for two historic buildings, one in a rural and one in an urban environment. Samples collected from the Romanesque Church of Torme and from two areas of the Cathedral of Burgos were studied by using optical and electron microscopy, x-ray spectrometry, x-ray diffraction, Fourier transform infrared spectroscopy and thermal analysis (TG/DTA/DTG). As result of the action of microorganisms, weddellite and whewellite were found to have formed over the entire external walls of the Church of Torme, built with dolomitic rock. Gypsum, formed by the effects of atmospheric pollution, appeared on lime plasters applied as protective coatings on the external stones of Burgos' Cathedral. Also discussed in this study is the different composition of these plasters, based on the use of calcareous and siliceous aggregates.

Non-isothermal microcalorimetric evaluations in quenched and in cold-rolled Cu-9Ni-5.5Sn alloys
Donoso, E; Dianez, MJ; Criado, JM
Revista de Metalurgia, **48** (2012) 67-75
January, 2012 | **DOI:** 10.3989/revmetalm.1136

The thermal aging of both a quenched and a cold rolled homogeneous supersaturated Cu-9 % wt Ni-5.5 wt % Sn alloy has been studied from differential scanning calorimetry (DSC) and microhardness measurements. An increase of the hardness during the aging of the quenched sample, because of the precipitation of a γ' phase, takes place. On the contrary, no hardness increase was observed during the aging of the cold rolled sample. A theoretical analysis of the enthalpy determined from the first DSC exothermic peak suggests that a segregation of the solute towards the dislocations occurs during the aging of the cold rolled alloy. The values of the n Avrami-Erofeev coefficients estimated from the kinetic analysis supports the above interpretations.

LIBROS Y OTRAS PUBLICACIONES / BOOKS AND OTHER PUBLICATIONS

Biological nanostructures associated to iberulites: a SEM study
J.L. Díaz-Hernández; P.J. Sánchez-Soto; A. Serrano-Delgado

En: Current microscopy contributions to advances in science and technology, Formatex Research Center, pp. 154-161
ISBN: 978-84-939843-5-9

Investigación interdisciplinar sobre pinturas murales (S. XV-XVI) en la Iglesia de 'Nuestra Señora de Las Nieves' (La Rinconada, Sevilla): caracterización de materiales y estudio de alteraciones
Antonio Ruiz-Conde; Pedro José Sánchez-Soto; José Pascual Cosp; Guadalupe Durán Domínguez; Mª Angeles González Godoy; María Arjonilla Alvarez; Juan José Lupión Alvarez
En: El patrimonio cultural y natural como motor de desarrollo: investigación e innovación, Universidad Internacional de Andalucía, pp. 967-984
ISBN: 978-84-7993-225-1

Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray analysis (EDX) of Daughter Minerals in Fluid Inclusions in Layered Silicate Materials
A. Ruiz-Conde; E. Garzón; P.J. Sánchez-Soto
En: Current microscopy contributions to advances in science and technology, Ed. Antonio Méndez-Vilas, pp. 1137-1145
ISBN: 978-84-939843-6-6

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

COMUNICACIONES EN CONGRESOS Y REUNIONES / COMMUNICATIONS IN CONGRESS AND MEETINGS

10th International Conference Solid State Chemistry
10 - 14 junio [Pardubice, Czech Republic]

Sample Controlled Reaction Temperature (SCRT): A New Method for Kinetic Analysis and Synthesis of Materials
J.M. Criado; L. A. Pérez- Maqueda; P.E. Sánchez-Jiménez; A. Perejón; M.J. Diánez
Comunicación oral

Study by TEM and DSC of the Hardening Mechanism of a Cu-10Ni-5.5Sn Alloy
M.J. Diánez; A. Perejón; M. J. Sayagués; L. A. Pérez- Maqueda; J.M. Criado
Poster

International congress of chemical engineering
24 - 27 junio [Sevilla, España]

Valorisation of rice and straw husks

M.D. Alcala, E.M. Jimenez, C. Real
Poster

Nanostructured precursors of advanced mullite ceramics and mullite-based composite materials
P.J. Sánchez-Soto; J. Pascual; E. Garzón; A. Ruiz-Conde
Poster

Fabrication of geopolymeric materials by alkaline activation and polymerization from clay
L. Pérez-Villarejo; P.J. Sánchez-Soto; A. Ruiz-Conde; S. Martínez-Martínez; D. Eliche-Quesada; F.J. Iglesias-Godino; F.A. Corpas-Iglesias
Poster

15th International Congress on Thermal Analysis and Calorymetry (ICTAC15)

20 - 24 agosto [Osaka, Japón]

Kinetic studies in solid state reactions by smart temperature methods and advanced analysis procedures
L.A. Pérez-Maqueda, J.M. Criado, P.E. Sánchez-Jiménez, A.Perejón
Conferencia invitada

20th International Congress of Chemical and Process Engineering, CHISA 2012 and 15th Conference on Process Integration, Modelling and Optimisation for Energy Saving and Pollution Reduction, PRES 2012

25 - 29 agosto [Praga, República Checa]

Enhancement of CO2 capture capacity by a fine cohesive powder
Valverde, J.M., Perejon, A. Perez-Maqueda, L.A., Duran, F.J., Pontiga, F., Soria-Hoyo, C., Quintanilla, M.A.S., Moreno, H., Espin, M.J.
Comunicación oral

EURO PM 2012 Congress & Exhibition

16 - 19 septiembre [Basel, Suiza]

Integration of Cermet Component Materials into a Solid Solution Master Alloy by Mechanically Self-Sustaining Reaction
J.M. Córdoba, E. Chicardi, M.A. Avilés, M.J. Sayagués, F.J. Gotor
Comunicación oral

The 15th European Microscopy Congress

16 - 21 septiembre [Manchester, Reino Unido]

Influence of annealing on the microstructure of the Cu-10Ni-5.5Sn alloy
M.J. Sayagués; M.J. Diánez; A. Perejón; P.E. Sánchez-Jiménez; L.A. Pérez Maqueda; J.M. Criado

Poster

Evolution of the microstructure of Ti0.8Ta0.2C0.5N0.5-Co composite material developed by a one or two-steps MSR process
M.J. Sayagués, E. Chicardi, J.M. Córdoba, F. J. Gotor
Poster

Microstructural analysis of La1-xSrxMnO3-δ (LSM) based cathodes obtained by mechano-chemical method at room temperature
R. Moriche, M.J. Sayagués, J.M. Córdoba, F. J. Gotor
Poster

XI Encontro da SBPMat
23 - 27 septiembre [Florianópolis, Brasil]

Effects of micro-and nanometer Zr oxides additions on the dielectric properties of Ca-$Cu_3Ti_4O_{12}$
José M. Criado; M. Jesús Diánez
Poster

International Congress on Science and Technology for the Conservation of Cultural Heritage
2 - 5 octubre [Santiago de Compostela, España]

Study of the Corrosion products of the Lead Blocks from the Historical Organ Jean Pierre Cavaillé (Vinça, France)
A. Justo-Estebaranz, L.K. Herrera, B. Sigüenza, M.C. Jiménez de Haro, A. Justo, O. Laguna
Poster

CONGRESOS Y REUNIONES NACIONALES / NATIONAL CONGRESS AND MEETINGS

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

XXII Simposio del grupo especializado de Cristalografía y crecimiento cristalino
26 – 29 junio [Sevilla, España]

Angel Justo Erbez [Comité Organizador, Conferencia de sesión]
María Jesús Diánez Millán [Comité Organizador]

COMUNICACIONES EN CONGRESOS Y REUNIONES / COMMUNICATIONS IN CONGRESS AND MEETINGS

VII Reunión Grupo Especializado de Física del Estado Sólido (GEFES)

23 - 27 enero [Sevilla, España]

Mechanochemistry as a method for the synthesis of multiferroic materials
A. Perejón, P.E. Sánchez-Jiménez, M.J. Diánez, J.M. Criado, L.A. Pérez-Maqueda
Poster

XII Congreso Nacional de Materiales / XII Congreso Iberoamericano de Materiales

30 mayo - 1 junio [Alicante, España]

Estudio por DSC, DRX y TEM de los cambios estructurales experimentales en función de la temperatura de recocido en una aleación Cu-10Ni-5.5Sn
M.J. Diánez, A. Perejón, P.E. Sánchez-Jiménez, M. J. Sayagués, L.A. Pérez- Maqueda, J.M. Criado
Poster

IV Congreso Nacional de Pulvimetalurgia

20 - 22 junio [Sevilla, España]

Effect of tantalum on the microstructure and mechanical properties of (Ti,Ta)(C,N) based cermets developed by MSR
J.M. Córdoba, E. Chicardi, F.J. Gotor, D. Moreno, J.A. Rodríguez, Y. Torres
Poster

XXII Simposio del grupo especializado de Cristalografía y crecimiento cristalino

26 - 29 junio [Sevilla, España]

Determinación por Difracción de rayos X de los Parámetros Cristalográficos de Minerales de Composición ($CaxMg_{1-x}$) CO_3 obtenidos por Síntesis Suave (Soft Synthesis)
M. J. Diánez, J. M. Criado, L. A. Pérez-Maqueda, A. Perejón, P. E. Sánchez-Jiménez
Poster

LII Congreso Anual de la Sociedad Española de Cerámica y Vidrio

3 - 6 octubre [Burgos, España]

Nuevos morteros con fibras funcionalizadas de interés para su aplicación a la restauración del patrimonio artístico y cultural
J. Cifuentes, M. Benítez, P.J. Sánchez-Soto, F.J. Rubio, J. Pascual-Cosp

Comunicación oral

Imitaciones de vajilla tipo Kuass en el bajo Guadalquivir: caracterización físicoquímica de las pastas e identificación de los centros de producción
V. Moreno-Megías, F.J. García-Fernández, A. Ruiz-Conde, P.J. Sánchez-Soto
Comunicación oral

La producción de contenedores anfóricos en la campiña del Guadalquivir: identificación de centros de producción y líneas de comercialización
F.J. García Fernández, P.J. Sánchez-Soto, A. Ruiz-Conde, V. Moreno-Megías, E. Ferrer Albelda
Poster

Estudio arquométrico de materiales cerámicos vidriados (datados en los siglos XI-XII) procedentes de intervenciones arqueológicas realizadas en los Reales Alcázares de Sevilla
A. Ruiz-Conde, V. Moreno-Megías, F.J. García-Fernández, J. Pascual-Cosp, P.J. Sánchez-Soto
Poster

12 Congreso Binacional de Metalurgia y Materiales. CONAMET/SAM 2012

22 - 26 octubre [Viña del Mar, Chile]

Desarrollo de nuevos sistemas de análisis termogravimétricos para la síntesis de materiales cerámicos
José M. Criado, M. Jesús Diánez, L.A. Pérez Maqueda, A. Perejón, P.E. Sánchez-Jiménez
Comunicación Oral

Efecto de la adición de ZrO, ZrO_2 micro y nanométrico como dopante de $CaCu_3Ti_4O_{12}$
José M. Criado, M. Jesús Diánez
Poster

Estudio de magnetita mineral como catalizador para la síntesis de nanotubos de carbono
José M. Criado, M. Jesús Diánez
Poster

FORMACION / TRAINING

TESIS DOCTORALES/ DOCTOR DEGREE THESIS

Título:	**Mecanosíntesis y caracterización de los materiales multiferroicos nanoestructurados $Bi_{1-x}R_xFeO_3$ (R = La, Y)**
Autor:	Antonio Perejón Pazo
Directores:	Luis A. Pérez Maqueda y Pedro E. Sánchez Jiménez

Calificación: Apto “Cum Laude”
Centro: Universidad de Sevilla

Título: **Síntesis y caracterización de alúminas bioinspiradas de alta superficie específica**
Autor: Mónica Benítez Guerrero
Directores: José Pascual Cosp y Luis A. Pérez Maqueda
Calificación: Apto “Cum Laude”
Centro: Universidad de Málaga

Formación de Graduados / Master Degree Thesis

Título: **Desarrollo de Cermets Basados en Soluciones Sólidas con Contenidos Variables en Cobalto**
Autor: Matías Villegas Calvo
Directores: Francisco José Gotor Martínez
Grado: Trabajo Fin del Master “Ciencia y Tecnología de Nuevos Materiales”
Centro: Universidad de Sevilla
Año Académico: 2012-2013

Título: **Desarrollo de Cátodos para SOFC Basados en Estructura Tipo Perovskita Sintetizados por Métodos Mecanoquímicos**
Autor: Rocío Moriche Tirado
Directores: María Jesús Sayagués de Vega y Francisco José Gotor Martínez
Grado: Trabajo Fin del Master “Ciencia y Tecnología de Nuevos Materiales”
Centro: Universidad de Sevilla
Año Académico: 2012-2013

Título: **Influencia del Tántalo en la Microestructura y Propiedades Termo-Mecánicas de Cermets Basados en Soluciones Sólidas Cuaternarias de Carbonitruros de Tántalo y Titanio**
Autor: Daniel Moreno Sánchez
Directores: Yadir Torres Hernández y Francisco José Gotor Martínez
Grado: Trabajo Fin del Master “Ingeniería de Materiales”
Centro: Universidad de Sevilla
Año Académico: 2012-2013

Título: **Preparación de materiales termoeléctricos tipo escuterudita mediante mechanosíntesis**
Autor: Rocío Rodríguez Laguna

Directores:	Luis A. Pérez Maqueda y P.E. Sánchez-Jiménez
Grado:	Trabajo Fin del Master "Ciencia y Tecnología de Nuevos Materiales"
Centro:	Universidad de Sevilla
Año Académico:	2011-2012

EQUIPAMIENTO CIENTÍFICO AVAILABLE EQUIPMENT

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

MATERIALES FUNCIONALES NANOESTRUCTURADOS
NANOSTRUCTURED FUNCTIONAL MATERIALS

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.

Innovative SOFC Architecture based on Triode Operation

Código/Code:	298300
Periodo/Period:	01-01-2012 / 30-11-2017
Organismo Financiador/Financial source:	Unión Europea
Importe total/Total amount:	256.363 €
Investigador responsable/Research head:	Agustín R. González-Elipe

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MINISTERIO DE ECONOMÍA Y COMPETITIVIDAD

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 delgadas 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 TiO_2 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 direct 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.

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 estudios 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 / 31-12- 2012
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.

Aplicaciones de cristales fotónicos en células solares: aumento de la eficiencia de conversión de potencia mediante la amplificación de la absorción óptica

Applications of photonic crystals in solar cells: power conversion efficiency enhancement though optical absorption amplification

Código/Code:	P08-FQM-03579 (Proyecto de Excelencia)
Periodo/Period:	14-01-2009 / 13-01- 2012
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	291.923 €
Investigador responsable/Research head:	Míguez García, Hernán R.
Componentes/Research group:	Ocaña, M., Calvo, M., Nuñez, N., Mihi, A., Lozano, G., Colodrero, S., Hidalgo, N., Sánchez Sobrado, O.

RESUMEN / ABSTRACT

El concepto central en el que se basa esta propuesta es que los cristales fotónicos, adecuadamente implementados en una célula solar, permiten obtener mejoras sustanciales de su eficiencia. Más concretamente, el presente proyecto pretende estudiar el efecto de la propagación anómala de la radiación electromagnética que tiene lugar en materiales fotónicos sobre la eficiencia y operatividad de células fotovoltaicas de colorante de diverso tipo. El proyecto propone la fabricación de láminas delgadas de partículas coloidales (diámetro comprendido entre 0.010 µm y 1 µm) de óxidos de distinto tipo (óxido de titanio, óxido de estaño, óxido de zinc, óxido de silicio, etc…), pudiendo estas partículas encontrarse tanto en una red ordenada (cristal coloidal) como en forma desordenada pero formando parte de una superestructura que presente orden en una escala de longitud mayor (estructuras multicapas de nanopartículas). Se ha demostrado recientemente que este tipo de estructuras, cuando se acoplan siguiendo un diseño preciso a un electrodo de una célula solar, dan lugar a un aumento extraordinario de la absorción óptica como consecuencia de la localización de luz en la lámina absorbente. Este aumento de la absorción se traduce en un aumento de la fotocorriente que da lugar a su vez a un aumento de la conversión de potencia de la celda. Algunos de los trabajos pioneros a escala mundial en la comprensión del efecto y la aplicación de cristales fotónicos en células solares han sido realizados en el Instituto de Ciencia de materiales de Sevilla y el objetivo primordial de este proyecto es establecer una línea experimental sólida en esta temática que ya ha dado resultados de gran impacto y extraordinariamente prometedores. El presente proyecto se enmarca dentro de la subárea de Nanociencias, Nanotecnología y Materiales dedicada a Nuevos Materiales Fotónicos para Células Solares y Pantallas, según establece en la convocatoria la Consejería de Innovación, Ciencia y Empresa de la Junta de Andalucía.

Porous photonic crystals introduced in heterojunction solar cells allow to enhance significantly their photovoltaic performance by increasing the light harvested by the device. This concept, pioneered by the multifunctional optical materials group, have lead to highly efficient and transparent dye solar cells that preserve their potential application as window modules, one of their main added values. The concepts proposed in this project are not only interesting from a fundamental point of view in photonics and energy conversion, but also of clear relevance for building integrated photovoltaics.

Materiales poliméricos y nanocomposites híbridos 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 / 31-12-2012
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	257.923,68 €
Investigador responsable/Research head:	Barranco Quero, Angel
Componentes/Research group:	Juan Pedro Espinós, Francisco Yubero, Ana Borrás, Fabián Frutos, Lola González-García, Said Hamad, S. Lago, Alberto Palmero, Carmen Ruiz-Herrero, Juan R. Sánchez-Valencia, Johan Toudert, Santiago Lago Aranda, Said Hamad, Victor Rico Gavira

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.

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.

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: 01-01-2010 / 31-12-2012
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 for-

mation 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.

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.

Transferencia / Technological Transfer

Convenios y Contratos / Contracts and Agreements

Desarrollo de nuevos pigmentos cerámicos con tonalidad roja o amarilla

Periodo/Period: 01-11-2011 / 31-12-2012
Organismo Financiador/Financial source: Al-Farben, S.A.
Investigador responsable/Research head: Manuel Ocaña Jurado
Componentes/Research group: Alberto Escudero Belmonte, Ana Isabel Becerro Nieto

Síntesis y aplicación de materiales nanostructurados multifuncionales en aglomerados pétreos

Periodo/Period: 01-06-2010 / 01-09-2012
Organismo Financiador/Financial source: Cosentino, S.A.
Investigador responsable/Research head: Manuel Ocaña Jurado
Componentes/Research group: Hernán Míguez García, Nuria Núñez, Mauricio Calvo Roggiani, Carolina Rosero Navarro

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

CONSOLIDER-INGENIO 2010 - CSD 2007-00007 - Proyecto: Hybrid optoelectronic and photovoltaic devices for renewable energy

Periodo/Period: 01-06-2010 / 01-09-2012
Organismo Financiador/Financial source: Universitat Jaume I de Castello
Investigador responsable/Research head: Hernán R. Míguez García

Componentes/Research group: Manuel Ocaña Jurado, Mauricio Calvo Roggiani, Nuria O. Nuñez Alvarez

Síntesis y aplicación de materiales nanostructurados multifuncionales en aglomerados pétreos

Periodo/Period: 01-06-2010 / 01-09-2012
Organismo Financiador/Financial source: Cosentino, S.A.
Investigador responsable/Research head: Francisco Yubero Valencia
Componentes/Research group: Agustín R. González-Elipe, Juan Pedro Espinós, Alberto Palmero, José Cotrino Bautista, Angel Barranco Quero, Jorge Gil Rostra, Victor H. Rico

Desarrollo de láminas delgadas fluorescentes sensibles a agentes del entorno y utilizables para fabricar sensores ópticos

Periodo/Period: 01-01-2010 / 31-12-2011
Organismo Financiador/Financial source: ARQUIMEA
Investigador responsable/Research head: Angel Barranco Quero

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

Estudio sobre el uso de combinaciones de reactores de descarga de barrera dieléctrica para la producción de hidrógeno a partir del reformado de hidrocarburos

Periodo/Period: 01-01-2011 / 31-12-2012
Organismo Financiador/Financial source: HYNERGREEN
Investigador responsable/Research head: José Cotrino Bautista

Sol y visión para la energía térmica actual SOLVENTA

Periodo/Period: 01-01-2011 / 31-12-2014
Organismo Financiador/Financial source: Proyecto INNPACTO acuerdo con la empresa ABENGOA SOLAR NEW TECHNOLOGIES
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

COOPERACIÓN INTERNACIONAL Y OTROS INTERNATIONAL COOPERATION AND OTHERS

Environmentally friendly processing of ceramics and glass (CERAMGLASS)

Periodo/Period:	2012 / 2015
Entidad Financiadora/Financial source:	European projects (Life program, contract number ENV/ES/000560)
Investigador responsable/Research head:	Agustín R. González-Elipe
Componentes/Research group:	Juan Pedro Espinós Manzorro, Angel Barranco Quero, Alberto Palmero Acebedo, Victor J. Rico Gavira, Ana Isabel Borrás Martos, José Cotrino Bautista, Francisco Yubero Valencia

PUBLICACIONES / PUBLICATIONS

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

Collective osmotic shock in ordered materials
Paul Zavala-Rivera, Kevin Channon, Vincent Nguyen, Easan Sivaniah, Dinesh Kabra, Richard H. Friend, S. K. Nataraj, Shaheen A. Al-Muhtaseb, Alexander Hexemer, Mauricio E. Calvo & Hernan Miguez
Nature Materials, **11** (2012) 53–57
January, 2012 | **DOI:** 10.1038/nmat3179

Osmotic shock in a vesicle or cell is the stress build-up and subsequent rupture of the phospholipid membrane that occurs when a relatively high concentration of salt is unable to cross the membrane and instead an inflow of water alleviates the salt concentration gradient. This is a well-known failure mechanism for cells and vesicles (for example, hypotonic shock) and metal alloys (for example, hydrogen embrittlement). We propose the concept of collective osmotic shock, whereby a coordinated explosive fracture resulting from multiplexing the singular effects of osmotic shock at discrete sites within an ordered material results in regular bicontinuous structures. The concept is demonstrated here using self-assembled block copolymer micelles, yet it is applicable to organized heterogeneous materials where a minority component can be selectively degraded and solvated whilst ensconced in a matrix capable of plastic deformation. We discuss the application of these self-supported, perforated multilayer materials in photonics, nanofiltration and optoelectronics.

Efficient Transparent Thin Dye Solar Cells Based on Highly Porous 1D Photonic Crystals
Colodrero, S; Forneli, A; Lopez-Lopez, C; Pelleja, L; Miguez, H; Palomares, E
Advanced Functional Materials, **22** (2012) 1303-1310

March, 2012 | **DOI:** 10.1002/adfm.201102159

A working electrode design based on a highly porous 1D photonic crystal structure that opens the path towards high photocurrents in thin, transparent, dye-sensitized solar cells is presented. By enlarging the average pore size with respect to previous photonic crystal designs, the new working electrode not only increases the device photocurrent, as predicted by theoretical models, but also allows the observation of an unprecedented boost of the cell photovoltage, which can be attributed to structural modifications caused during the integration of the photonic crystal. These synergic effects yield conversion efficiencies of around 3.5% by using just 2 mu m thick electrodes, with enhancements between 100% and 150% with respect to reference cells of the same thickness.

Introducing structural colour in DSCs by using photonic crystals: interplay between conversion efficiency and optical properties
Colonna, D; Colodrero, S; Lindstrom, H; Di Carlo, A; Miguez, H
Energy & Environmental Science, **5** (2012) 8238-8243
August, 2012 | **DOI:** 10.1039/c2ee02658a

Herein we analyze experimentally the effect that introducing highly reflecting photonic crystals, operating at different spectral ranges, has on the conversion efficiency of dye sensitized solar cells. The interplay between structural colour and cell performance is discussed on the basis of the modified spectral response of the photogenerated current observed and the optical characterization of the cells. We demonstrate that, with the approach herein discussed, it is possible to achieve relatively high efficiencies using thin electrodes while preserving transparency. At the same time, the appearance of the device can be controllably modified, which is of relevance for their potential application in building integrated photovoltaics (BIPV) as window modules.

Self-assembly at room temperature of thermally stable discrete and extended oligomers of polycyclic aromatics on Ag(100): induced dipoles and cooperative effects
Papageorgiou, AC; Alavi, A; Lambert, RM
Chemical Communications, **48** (2012) 3394-3396
April, 2012 | **DOI:** 10.1039/c2cc17728e

Thermally stable nanoarchitectures are realized on the Ag(100) surface by self-assembly of asymmetrically substituted arenes. The process is instigated by adsorption-induced molecule → surface charge transfer that gives rise to in-plane dipole moments. Observation and calculation indicate that cooperative interactions further enhance the stability of these polarizable systems.

Enhanced diffusion through porous nanoparticle optical multilayers
Lopez-Lopez, C; Colodrero, S; Raga, SR; Lindstrom, H; Fabregat-Santiago, F; Bisquert, J; Miguez, H
Journal of Materials Chemistry, **22** (2012) 1751-1757
February, 2012 | **DOI:** 10.1039/c1jm15202e

Herein we demonstrate improved mass transport through nano-particle one-dimensional photonic crystals of enhanced porosity. Analysis is made by impedance spectroscopy using iodine and ionic liquid based electrolytes and shows that newly created large pores and increased porosity improve the diffusion of species through the photonic crystal. This achievement is based on the use of a polymeric porogen (polyethylene glycol), which is mixed with the precursor suspensions used for the deposition of nanoparticle TiO2 and SiO2 layers and then eliminated to generate a more open interconnected void network, as confirmed by specular reflectance porosimetry. A compromise between pore size and optical quality of these periodic structures is found.

Superhydrophobic supported Ag-NPs@ZnO-nanorods with photoactivity in the visible range
Macias-Montero, M; Borras, A; Saghi, Z; Romero-Gomez, P; Sanchez-Valencia, JR; Gonzalez, JC; Barranco, A; Midgley, P; Cotrino, J; Gonzalez-Elipe, AR
Journal of Materials Chemistry, **22** (2012) 1341-1346
January, 2012 | **DOI:** 10.1039/C1JM13512K

In this article we present a new type of 1D nanostructures consisting of supported hollow ZnO nanorods (NRs) decorated with Ag nanoparticles (NPs). The 3D reconstruction by high-angle annular dark field scanning transmission electron microscopy (HAADF-STEM) electron tomography reveals that the Ag NPs are distributed along the hollow interior of the ZnO NRs. Supported and vertically aligned Ag-NPs@ZnO-NRs grow at low temperature (135 °C) by plasma enhanced chemical vapour deposition on heterostructured substrates fabricated by sputtered deposition of silver on flat surfaces of Si wafers, quartz slides or ITO. The growth mechanisms of these structures and their wetting behavior before and after visible light irradiation are critically discussed. The as prepared surfaces are superhydrophobic with water contact angles higher than 150°. These surfaces turn into superhydrophilic with water contact angles lower than 10° after prolonged irradiation under both visible and UV light. The evolution rate of the wetting angle and its dependence on the light characteristics are related to the nanostructure and the presence of silver embedded within the ZnO NRs.

Weakly Interacting Molecular Layer of Spinning C60 Molecules on TiO2 (110) Surfaces
Sanchez-Sanchez, C; Lanzilotto, V; Gonzalez, C; Verdini, A; de Andres, PL; Floreano, L; Lopez, MF; Martin-Gago, JA
Chemistry-A European Journal, **18** (2012) 7382-7387
June, 2012 | **DOI:** 10.1002/chem.201200627

The adsorption of C60, a typical acceptor organic molecule, on a TiO2 (110) surface has been investigated by a multitechnique combination, including van der Waals density functional calculations. It is shown that the adsorbed molecules form a weakly interacting molecular layer, which sits on the fivefold-coordinated Ti that is confined between the prominent bridging oxygen rows.

Effect of diffuse light scattering designs on the efficiency of dye solar cells: An integral optical and electrical description
Galvez, FE; Kemppainen, E; Miguez, H; Halme, J
Journal of Physical Chemistry C, **116** (2012) 11426-11433
May, 2012 | **DOI:** 10.1021/jp2092708

Herein, we present an integral optical and electrical theoretical analysis of the effect of different diffuse light scattering designs on the performance of dye solar cells. Light harvesting efficiencies and electron generation functions extracted from optical numerical calculations based on a Monte Carlo approach are introduced in a standard electron diffusion model to obtain the steady-state characteristics of the different configurations considered. We demonstrate that there is a strong dependence of the incident photon to current conversion efficiency, and thus of the overall conversion efficiency, on the interplay between the value of the electron diffusion length considered and the type of light scattering design employed, which determines the spatial dependence of the electron generation function. Other effects, like the influence of increased photoelectron generation on the photovoltage, are also discussed. Optimized scattering designs for different combinations of electrode thickness and electron diffusion length are proposed.

Nanoporous silica microparticle interaction with toll-like receptor agonists in macrophages
Cejudo-Guillen, M; Ramiro-Gutierrez, M L; Labrador-Garrido, A; Diaz-Cuenca, A; Pozo, D
Acta Biomaterialia, **8** (2012) 4295-4303
December, 2012 | **DOI:** 10.1016/j.actbio.2012.07.026

Nanoporous silica microparticles (NSiO2-MP) are considered to be potential drug delivery systems and scaffolding platforms in tissue engineering. However, few biocompatibility studies regarding NSiO2-MP interaction with the immune system have been reported. Toll-like receptors (TLR) are involved in host defence as well as autoimmune and inflammatory diseases. The results show that NSiO2-MP up to 100 μg ml−1 do not affect macrophage cell viability after 24 h cell culture. Moreover, NSiO2-MP do not compromise the cell viability of TLR-activated Raw 264.7 cells, for either cell surface TLR (TLR1/TLR2/TLR4/TLR6) or endocytic compartment TLR (TLR3/TLR7/TLR9). Furthermore, Raw 264.7 cells do not respond to NSiO2-MP exposure in terms of IL-6 or IL-10 secretion. NSiO2-MP co-treatment in the presence of TLR ligands does not impair or enhance the secretion of the pro-inflammatory cytokine IL-6 or the regulatory cytokine IL-10. Thus, NSiO2-MP do not affect macrophage polarization towards a pro-inflammatory or immunosuppressive status, representing added value in terms of biocompatibility compared with other SiO2-based micro- and nanoparticles.

Adsorption Geometry Determines Catalytic Selectivity in Highly Chemoselective Hydrogenation of Crotonaldehyde on Ag(111)
Brandt, Katrin; Chiu, May E.; Watson, David J.; Tikhov, Mintcho S.; Lambert, Richard M.
Journal of Physical Chemistry C, **116** (2012) 4605-4611
January, 2012 | **DOI:** 10.1021/jp208831h

The chemoselective hydrogenation of crotonaldehyde to crotyl alcohol was studied by temperature-programmed desorption/reaction, high-resolution XPS, and NEXAFS. The organic molecule adsorbed without decomposition, all three possible hydrogenation products were formed and desorbed, and the clean overall reaction led to no carbon deposition. Selectivities up to 95% were found under TPR conditions. The observed behavior corresponded well with selectivity trends previously reported for Ag/SiO2 catalysts, and the present findings permit a rationalization of the catalytic performance in terms of pronounced coverage-dependent changes in adsorption geometries of the reactant and the products. Thus, at low coverages, the C═O bond in crotonaldehyde lies almost parallel to the metal surface, whereas the C═C was appreciably tilted, favoring hydrogenation of the former and disfavoring hydrogenation of the latter. With increasing coverage of reactants, the C═C bond was forced almost parallel to the surface, rendering it vulnerable to hydrogenation, thus markedly decreasing selectivity toward formation of crotyl alcohol. Butanol formation was the result of an overall two-step process: crotonaldehyde → crotyl alcohol → butanol, further hydrogenation of the desired product crotyl alcohol being promoted at high hydrogen coverage due to the C═C bond in the unsaturated alcohol being driven from a tilted to a flat-lying geometry. Finally, an explanation is offered for the strikingly different behavior of Ag(111) and Cu(111) in the chemoselective hydrogenation of crotonaldehyde in terms of the different degrees of charge transfer from metal to C═O π bond, as suggested by C 1s XPS binding energies.

Investigation of the growth mechanisms of a-CH x coatings deposited by pulsed reactive magnetron sputtering

Lopez-Santos, C; Colaux, JL; Gonzalez, JC; Lucas, S

Journal of Physical Chemistry C, **116** (2012) 12017-12026

June, 2012 | **DOI:** 10.1021/jp300697s

The study of the growth mechanisms of amorphous hydrogenated carbon coatings (a-CH x) deposited by reactive pulsed magnetron discharge in Ar + C 2H 2, Ar + H 2, and Ar + C 2H 2 + H 2 low-pressure atmospheres is presented in this work. Hydrogen-containing species of the reactant gas affect the microstructure and surface properties of the a-CH x thin films. The dynamic scaling theory has been used to relate the main reactive species involved in the deposition process to the growth mechanisms of the thin film by means of the analysis of the roughness evolution. Anomalous scaling effects have been observed in smooth a-CH x coatings. Dynamic scaling exponents α, β, and z indicate a general growth controlled by surface diffusion mechanisms. Hydrogen species have an influence on the lateral growth of the a-CH x coatings and are involved in the development of a polymeric-like structure. Meanwhile, hydrocarbon species promote the generation of higher aggregates, which increases the roughness of a more sp 2 clustering structure of the a-CH x coating.

Plasma deposition of perylene-adamantane nanocomposite thin films for NO 2 room-temperature optical sensing

Aparicio, FJ; Blaszczyk-Lezak, I; Sanchez-Valencia, JR; Alcaire, M; Gonzalez, JC; Serra, C; Gonzalez-Elipe, AR; Barranco, A

Journal of Physical Chemistry C, **116** (2012) 8731-8740

April, 2012 | **DOI:** 10.1021/jp209272s

This work reports the preparation, by a new remote assisted plasma deposition process, of luminescent nanocomposite thin films consisting of an insoluble organic matrix where photonically active perylene molecules are embedded. The films are obtained by the remote plasma deposition of adamantane and perylene precursor molecules. The results show that the adamantane precursor is very effective to improve the perylene–adamantane nanocomposite transparency in comparison with plasma deposited perylene films. The plasma deposited adamantane films have been characterized by secondary-ion mass spectrometry and FT-IR spectroscopy. These techniques and atomic force microscopy (AFM) have been also used for the characterization of the nanocomposite films. Their optical properties (UV–vis absorption, fluorescence, and refractive index) have been also determined and their sensing properties toward NO2 studied. It is found that samples with the perylene molecules embedded within the transparent plasma deposited matrix are highly sensitive toward this gas and that the sensitivity of the films can be adjusted by modifying the aggregation state of the perylene molecules, as determined by the analysis of their fluorescence spectra. By monitoring the fluorescence emission of these films, it has been possible to detect a NO2 concentration as low as 0.5 ppm in air at room temperature. Because of their chemical stability and transparency in the UV region, the remote plasma deposited adamantane thin films have revealed as an optimum host matrix for the development of photonically active composites for sensing applications.

Revealing Structural Detail in the High Temperature La2Si2O7–Y2Si2O7 Phase Diagram by Synchrotron Powder Diffraction and Nuclear Magnetic Resonance Spectroscopy

Fernandez-Carrion, AJ; Allix, M; Florian, P; Suchomel, MR; Becerro, AI
Journal of Physical Chemistry C, **116** (2012) 21523-21535
October, 2012 | **DOI:** 10.1021/jp305777m

High resolution synchrotron powder XRD, 89Y CPMG NMR, and 139La MAS NMR spectroscopy have been applied to eventually draw the phase diagram of the La2Si2O7–Y2Si2O7 system. The diagram presents a solid solubility region of G-(La,Y)2Si2O7, which extends to the La0.9Y1.1Si2O7 composition at any temperature of this study. Compositions richer in Y show two-phase domains, with G + α at T < 1450 °C and G + δ at T > 1450 °C. The Y-rich extreme is more complex, showing two solid solution regions of δ- and γ-(La,Y)2Si2O7 polymorphs which appear with increasing Y content, respectively. It is interesting to note that the La for Y substitution mechanism in the G-(La,Y)2Si2O7 polymorph is not homogeneous, but a preferential occupation of Y for the RE2 site is observed. Finally, the 89Y and 139La isotropic chemical shift values in G-(La,Y)2Si2O7 have been described here for the first time and assigned to the different RE crystallographic sites of the unit cell.

Microwave-Assisted Synthesis and Luminescence of Mesoporous REDoped YPO4 (RE = Eu, Ce, Tb, and Ce plus Tb) Nanophosphors with Lenticular Shape

Rodriguez-Liviano, S; Aparicio, FJ; Rojas, TC; Hungria, AB; Chinchilla, LE; Ocana, M
Crystal Growth and Design, **12** (2012) 635-645
February, 2012 | **DOI:** 10.1021/cg201358c

Mesoporous tetragonal RE:YPO 4 nanophosphors (RE = Eu, Ce, Tb, and Ce + Tb) with a lenticular morphology, narrow size distribution, and high surface area have been prepared by an homogeneous precipitation procedure consisting of aging, at low temperature (80-120 °C) in a microwave oven, ethylene glycol solutions containing only yttrium acetylacetonate and phosphoric acid. This synthesis method involves important advantages such as its simplicity, rapidness (reaction time = 7 min), and high reaction yields. The mechanism of nanoparticle growth has been also addressed finding that the lenticular nanoparticles are formed through an ordered aggregation of smaller entities, which explains their porosity. In all cases, the doping levels were systematically varied in order to optimize the nanophosphors luminescence. All optimum nanophosphors presented a high luminescence quantum yield (QY). In particular, for the Eu and Tb doped systems, the obtained QY values (60% for Eu and 80% for Tb) were the highest so far reported for this kind of nanomaterial. The morphological, microstructural, and luminescent properties of these nanophosphors and their dispersibility in water make them suitable for biomedical applications.

Synthesis and Structure Resolution of RbLaF4
Rollet, AL; Allix, M; Veron, E; Deschamps, M; Montouillout, V; Suchomel, MR; Suard, E; Barre, M; Ocana, M; Sadoc, A; Boucher, F; Bessada, C; Massiot, D; Fayon, F
Inorganic Chemistry, 51 (2012) 2272-2282
March, 2012 | **DOI:** 10.1021/ic202301e

The synthesis and structure resolution of RbLaF4 are described. RbLaF4 is synthesized by solid-state reaction between RbF and LaF3 at 425 degrees C under a nonoxidizing atmosphere. Its crystal structure has been resolved by combining neutron and synchrotron powder diffraction data refinements (Pnma, a = 6.46281(2) angstrom, b = 3.86498(1) angstrom, c = 16.176:29(4) angstrom, Z = 4). One-dimensional Rb-87, La-139, and F-19 MAS NMR spectra have been recorded and are in agreement with the proposed structural model. Assignment of the F-19 resonances is performed on the basis of both F-19-La-139 J-coupling multiplet patterns observed in a heteronudear DQ-filtered J-resolved spectrum and F-19-Rb-87 HMQC MAS experiments. DFT calculations of both the F-19 isotropic chemical shieldings and the Rb-87, La-139 electric field gradient tensors using the GIPAW and PAW methods implemented in the CASTEP code are in good agreement with the experimental values and support the proposed structural model. Finally, the conductivity of RbLaF4 and luminescence properties of Eu-doped LaRbF4 are investigated.

Optical interference for the matching of the external and internal quantum efficiencies in organic photovoltaic cells
Betancur, R; Martinez-Otero, A; Elias, X; Romero-Gomez, P; Colodrero, S; Miguez, H; Martorell, J
Solar Energy Materials and Solar Cells, **104** (2012) 87-91
September, 2012 | **DOI:** 10.1016/j.solmat.2012.04.047

We demonstrate experimentally that an appropriate combination of the layer thicknesses in an inverted P3HT:PCBM cell leads to an optical interference such that the EQE amounts to 91% of IQE. We observe that reflectivity between layers is minimized in a wavelength range of more than 100 nm. In that range the EQE closely matches the IQE. The role played by the optical interference in improving the performance of the fabricated solar cells is confirmed by EQE calculated numerically using a model based on the transfer matrix method. Additionally, we observed that a similar cell with an active material 1.7 times thicker exhibited a lower PCE. The poor photon harvesting in the later cell configuration is attributed to an EQE that amounts only to 72% of the IQE.

Electrochromic Behavior of WxSiyOz Thin Films Prepared by Reactive Magnetron Sputtering at Normal and Glancing Angles

Gil-Rostra, J; Cano, M; Pedrosa, JM; Ferrer, FJ; Garcia-Garcia, F; Yubero, F; Gonzalez-Elipe, AR
ACS Applied Materials & Interfaces, **4** (2012) 628-638
February, 2012 | **DOI:** 10.1021/am2014629

This work reports the synthesis at room temperature of transparent and colored WxSiyOz thin films by magnetron sputtering (MS) from a single cathode. The films were characterized by a large set of techniques including X-ray photoelectron spectroscopy (XPS), Rutherford backscattering spectrometry (RBS), Fourier transform infrared (FT-IR), and Raman spectroscopies. Their optical properties were determined by the analysis of the transmission and reflection spectra. It was found that both the relative amount of tungsten in the W–Si MS target and the ratio O2/Ar in the plasma gas were critical parameters to control the blue coloration of the films. The long-term stability of the color, attributed to the formation of a high concentration of W5+ and W4+ species, has been related with the formation of W–O–Si bond linkages in an amorphous network. At normal geometry (i.e., substrate surface parallel to the target) the films were rather compact, whereas they were very porous and had less tungsten content when deposited in a glancing angle configuration. In this case, they presented outstanding electrochromic properties characterized by a fast response, a high coloration, a complete reversibility after more than one thousand cycles and a relatively very low refractive index in the bleached state.

Selective Detection of Volatile Organic Compounds by Spectral Imaging of Porphyrin Derivatives Bound to TiO2 Porous Films

Roales, J; Pedrosa, JM; Castillero, P; Cano, M; Richardson, TH; Barranco, A; Gonzalez-Elipe, AR
ACS Applied Materials & Interfaces, **4** (2012) 5147-5154
October, 2012 | **DOI:** 10.1021/am301069

In this work, the carboxylic acid derivatives of a free-base porphyrin, 5,10,15,20-tetrakis(4-carboxyphenyl)-21H,23H-porphyrin, and 10 of its metal derivatives (TCPPs) have been used for optical gas sensing. For this purpose, microstructured columnar TiO2 thin films prepared by GAPVD (glancing angle physical vapor deposition) have been used as host materials for the porphyrins as they are non–dispersive and porous, allowing their use for UV–visible spectroscopy and gas sensing. The chemical binding between the dye molecules and the TiO2 has been studied through infrared spectroscopy, and the obtained spectral changes have been found to be compatible with chelating and/or bidentate binding modes of the carboxylate groups on the TiO2 surface. When hosted in the film, the UV–visible spectra of the porphyrins featured a blue shift and broadening of the Soret band with respect to the solution, which has been attributed to the formation of π–π aggregates between porphyrin molecules. The composite porphyrin/TiO2 films obtained from each of the 11 porphyrins have been exposed to 12 different volatile organic compounds (VOCs), and their respective gas–sensitive properties have been analyzed as a function of the spectral changes in their Soret band region in the presence of the analytes. The set of composite films has shown high selectivity to the analyzed volatile compounds. For each analyte, an innovative way of showing the different responses has been developed. By means of this procedure, an imagelike recognition pattern has been obtained, which allows an easy identification of every compound. The kinetics of the exposure to several analytes showed a fast, reversible and reproducible response, with response times of a few seconds, which has been attributed to both the sensitivity of the porphyrins and the high porosity of the TiO2 films. Also, increasing concentrations of the analytes resulted in an increase in the magnitude of the response, indicating that the sensor behavior is also concentration-dependent.

Characterization of Mesoporous Thin Films by Specular Reflectance Porosimetry

Hidalgo, N; Lopez-Lopez, C; Lozano, G; Calvo, ME; Miguez, H
Langmuir, **28** (2012) 13777-13782
October, 2012 | **DOI:** 10.1021/la3025793

The pore size distribution of mesoporous thin films is herein investigated through a reliable and versatile technique coined specular reflectance porosimetry. This method is based on the analysis of the gradual shift of the optical response of a porous slab measured in quasi-normal reflection mode that occurs as the vapor pressure of a volatile liquid varies in a closed chamber. The fitting of the spectra collected at each vapor pressure is employed to calculate the volume of solvent contained in the interstitial sites and thus to obtain adsorption–desorption isotherms from which the pore size distribution and internal and external specific surface areas are extracted. This technique requires only a microscope operating in the visible range attached to a spectrophotometre. Its suitability to analyze films deposited onto arbitrary substrates, one of the main limitations of currently employed ellipsometric porosimetry and quartz balance techniques, is demonstrated. Two standard mesoporous materials, supramolecularly templated mesostructured films and packed nanoparticle layers, are employed to prove the concept proposed herein.

Following the Wetting of One-Dimensional Photoactive Surfaces

Macias-Montero, M; Borras, A; Alvarez, R; Gonzalez-Elipe, AR
Langmuir, **28** (2012) 15047-15055
October, 2012 | **DOI:** 10.1021/la3028918

This article aims toward a full description of the wetting conversion from superhydrophobicity to superhydrophilicity under illumination with UV light of high-density ZnO nanorods surfaces by (i) following the evolution of the clusters and superstructures formed by the nanocarpet effect as a function of the water contact angle (WCA); (ii) characterization of the superhydrophobic and superhydrophilic states with an environmental scanning electron microscope (ESEM); and (iii) using the nanocarpet effect as a footprint of both local and apparent water contact angles. Thus, the main objective of the article is to provide a general vision of the wettability of 1D photoactive surfaces. In parallel, the nanocarpet (NC) formation by clustering of vertically aligned ZnO nanorods (NR) when water is dripped on their surface and then dried is studied for the first time by taking advantage of the possibility of tuning the surface water contact angle of the ZnO NR structure under UV preillumination. As a result, we demonstrate the feasibility of controlling the size and other morphological characteristics of the NCs. Moreover, a strong anisotropic wetting behavior, characterized by a Δθ = θ – θ = 30°, is shown on an asymmetrically aligned NC surface resulting from arrays of tilted NRs. The study of the condensation/evaporation of water on/from an as-prepared (superhydrophobic) or a preilluminated (superhydrophilic) NR surface examined by an environmental scanning electron microscope has evidenced the formation of supported water droplets with polygonal shapes in the first case and the complete filling of the inter-NR space in the latter. The long-term stability of the NC clusters has been utilized as a footprint to track the penetration depth of water within the inter-NR space in the three borderline regions of water droplets. This analysis has shown that for moderately hydrophobic surfaces (i.e., water contact angles lower than 130°) water droplets do not present a well-defined borderline trace but a spreading region where water penetrates differently with the NR interspace. The transition from a Cassie–Baxter to a modified Cassie–Baxter to finish in a Wenzel wetting state is found on these surfaces depending on the UV preillumination time and is explained with a model where water interaction with the NR units is the critical factor determining the macroscopic wetting behavior of these surfaces.

Integration of Gold Nanoparticles in Optical Resonators
Jimenez-Solano, A; Lopez-Lopez, C; Sanchez-Sobrado, O; Luque, JM; Calvo, ME; Fernandez-Lopez, C; Sanchez-Iglesias, A; Liz-Marzan, LM; Miguez, H
Langmuir, **28** (2012) 9161-9167
June, 2012 | **DOI:** 10.1021/la300429k

The optical absorption of one-dimensional photonic crystal based resonators containing different types of gold nanoparticles is controllably modified by means of the interplay between planar optical cavity modes and localized surface plasmons. Spin-casting of metal oxide nanoparticle suspensions was used to build multilayered photonic structures that host (silica-coated) gold nanorods and spheres. Strong reinforcement and depletion of the absorptance was observed at designed wavelength ranges, thus proving that our method provides a reliable means to modify the optical absorption originated at plasmonic resonances of particles of arbitrary shape and within a wide range of sizes. These observations are discussed on the basis of calculations of the spatial and spectral dependence of the optical field intensity within the multilayers.

Correlation lengths, porosity and water adsorption in TiO2 thin films prepared by glancing angle deposition
Gonzalez-Garcia, L; Parra-Barranco, J; Sanchez-Valencia, JR; Barranco, A; Borras, A; Gonzalez-Elipe, AR; Garcia-Gutierrez, MC; Hernandez, JJ; Rueda, DR; Ezquerra, TA
Nanotechnology, **23** (2012) 205701
May, 2012 | **DOI:** 10.1088/0957-4484/23/20/205701

This paper reports a thorough microstructural characterization of glancing angle deposited (GLAD) TiO 2 thin films. Atomic force microscopy (afm), grazing-incidence small-angle x-ray scattering (GISAXS) and water adsorption isotherms have been used to determine the evolution of porosity and the existence of some correlation distances between the nanocolumns constituting the basic elements of the films nanostructure. It is found that the deposition angle and, to a lesser extent, the film thickness are the most important parameters controlling properties of the thin film. The importance of porosity and some critical dimensions encountered in the investigated GLAD thin films is highlighted in relation to the analysis of their optical properties when utilized as antireflective coatings or as hosts and templates for the development of new composite materials.

Vertical and tilted Ag-NPs@ZnO nanorods by plasma-enhanced chemical vapour deposition
Macias-Montero, M; Borras, A; Saghi, Z; Espinos, JP; Barranco, A; Cotrino, J; Gonzalez-Elipe, AR
Nanotechnology, 23 (2012) 255303
June, 2012 | DOI: 10.1088/0957-4484/23/25/255303

Supported ZnO nanorods have been prepared at 405 K by plasma-enhanced chemical vapour deposition (PECVD) using diethylzinc as precursor, oxygen plasma and silver as the promotion layer. The nanorods are characterized by a hollow and porous microstructure where partially percolated silver nanoparticles are located. By changing different deposition parameters like the thickness of the silver layer, the type of oxidation pretreatment or the geometry of the deposition set-up, the length, the width and the tilting angle of the nanorods with respect to the substrate can be modified. Other nanostructures like nanobushes, zigzag linear structures and stacked bilayers with nanocolumns of TiO 2 can also be prepared by adjusting the deposition conditions. A phenomenological model relying on the assessment of the diverse nanostructure morphologies and the evidence provided by an in situ x-ray photoelectron spectroscopy (XPS) experiment has been proposed to describe their formation mechanism. From this analysis it is deduced that the effect of the electrical field of the plasma sheath, the high mobility of silver and silver oxide, and the diffusion of the precursor molecules are some of the critical factors that must converge by the formation of the nanorods.

Effect of nanostructured electrode architecture and semiconductor deposition strategy on the photovoltaic performance of quantum dot sensitized solar cells
Samadpour, M; Gimenez, S; Boix, PP; Shen, Q; Calvo, ME; Taghavinia, N; Zad, AI; Toyoda, T; Miguez, H; Mora-Sero, I
Electrochimica Acta, **75** (2012) 139-147
July, 2012 | **DOI:** 10.1016/j.electacta.2012.04.087

Here we analyze the effect of two relevant aspects related to cell preparation on quantum dot sensitized solar cells (QDSCs) performance: the architecture of the TiO2 nanostructured electrode and the growth method of quantum dots (QD). Particular attention is given to the effect on the photovoltage, V oc, since this parameter conveys the main current limitation of QDSCs. We have analyzed electrodes directly sensitized with CdSe QDs grown by chemical bath deposition (CBD) and successive ionic layer adsorption and reaction (SILAR). We have carried out a systematic study comprising structural, optical, photophysical and photoelectrochemical characterization in order to correlate the material properties of the photoanodes with the functional performance of the manufactured QDSCs. The results show that the correspondence between photovoltaic conversion efficiency and the surface area of TiO2 depends on the QDs deposition method. Higher V oc values are systematically obtained for TiO 2 morphologies with decreasing surface area and for cells using CBD growth method. This is systematically correlated to a higher recombination resistance of CBD sensitized electrodes. Electron injection kinetics from QDs into TiO2 also depends on both the TiO2 structure and the QDs deposition method, being systematically faster for CBD. Only for electrodes prepared with small TiO2 nanoparticles SILAR method presents better performance than CBD, indicating that the small pore size disturb the CBD growth method. These results have important implications for the optimization of QDSCs.

The effect of nickel on alloy microstructure and electrochemical behaviour of AA1050 aluminium alloy in acid and alkaline solutions
Garcia-Garcia, FJ; Skeldon, P; Thompson, GE; Smith, GC
Electrochimica Acta, **75** (2012) 229-238
July, 2012 | **DOI:** 10.1016/j.electacta.2012.04.106

The study investigates the influence of nickel and magnesium additions to AA1050 aluminium alloy on the alloy electrochemical behaviour in sodium hydroxide and hydrochloric solutions under conditions relevant to industries that use alkaline etching as a standard surface treatment procedure and to the lithographic and electronic industries where surface convolution is assisted by pitting in hydrochloric acid. Scanning and transmission electron microscopes were used to characterize the intermetallic particles, and scanning Kelvin probe microscopy was utilised in monitoring the surface potential. Nickel is shown to be incorporated into second phase particles, which mostly consisted of Al3Fe and α-(AlFeSi) phases, resulting in enhanced cathodic activity on the aluminium surface. Consequently, the dissolution rates of the superpure aluminium, alloys without nickel addition and alloy with nickel addition are increased respectively in sodium hydroxide, and increased pitting is respectively promoted in hydrochloric acid. In contrast, the addition of magnesium to the alloy had negligible influence on the etching and pitting behaviour.

Quantum-Mechanical Study on the Aquaions and Hydrolyzed Species of Po(IV), Te(IV), and Bi(III) in Water
Ayala, Regla; Manuel Martinez, Jose; Pappalardo, Rafael R.; Sanchez Marcos, Enrique
Journal of Physical Chemistry B, **113** (2009) 487-496
December, 2012 | **DOI:** 10.1021/jp309439f

A systematic study of [M(H2O)n(OH)m]q+ complexes of Te(IV) and Bi(III) in solution has been undertaken by means of quantum mechanical calculations. The results have been compared with previous information obtained for the same type of Po(IV) complexes (J. Phys. Chem. B 2009, 113, 487) to get insight into the similarities and differences among them from a theoretical view. The evolution of the coordination number (n + m) with the degree of hydrolysis (m) for the stable species shows a systematic decrease regardless the ion. A general behavior on the M–O distances when passing from the gas phase to solution, represented by the polarizable continuum model (PCM), is also observed: RM–O values corresponding to water molecules decrease, while those of the hydroxyl groups slightly increase. The hydration numbers of aquaions are between 8 and 9 for the three cations, whereas hydrolyzed species behave differently for Te(IV) and Po(IV) than for Bi(III), which shows a stronger trend to dehydrate with the hydrolysis. On the basis of the semicontinuum solvation model, the hydration Gibbs energies are −800 (exptl −834 kcal/mol), −1580 and −1490 kcal/mol for Bi(III), Te(IV), and Po(IV), respectively. Wave function analysis of M–O and O–H bonds along the complexes has been carried out by means of quantum theory of atoms in molecule (QTAIM). Values of electron density and its Laplacian at bond critical points show different behaviors among the cations in aquaions. An interesting conclusion of the QTAIM analysis is that the prospection of the water O–H bond is more sensitive than the M–O bond to the ion interaction. A global comparison of cation properties in solution supplies a picture where the Po(IV) behavior is between those of Te(IV) and Bi(III), but closer to the first one.

Critical thickness and nanoporosity of TiO2 optical thin films

Borras, A; Alvarez, R; Sanchez-Valencia, JR; Ferrer, J; Gonzalez-Elipe, AR
Microscopy and Microanalysis, **18** (2012) 1-9
September, 2012 | **DOI:** 10.1016/j.micromeso.2012.04.035

This work reports on the porosity and refraction index of TiO2 thin films as a function of the film thickness. Samples were fabricated by plasma enhanced chemical vapor deposition (PECVD) in a microwave electron cyclotron resonance (MW-ECR) reactor at room temperature using titanium tetra-isopropoxide (MP) as precursor. Experimental parameters such as plasma gas composition (pure oxygen and argon/oxygen mixtures) and pressure (either ECR conditions or "normal" pressure, i.e. 10(-4) or 10(-3) torrs correspondently) were varied. The evolution of the thin film microstructure, porosity and optical properties is critically studied by AFM, SEM, water adsorption isotherms, ellipsometry and UV-Vis transmittance and the existence of a certain critical thickness (t(c)) demonstrated. The porosity of the films with thicknesses ranging from several tens of nanometers up to half a micrometer is evaluated by QCM-isotherms at room temperature. The dependency of this critical thickness with the plasma conditions is evaluated experimental and theoretically. Thus, the microstructure change at t(c) is attributed to a transition from a surface diffused dominated growth mechanism for t < t(c) to another where shadowing is predominant. Dynamic scaling analysis of the two regimes and their Monte Carlo simulation complete the reported study.

Roughness assessment and wetting behavior of fluorocarbon surfaces

Terriza, A; Alvarez, R; Borras, A; Cotrino, J; Yubero, F; Gonzalez-Elipe, AR
Journal of Colloid and Interface Science, **376** (2012) 274-282
June, 2012 | **DOI:** 10.1016/j.jcis.2012.03.010

The wetting behavior of fluorocarbon materials has been studied with the aim of assessing the influence of the surface chemical composition and surface roughness on the water advancing and receding contact angles. Diamond like carbon and two fluorocarbon materials with different fluorine content have been prepared by plasma enhanced chemical vapor deposition and characterized by X-ray photoemission, Raman and FT-IR spectroscopies. Very rough surfaces have been obtained by deposition of thin films of these materials on polymer substrates previously subjected to plasma etching to increase their roughness. A direct correlation has been found between roughness and water contact angles while a superhydrophobic behavior (i.e., water contact angles higher than 150° and relatively low adhesion energy) was found for the films with the highest fluorine content deposited on very rough substrates. A critical evaluation of the methods currently used to assess the roughness of these surfaces by atomic force microscopy (AFM) has evidenced that calculated RMS roughness values and actual surface areas are quite dependent on both the scale of observation and image resolution. A critical discussion is carried out about the application of the Wenzel model to account for the wetting behavior of this type of surfaces.

Tuning liver stiffness against tumours: An in vitro study using entrapped cells in tumour-like microcapsules

Leal-Egana, A; Fritsch, A; Heidebrecht, F; Diaz-Cuenca, A; Nowicki, M; Bader, A; Kas, J
Journal of the mechanical behavior of biomedical materials, **9** (2012) 113-121
May, 2012 | **DOI:** 10.1016/j.jmbbm.2012.01.013

Liver fibrosis is a reversible pathology characterized by the up-regulated secretion and deposition of ECM proteins and inhibitors of metalloproteinases, which increase the stiffness and viscosity of this organ. Since recent studies have shown that fibrosis preceded the generation of hepatocellular carcinomas, we hypothesize that liver fibrosis could play a role as a mechanism for restricting uncontrolled cell proliferation, inducing the mortality of cancer cells and subsequent development of primary tumours. With this purpose, in this work we analysed in vitro how the modulation of stiffness can influence proliferation, viability and aggregation of hepatocarcinoma cells (HepG(2)) embedded in 3D micromilieus mimicking values of elasticity of fibrotic liver tissues. Experiments were performed by immobilizing up to 10 HepG(2) cells within microcapsules made of 0.8%, 1.0% and 1.4% w/v alginate which, besides having values of elasticity from the lower-healthy to the upper-fibrotic range liver tissues, lacked domains for proteases, mimicking the micromilieu existing in hepatic primary tumours. Our results show that entrapped cells exhibited a short duplication phase followed by an irreversible decay stage, in which cell mortality could be mediated by two mechanisms: mechanical stress, in the case of cells entrapped in a stiffer micromilieu; and mass transfer limitations produced by pore coarsening at the interface cell-matrix, in softer micromilieus.
According to the authors' knowledge, this work represents the first attempt to elucidate the role of liver fibrosis during Hepatocarcinoma pathologies, suggesting that the generation of a non-biodegradable and mechanically unfavourable environment surrounding cancer cells could control the proliferation, migration of metastatic cells and the subsequent development of primary tumours.

Charge collection properties of dye-sensitized solar cells based on 1-dimensional TiO2 porous nanostructures and ionic-liquid electrolytes

Gonzalez-Garcia, Lola; Idigoras, Jesus; Gonzalez-Elipe, Agustin R.; Barranco, Angel; Anta, Juan A.
Journal of Photochemistry and Photobiology A-Chemistry, **241** (2012) 58-66
August, 2012 | **DOI:** 10.1016/j.jphotochem.2012.05.015

Dye solar cells consisting of mesoporous TiO 2 nanocolumnar films sensitized with a highly absorptive indoline dye were studied to estimate the charge collection efficiency provided by porous 1-dimensional (1-D) nanostructures in combination with viscous, fast-recombining electrolytes. The TiO 2 mesoporous nanostructured films were prepared by physical vapor deposition at glancing incidence (GLAD-PVD). Electrochemical Impedance Spectroscopy (EIS) in the dark was utilized to extract the transport and recombination properties of the fabricated devices. Due to their high porosity, the TiO 2 nanocolumnar electrodes incorporated a dye amount similar to that admitted by nanoparticulated electrodes with higher thickness. This fact, together with the longer lifetimes of electrons obtained for the GLAD-PVD electrodes, lead to an overall improvement of the charge collection and photovoltaic properties with respect to randomly packed electrodes. Measured diffusion lengths were improved by a factor between 2 and 3 with respect to the disordered nanostructure. The present results demonstrate the capability of partially ordered nanostructures to improve charge collection in devices constructed with fast-recombining electrolytes.

Structural and kinetic study of phase transitions in LaYSi2O7
Fernandez-Carrion, AJ; Escudero, A; Suchomel, MR; Becerro, AI
Journal of the European Ceramic Society, **32** (2012) 2477-2486
August, 2012 | **DOI:** 10.1016/j.jeurceramsoc.2012.03.009

Phase transitions in LaYSi 2O7 have been investigated as a function of temperature using XRD, NMR and TEM. Previously described empirical crystal structure guidelines based on average cation radius in rare-earth RE 2Si2O7-type disilicates predict a stable tetragonal A-LaYSi2O7 polymorph at temperatures below 1500°C. This study demonstrates that A-LaYSi2O7 is not thermodynamically stable at these temperatures and suggests that guidelines based on average cation size do not accurately describe the equilibrium behaviour of this silicate system. The A to G-type polymorph transition is extremely sluggish; complete transformation requires ~250h at 1200°C, and more than 3 weeks of calcination at 1100°C. This observation is important when this type of material is used as environmental barrier coating (EBC) of advanced ceramics. Analysis of XRD and TEM data reveal complete substitution of Y and La on the rare-earth cation sites in both LaYSi2O7 polymorphs, but indicate preferential site occupancies in the G-type polymorph.

Aluminum incorporation in alpha-PbO2 type TiO2 at pressures up to 20 GPa
Escudero, A; Langenhorst, F
Physics of the Earth and Planetary Interiors, **190** (2012) 87-94
January, 2012 | **DOI:** 10.1016/j.pepi.2011.11.002

Aluminum incorporation into the high pressure polymorph of TiO2 with the structure of alpha-PbO2 has been studied from 10 to 20 GPa and 1300 degrees C by XRD, high-resolution Al-27 MAS-NMR and TEM. Al-doped alpha-PbO2 type TiO2 can be recovered at atmospheric pressure. Al2O3 solubility in alpha-PbO2 type TiO2 increases with increasing the synthesis pressure. The alpha-PbO2 type TiO2 polymorph is able to incorporate up to 35 wt.% Al2O3 at 13.6 GPa and 1300 degrees C, being the substitution of Ti4+ by Al3+ on normal octahedral sites and the formation of oxygen vacancies the mechanism of solubility. The transition to the higher pressure TiO2 polymorph with the ZrO2 baddeleyite structure, akaogiite, has not been observed in the quenched samples at room pressure. The microstructure of the recovered sample synthesized at 16 GPa and 1300 degrees C points to the existence of an intermediate non-quenchable aluminum titanium oxide phase at these conditions.

Aluminum solubility in TiO2 rutile at high pressure and experimental evidence for a CaCl2-structured polymorph
Escudero, A; Langenhorst, F; Muller, WF
American Mineralogist, **97** (2012) 1075-1082
July, 2012 | **DOI:** 10.2138/am.2012.4049

Aluminum incorporation into TiO2 has been studied in the TiO2-Al2O3 system as a function of pressure at temperatures of 900 and 1300 °C using commercial Al2TiO5 nanopowder as starting material. A new orthorhombic TiO2 polymorph with the CaCl 2 structure has been observed in the recovered samples synthesized from 4.5 to 7 GPa and 900 °C and from 2.5 to 7 GPa at 1300 °C. The phase transition to the α-PbO2 type TiO2 phase takes place between 7 and 10 GPa at both temperatures. Two mechanisms of Al incorporation in TiO2 rutile have been observed in the recovered samples. The substitution of Ti 4+ by Al 3+ on normal octahedral sites is dominant at lower pressures. High pressure induces the incorporation of Al3+ into octahedral interstices of the rutile structure, which is responsible for an orthorhombic distortion of the TiO2 rutile structure and gives rise to a (110) twinned CaCl2 type structure. This phase is probably a result of temperature quench at high pressure. Aluminum solubility in TiO2 increases with increasing pressure. TiO2 is able to accommodate up to 9.8 wt% Al2O3 at 7 GPa and 1300 °C. Temperature has a large effect on the aluminum incorporation in TiO2, especially at higher pressures. High pressure has a strong effect on both the chemistry and the microstructure of Al-doped TiO2. Enhanced aluminum concentration in TiO2 rutile as well as TiO2 grains with a microstructure consisting of twins are a clear indication of high-pressure conditions.

Incorporation of Si into TiO2 phases at high pressure
Escudero, A; Langenhorst, F
American Mineralogist, **97** (2012) 524-531
April, 2012 | **DOI:** 10.2138/am.2012.3941

Silicon incorporation in TiO2 phases at increasing pressures until 20 GPa at 1300 °C has been studied by XRD and TEM. Rutile is the stable Si-doped TiO2 phase until at least 7 GPa, transforming into α-PbO2 structured TiO2 between 7 and 10 GPa. The further transformation to the TiO2 polymorph with the baddeleyite structure, akaogiite, has not been observed on the quenched samples. XRD and TEM-EDX data suggest that the Si-doped TiO2 akaogiite polymorph is non-quenchable and reverts to a-PbO2 structured TiO2 when releasing the pressure. This transformation gives rise to α-PbO2 structured TiO2 grains decorated with p fringes stacking faults. Silicon solubility in TiO2 phases increases with increasing the synthesis pressure until 16 GPa, implying the substitutional solid solution to be the mechanism of solubility. The influence of the dopants on the stability of the rutile and the α-PbO2 structured TiO2 has also been analyzed.

Influence of plasma-generated negative oxygen ion impingement on magnetron sputtered amorphous SiO2 thin films during growth at low temperatures
Macias-Montero, M; Garcia-Garcia, FJ; Alvarez, R; Gil-Rostra, J; Gonzalez, JC; Cotrino, J; Gonzalez-Elipe, AR; Palmero, A
Journal of Applied Physics, **111** (2012)
March, 2012 | **DOI:** 10.1063/1.3691950

Growth of amorphous SiO2 thin films deposited by reactive magnetron sputtering at low temperatures has been studied under different oxygen partial pressure conditions. Film microstructures varied from coalescent vertical column-like to homogeneous compact microstructures, possessing all similar refractive indexes. A discussion on the process responsible for the different microstructures is carried out focusing on the influence of (i) the surface shadowing mechanism, (ii) the positive ion impingement on the film, and (iii) the negative ion impingement. We conclude that only the trend followed by the latter and, in particular, the impingement of O- ions with kinetic energies between 20 and 200 eV, agrees with the resulting microstructural changes. Overall, it is also demonstrated that there are two main microstructuring regimes in the growth of amorphous SiO2 thin films by magnetron sputtering at low temperatures, controlled by the amount of O2 in the deposition reactor, which stem from the competition between surface shadowing and ion-induced adatom surface mobility.

Chromium incorporation into TiO2 at high pressure
Escudero, A; Langenhorst, F
Journal of Solid State Chemistry, **190** (2012) 61-67
June, 2012 | **DOI:** 10.1016/j.jssc.2012.01.041

Chromium incorporation into TiO 2 up to 3 GPa at 1300 °C and 900 °C has been studied by XRD as well as TEM. A CaCl 2 type TiO 2 polymorph has been observed in the quenched samples from high pressure. Two different mechanisms of solubility occur in the recovered samples. Chromium replaces titanium on normal octahedral sites but it also occupies interstitial octahedral sites, especially in the samples recovered from higher pressures. Interstitial chromium is responsible for an orthorhombic distortion of the TiO 2 rutile structure in the quenched samples and gives rise to a (1 1 0) twinned CaCl 2-structured polymorph. This phase is very likely the result of temperature quench at high pressure. The formation of this phase is directly related to the chromium content of the TiO 2 grains. Chromium solubility in TiO 2 increases with increasing the synthesis pressure. TiO 2 is able to accommodate up to 15.3 wt% Cr 2O 3 at 3 GPa and 1300 °C, compared to 5.7 wt% at atmospheric pressure at the same temperature.

DLC coatings for UHMWPE: Relationship between bacterial adherence and surface properties
Del Prado, G; Terriza, A; Ortiz-Perez, A; Molina-Manso, D; Mahillo, I; Yubero, F; Puertolas, JA; Manrubia-Cobo, M; Barrena, EG; Esteban, J
Journal of Biomedical Materials Research Part A, **100A** (2012) 2813-2820
October, 2012 | **DOI:** 10.1002/jbm.a.34220

Development of intrinsically antibacterial surfaces is of key importance in the context of prostheses used in orthopedic surgery. This work presents a thorough study of several plasma-based coatings that may be used with this functionality: diamond-like carbon (DLC), fluorine-doped DLC (F-DLC), and a high-fluorine-content-carbon-fluor polymer (CFX). The coatings were obtained by a radio-frequency plasma-assisted deposition on ultra high molecular weight polyethylene (UHMWPE) samples and physicochemical properties of the coated surfaces were correlated with their antibacterial performance against collection and clinical Staphylococcus aureus and Staphylococcus epidermidis strains. The fluorine content and the relative amount of CC and CF bonds were controlled by X-ray photoelectron spectroscopy, and hydrophobicity and surface tension by contact angle measurements. Surface roughness was studied by Atomic Force Microscopy. Additional nanoidentation studies were performed for DLC and F-DLC coatings. Unpaired t test and regression linear models evaluated the adherence of S. aureus and S. epidermidis on raw and coated UHMWPE samples. Comparing with UHMWPE, DLC/UHMWPE was the least adherent surface with independence of the bacterial species, finding significant reductions ($p \leq 0.001$) for nine staphylococci strains. Bacterial adherence was also significantly reduced in F-DLC/ UHMWPE and CFx/UHMWPE for six strains. © 2012 Wiley Periodicals, Inc. J Biomed Mater Res Part A 100A:2813–2820, 2012.

Wall slip phenomena in concentrated ionic liquid-based magnetorheological fluids

Gomez-Ramirez, A; Lopez-Lopez, MT; Gonzalez-Caballero, F; Duran, JDG

Rheologica Acta, **51** (2012) 793-803
September, 2012 | **DOI:** 10.1007/s00397-012-0639-5

Ionic liquids (ILs) have been recently proposed as carrier for magnetorheological (MR) fluids. Their special properties, such as very low vapor pressure and high thermal stability, make ILs highly suitable dispersion media to increase the broad range of technological applications that magnetorheological fluids already have. It has been just reported that using ILs as carriers in MR fluids an improvement in the colloidal stability and suspension redispersibility is obtained. In this work, the magnetorheological behavior of highly concentrated suspensions in ILs is studied. Two kinds of suspensions were analyzed: using an ionic liquid of low conductivity and a mineral oil as carriers. In both cases, silica-coated iron microparticles were used as solid phase, being the solid volume concentration of 50% vol. A complete magnetorheological analysis focused on the wall slip phenomenon was performed. Steady-state and oscillatory experiments were carried out. In order to study wall slip effects, all experiments were performed with a plate-plate system, using both smooth and rough measuring surfaces. A significant effect of wall slip was observed when the experiments were performed using smooth surfaces. The novelty of this paper is mainly based on (1) the use of an ionic liquid as carrier to prepare magnetic suspensions, and (2) the analysis of wall slip phenomena in MR fluids with a particle content close to the maximum packing fraction.

Attenuation lengths of high energy photoelectrons in compact and mesoporous SiO2 films

Ferrer, FJ; Gil-Rostra, J; Gonzalez-Garcia, L; Rubio-Zuazo, J; Romero-Gomez, P; Lopez-Santos, MC; Yubero, F

Surface Science, **606** (2012) 820-824

May, 2012 | **DOI:** 10.1016/j.susc.2012.01.017

We have experimentally evaluated attenuation lengths (AL) of photoelectrons traveling in compact and micro and mesoporous (~ 45% voids) SiO2 thin films with high (8.2-13.2 keV) kinetic energies. The films were grown on polished Si(100) wafers. ALs were deduced from the intensity ratio of the Si1s signal from the SiO2 film and Si substrate using the two-peaks overlayer method. We obtain ALs of 15-22 nm and 23-32 nm for the compact and porous SiO2 films for the range of kinetic energies considered. The observed AL values follow a power law dependence on the kinetic energy of the electrons where the exponent takes the values 0.81 ± 0.13 and 0.72 ± 0.12 for compact and porous materials, respectively.

Analysis of multifunctional titanium oxycarbide films as a function of oxygen addition

Chappe, JM; Fernandes, AC; Moura, C; Alves, E; Barradas, NP; Martin, N; Espinos, JP; Vaz, F

Surface and Coatings Technology, **206** (2012) 2525-2534
January, 2012 | **DOI:** 10.1016/j.surfcoat.2011.11.005

Reactive magnetron sputtering was used to deposit titanium oxycarbide thin films. The overall set of results showed that the oxygen flow rate, and thus the composition of the atmosphere in the deposition chamber, controls the composition of the titanium oxycarbide thin films obtained by reactive sputtering. Rutherford Backscattering Spectroscopy analysis revealed the existence of three major types of films, indexed to their particular composition ratios. A detailed study by X-ray photoelectron spectroscopy was carried out in order to characterize the evolution of the TiC, CO and CC bonds induced by the increase of the oxygen partial pressure, which was found to be closely related with the different zones of composition that were suggested. Micro-Raman spectroscopy and X-ray diffraction measurements allowed describing the complex nature of the film structure, namely in what concerns different phases and their evolution, texture phenomena and grain size evolution as a function of the particular composition and film types (different zones). Electrical conductivity revealed a transition from a metallic to a semi-conducting behavior as a function of the oxygen concentration in the films, in good agreement with the different zones that were suggested. Similarly, optical properties supported this gradual change and for oxygen contents higher than 67 at.%, the films exhibited typical reflectance of insulator materials (interferences) in the UV, visible and near IR regions.

XPS and AES analyses of cerium conversion coatings generated on AA5083 by thermal activation

Sanchez-Amaya, JM; Blanco, G; Garcia-Garcia, FJ; Bethencourt, M; Botana, FJ

Surface and Coatings Technology, **213** (2012) 105-116
December, 2012

This paper describes the deep analysis of cerium conversion coatings developed with thermal activation on AA5083 under optimum processing conditions. Scanning electron microscopy (SEM), electron dispersive spectroscopy (EDX), X-ray photoelectron spectroscopy (XPS) and Auger electron spectroscopy (AES) were employed to study these layers. Ar+ sputtering was also employed to analyse the coatings' core. Although conversion coatings based on Ce salts have been widely characterised in the literature for different aluminium alloys, the coatings

developed with thermal activation on Al–Mg alloys have not been previously investigated with these techniques. SEM/EDX studies have demonstrated the existence of a heterogeneous layer formed by a film of aluminium oxide/hydroxide on the matrix as well as a series of dispersed islands of cerium deposited on the cathodic intermetallics. These results have been further confirmed by means of XPS. The XPS and AES results revealed that the outer layer comprises a mixture coating of Ce3 + (70%) and Ce4 + (30%) compounds. Although only Ce3 + compounds were detected at the inner part of the coating, possible reduction of Ce(IV) to Ce(III) due to the Ar+ beam could not be discarded. Obtained results allowed authors to confirm that the cerium conversion coatings developed have a similar structure to those previously reported for other aluminium alloys.

Characterization of highly crosslinked polyethylenes by colorimetry
Martinez-Morlanes, MJ; Terriza, A; Yubero, F; Puertolas, JA
Polymer Testing, **31** (2012) 841-847
September, 2012 | **DOI:** 10.1016/j.polymertesting.2012.06.005

Colour analysis appears to be a robust technique for characterizing vitamin E doping and gamma irradiation of medical grade polyethylene samples. The analysis procedure described in this paper is of great interest because it can easily distinguish between polyethylene samples with differences in vitamin E (α-tocopherol) doping of about 0.1 wt% and gamma irradiation doses of 30 kGy. It is found that the colour differences (with respect to untreated samples) induced by gamma irradiation and/or vitamin E doping add-up linearly. This method for detecting the presence of vitamin E is fast, simple and non-destructive.

Novel approaches to flexible visible transparent hybrid films for ultraviolet protection
Calvo, ME; Smirnov, JRC; Miguez, H
Journal of Polymer Science Part B-Polymer Physics, **50** (2012) 945-956
July, 2012 | **DOI:** 10.1002/polb.23087

Herein, we present an overview of the most recent achievements and innovations regarding the development of flexible visible transparent films for selective ultraviolet (UV) shielding, with focus on those based on hybrid inorganic-organic materials. The main synthetic paths used nowadays to ensure a high degree of protection are reviewed. Polymers containing organic UV absorbing molecules, hybrid mixtures of polymers and nanoparticles, and the recently introduced series of structures displaying structural color, are identified as the three main types of materials used for this purpose. The use of biocompatible and flexible films to achieve spectrally selective UV protection can find applications in a wide diversity of fields such as photo-treatment of skin diseases, food and beverage packing, and storage of cosmetics. © 2012 Wiley Periodicals, Inc. J Polym Sci Part B: Polym Phys, 2012 In this review, the different approaches taken to obtain flexible and transparent films that block ultraviolet radiation based on the use of hybrid materials are covered. The synthetic pathways that lead to films that can shield against UV radiation either by absorption or by interference are described.

Microstructure, composition and P-T conditions of rutile from diamondiferous gneiss of the Saxonian Erzgebirge, Germany
Escudero, A; Miyajima, N; Langenhorst, F
Chemie der erde-geochemistry, **72** (2012) 25-30
January, 2012 | **DOI:** 10.1016/j.chemer.2011.11.001

The chemical composition and microstructure of rutile grains in a ultra-high pressure metamorphic gneiss of the Saxonian Erzgebirge, Germany have been studied by Raman spectroscopy, SEM, EMPA and TEM. Rutile inclusions in garnet contain free dislocations, iron-enriched dislocations and exsolved ilmenite lamellae, while subgrain boundaries are observed in rutile grains of the rock matrix. The previously reported alpha-PbO_2 type TiO_2 phase could not be confirmed by our TEM observations. On the basis of Zr solubility in the rutile and the presence of microdiamonds, minimum metamorphic peak conditions of 3.95 GPa and 915 degrees C are estimated.

Quantification of low levels of fluorine content in thin films
Ferrer, FJ; Gil-Rostra, J; Terriza, A; Rey, G; Jimenez, C; Garcia-Lopez, J; Yubero, F
Nuclear Instruments & Methods in Physics Research Section B-Beam Interactions with Materials and Atoms, **274** (2012) 65-69
March, 2012 | **DOI:** 10.1016/j.nimb.2011.11.042

Fluorine quantification in thin film samples containing different amounts of fluorine atoms was accomplished by combining proton-Rutherford Backscattering Spectrometry (p-RBS) and proton induced gamma-ray emission (PIGE) using proton beams of 1550 and 2330 keV for p-RBS and PIGE measurements, respectively. The capabilities of the proposed quantification method are illustrated with examples of the analysis of a series of samples of fluorine-doped tin oxides, fluorinated silica, and fluorinated diamond-like carbon films. It is shown that this procedure allows the quantification of F contents as low as 1 at.% in thin films with thicknesses in the 100-400 nm range.

Software package to calculate the effects of the core hole and surface excitations on XPS and AES
Tougaard, S; Yubero, F
Surface and Interface Analysis, **44** (2012) 1114-1118
August, 2012 | **DOI:** 10.1002/sia.4855

We report on a new software package that allows to calculate the energy loss processes in a photo- and Auger electron spectrum. The calculations are performed within our previously published semiclassical dielectric response model. The model takes into account energy loss, which takes place because of the sudden creation of the static core hole and as the photoelectron travels in the bulk, passes the surface region and continues in the vacuum where it interacts with its image charge before it ends up in the electron spectrometer. It is a one-step model, which includes interference effects between these excitations. The only input in the calculations is the dielectric function of the material. We discuss the capabilities of the software and illustrate some examples of its practical application, including comparison with experimental spectra. We hope the software will be useful for the investigations of fundamental excitation mechanisms in XPS and AES. The software is free for noncommercial use.

LIBROS Y OTRAS PUBLICACIONES / BOOKS AND OTHER PUBLICATIONS

Optical emission spectroscopic evaluation of different microwave plasma discharges and its potential application for sterilization processes
J.L. Hueso; V.J. Rico; A. Yanguas-Gil; J. Cotrino; A.R. González-Elipe
En: Plasma for Bio-Decontamination, Medicine and Food Security Plasma for Bio-Decontamination, Medicine and Food Security (Editorial Springer), pp. 121-132
ISBN: 978-94-007-2851-6

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

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

SPIE Photonics Europe 2012

16 – 19 abril [Bruselas, Bélgica]
Hernán R.Míguez García [Chair, Chair Session]

COMUNICACIONES EN CONGRESOS Y REUNIONES / COMMUNICATIONS IN CONGRESS AND MEETINGS

SPIE Photonics Europe 2012

16 – 19 abril [Bruselas, Bélgica]

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

Optical field confinement effects on the absorption of compounds embedded periodic nanostructured multilayers
Mauricio E. Calvo, Miguel Anaya, Gabriel Lozano, Hernán R. Míguez, Olalla Sánchez-Sobrado, Luis M. Liz-Marzán, Ana M. Sánchez-Iglesias, José Miguel Luque
Poster

International Workshop on Advanced Processes in Optical Sensing and Photonic Applications

21 – 25 mayo [Madrid, España]

Nanoporous and mesoporus photonic multilayers for applications in sensing
H. Míguez
Conferencia Invitada

SimOEP12: International Conference on Simulation of Organic Electronics and Photovoltaics

10 – 14 junio [Valencia, España]

Modelling the effect of different optical designs on the performance of dye sensitized solar cells
H. Míguez
Conferencia Invitada

1st Al-Nanofunc Workshop: Advanced Microstructural Characterization of Nanomaterials

5 – 6 julio [Sevilla, España]

Photonic Crystals for Enhanced Light Harvesting in Dye Solar Cells.
C. López-López; S. Colodrero; H. Míguez
Comunicación oral

Controllable synthesis and luminescence properties of GdPO4- based nanophosphors
Becerro A.I.; Ocaña M.
Poster

Tailored luminescent emission of dyes embedded in porous resonators
A. Jiménez-Solano; J. M. Luque; M. E. Calvo; F. Fernández-Lázaro; H. Míguez
Poster

Spray pyrolisis synthesis of A-La2Si2O7: Crystal structure and luminescence
A.J. Fernández-Carrión; M. Ocaña; A.I. Becerro
Poster

Biomimetic nano-mineralization of porous gelatin scaffolds for Bone Tissue-Engineering
S. Borrego-González; M.L. Ramiro-Gutiérrez; A. Díaz-Cuenca
Poster

Nano-Porous-Ordered Bioactive Scaffolds for Hard Tissue Engineering
M.L. Ramiro-Gutiérrez; A. Díaz-Cuenca
Poster

16th International Symposium on Small Particles and Inorganic Clusters (ISSPIC XVI)

8 – 13 julio [Leuven, Bélgica]

Microwave-assisted synthesis and luminescence of mesoporous Ln-doped YPO4 (Ln=Eu, Ce, Tb and Ce Tb) nanophosphors with lenticular shape
S. Rodriguez-Liviano; F. J. Aparicio; T.C. Rojas; A.B. Hungría; L.E. Chinchilla; M. Ocaña
Poster

13th Edition of Trends in Nanotechnology International Conference (TNT2012)

10 – 14 septiembre [Madrid, España]

Green synthesis of silver nanoparticles mediated by bee products.
R. Mendoza-Reséndez; N. O. Nuñez; C. Luna
Poster

Optical Nanostructures and Advanced Materials for Photovoltaics (PV), part of Renewable Energy and the Environment Conference series of the Optical Society of America

12 noviembre [Paises Bajos]

Integration of Periodic Optical Nanostructures in Dye Sensitized Solar Cells: Towards Efficient and Transparent Cells
Hernán Míguez
Conferencia Invitada

Diffuse Scattering Designs of Dye Solar Cells
Hernán Míguez
Conferencia Invitada

13th International Conference on Plasma Surface Engineering (PSE 2012)

15 – 19 septiembre [Garmisch-Partenkinchen, Alemania]

Growth regimes of microstructured gold thin films deposited by magnetron sputtering at oblique angles and at low temperatures
R. Álvarez; J. M. García-Martín; M. Macías-Montero; L. González-García; J.C. González; P. Romero-Gómez; J. Cotrino; A.R. González-Elipe; A. Palmero
Poster

CONGRESOS Y REUNIONES NACIONALES / NATIONAL CONGRESS AND MEETINGS

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

Conferencia Española de Nanofotonica, CEN 2012

1 – 4 octubre [Sevilla, España]
Hernán R.Míguez García [Chair]

COMUNICACIONES EN CONGRESOS Y REUNIONES / COMMUNICATIONS IN CONGRESS AND MEETINGS

VII Reunión grupo Especializado de Física de Estado Sólido (GEFES)

25 – 27 enero [Sevilla, España]

Tailored luminescent emission of dyes embedded in porous resonators
A. Jiménez-Solano; J. M. Luque; M. E. Calvo; F. Fernández-Lázaro; H. Míguez
Poster

XXII Simposio del grupo especializado de Cristalografía y crecimiento cristalino

26 – 29 junio [Sevilla, España]

Apparent bulk thermal expansion coefficient for rare earth disilicates
A.J. Fernández-Carrión; M. Allix; A.I. Becerro
Poster

Conferencia Española de Nanofotonica, CEN 2012

1 – 4 octubre [Sevilla, España]

Microwave assisted synthesis of PAA functionalised europium doped calcium hydroxyapatite and fluoroapatite nanospindles for biomedical applications
Alberto Escudero, Jesús M de la Fuente, Manuel Ocaña
Comunicación oral

Multicolor and white light emission of GdPO4 nanophosphors with novel 3D architecture
Ana I. Becerro; Sonia Rodriguez-Liviano; Alberto J. Fernandez-Carrión; Manuel Ocaña
Comunicación oral

Optimized integration of one-dimensional photonic crystals in dye solar cells
C. López-López; S. Colodrero; H. Míguez
Poster

Integration of gold nanoparticles in photonic crystals: effect of the interplay between plasmonic and optical cavity resonances
Alberto Jiménez-Solano; Carmen López-López; Olalla Sánchez-Sobrado; José Miguel Luque; Mauricio E. Calvo; Cristina Fernández-López; Ana Sánchez-Iglesias; Luis M. Liz-Marzán; Hernán Míguez
Poster

FORMACION / TRAINING

TESIS DOCTORALES/ DOCTOR DEGREE THESIS

Título:	**Aplicaciones de láminas con mesoestructura controlada en cristales fotónicos**
Autor:	Nuria Hidalgo Serrano
Directores:	Hernán Míguez García
Calificación:	Apto “Cum Laude”
Centro:	Universidad de Sevilla

PREMIOS Y RECONOCIMIENTOS / PRIZES AND ACKNOWLEDGEMENTS

Premio Extraordinario de Doctorado de la Universidad de Sevilla a **Gabriel Lozano García** por el trabajo de Tesis Doctoral titulado “Análisis del crecimiento y las propiedades ópticas en alta energía de cristales fotónicos coloidades”. Dirigida por el Investigador Científico del CSIC Hernán Míguez García. Esta investigación se ha centrado en el estudio del mecanismo de autoensamblado por deposición vertical y de las propiedades ópticas que exhiben los materiales fotónicos fabricados mediante esta técnica. Estos materiales, conocidos como ópalos artificiales, se emplean para el estudio fundamental de la interacción de la luz con estructuras tridimensionalmente periódicas y para el desarrollo de innumerables aplicaciones. La tesis lleva por título “Análisis del crecimiento y las propiedades ópticas en alta energía de cristales fotónicos coloidales”.

Premio CicCartuja - Ebro Foods a jóvenes investigadores para **Nuria Hidalgo Serrano,** por su trabajo en resonadores porosos mesoestructurados en estructuras de cristal fotónico y el estudio de su aplicación para el reconocimiento óptico de moléculas en función de su tamaño. Hidalgo está vinculada al Grupo que dirige Hernán R. Míguez, Investigador Científico del CSIC, cuya línea de trabajo se centra en el desarrollo de nanomateriales ópticos. Este trabajo avanza en el descubrimiento de cristales que permiten detectar ópticamente líquidos y gases. Este estudio abre una puerta a la consecución de nuevos materiales con cualidades sensoras que podrían ser aplicables a nivel industrial o, incluso, en la vida cotidiana, como sensores de polución en las vías urbanas, antiincendios en los edificios o detectores de gases en fábricas.

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 spectroscopico 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

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 renewable energy sectors.

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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 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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Papel de los aditivos en los sistemas composites de hidruros metálicos reactivos para almacenamiento de hidrógeno

Role of additives in the reactive hydride composite systems for hydrogen storage

Código/Code:	CTQ2009-13440
Periodo/Period:	01/01/2010 AL 31/12/2012
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	174.240 €
Investigador responsable/Research head:	Fernández Camacho, A.
Componentes/Research group:	López Cartés, C., Rojas Ruíz, T.C., Arzac, G.; Hufschmidt, D., Cecchini, R., Deprez, E

RESUMEN / ABSTRACT

Dada la problemática actual por el agotamiento a corto-medio plazo de los combustibles fósiles y los cambios climáticos causados por el efecto invernadero, se hace necesaria la reconsideración de una política energética global. El hidrógeno como vector de transporte y almacenamiento de energía es un candidato muy atractivo por tratarse de una alternativa viable y limpia.

En el presente proyecto se propone el estudio de los llamados sistemas composites de hidruros reactivos (RHC) para almacenamiento de hidrógeno. Estos sistemas se basan en acoplar un hidruro metálico sencillo (i.e. MgH_2) con un hidruro complejo (típicamente un compuesto borohidruro, i.e $LiBH_4$) para dar una reacción reversible que produce o consume hidrógeno. El sistema puede así usarse como material para almacenamiento de hidrógeno de acuerdo a la siguiente reacción: $MgH_2+2LiBH_4 \leftrightarrow MgB_2+LiH+4H_2$ (11.4 wt% capacidad de almacenamiento de hidrógeno). La reacción mejora el balance de calor, en comparación con el MgH2 puro, al reducir la liberación de calor durante el proceso de carga. Para mejorar los aspectos cinéticos (reducción de las temperaturas y tiempos de operación) se ha propuesto el uso de catalizadores y/o aditivos. El principal objetivo del proyecto es comprender el papel de estos aditivos para mejorar las cinéticas de sorción de hidrógeno. En particular se han seleccionado como aditivos para este estudio los productos comerciales Ti-Isopropoxide ($TiO_4C_{12}H_{28}$), TiO_2 y VCl_3 . También se prepararan en nuestro laboratorio otros catalizadores como Co_3B, Ni_3B o RuCo que igualmente se ensayarán.

Los sistemas se prepararán y activarán por molienda de alta energía de los dos materiales hidruros molidos juntos con ó sin aditivos (5-10 mol%). Los estudios cinéticos se llevarán a cabo a través de medidas de sorción gravimétrica y volumétrica de hidrógeno (desorción o absorción vs. tiempo a T constante) y de la calorimetría de barrido diferencial (DSC). Se llevará también a cabo un estudio exhaustivo de caracterización microestructural y química de los sistemas en las diferentes etapas (tras la molienda, desorbidos y re-absorbidos) con las siguientes técnicas: Difracción de rayos X (XRD), microscopía electrónica de transmisión (TEM) acoplada al análisis EDX (energía dispersiva de rayos X) y EELS (espectroscopía de pérdida de energía de electrones), espectroscopía de fotoemisión (XPS) y espectroscopía de absorción de rayos X (XAS). El estudio comparativo de las muestras con y sin aditivos y la correlación entre los estudios cinéticos y el análisis microestructural y químico, deben clarificar el mecanismo de la mejora cinética producida por los aditivos. Estos mecanismos están a día de hoy lejos de ser comprendidos. Sobre la base del conocimiento adquirido se espera mejorar de manera significativa estos sistemas en relación a sus aplicaciones para almacenamiento de hidrógeno.

Due to the expected short-medium term exhaustion of fossil fuels and due to clime changes produced by the green house effect, it is necessary to reconsider a new global energy policy. Hydrogen, as a vector for energy storage and transport, is an attractive candidate for a clean handling of energy.

In the present project it is proposed the study of the so called reactive hydride composite systems (RHC) for hydrogen storage. These systems are based in the coupling of a single metal hydride (i.e. MgH_2) with a complex hydride (typically a borohydride compound, i.e $LiBH_4$) to give a reversible reaction that is producing or consuming hydrogen. The system can so be used as a hydrogen storage material according to following reaction: $MgH_2+2LiBH_4 \leftrightarrow MgB_2+LiH+4H_2$ (11.4 wt% hydrogen storage capacity). The reaction is improving the heat transfer handling, as compared to pure MgH_2, by reducing heat release during the charging process.

To improve the kinetic aspects (reduction of operation temperatures and times) it has been proposed the use of catalysts a/o additives. The main objective of the project is to understand the role of these additives to improve the hydrogen sorption kinetics. In particular commercial Ti-Isopropoxide ($TiO_4C_{12}H_{28}$) , TiO_2 and VCl_3 have been selected as additives for this study. Also other catalysts like Co_3B, Ni_3B or RuCo will be prepared in our laboratory and also tested.

The systems will be prepared and activated by high energy ball milling of the two hydride materials milled together with or without the additives (5-10 mol%). Kinetic studies will be carried out by gravimetric and volumetric hydrogen sorption measurements (hydrogen desorption or adsorption vs. time at constant T) and differential scanning calorymetry (DSC) analysis. An exhaustive microstructural and chemical analysis of the systems at the different step (as prepared, desorbed and re-absorbed) will be undertaken by following techniques: X-Ray Diffraction (XRD), Transmission Electron Microscopy (TEM) coupled to EDX (energy dispersive X-Ray) and EELS (Electron Energy Loss Spectroscopy) analysis, X-Ray Photoelectrton Spectroscopy (XPS) and X-Ray absorption Spectroscopy (XAS). The comparative study of the samples, with and without additives, and the correlation between the kinetic studies and the microstructural and chemical analysis, should clarify the mechanisms of the kinetic improvements produced by the additives. These mechanisms are today far from being understood. On basis of the acquired knowledge we expect to significantly improve the systems with respect to hydrogen storage applications.

Acoplamiento dinámico de fronteras de grano - segregación de impurezas en policristales nanoestructurados: aplicación a la circonia tetragonal dopada con itrio policristalina (YTZP)

The coupling of grain boundary dynamics and impurity segregation in nanostructured polycrystals: application to yttria tetragonal zirconia polycrystal (YTZP).

Código/Code:	MAT2009-14351-C02-01
Periodo/Period:	01/01/2010 – 31/12/2012
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	87.120 €
Investigador responsable/Research head:	Gómez García, D.
Componentes/Research group:	Francisco Luis Cumbrera Hernández, Arturo Domínguez Rodríguez, Robert Luis González Romero

RESUMEN / ABSTRACT

El proyecto que se presenta estudiará, mediante simulación por ordenador a distintas escalas, la evolución microestructural de un policristal a temperatura constante y bajo la aplicación de un campo de tensiones mecánicas; en particular, se prestará especial atención a los sistemas nanométricos. Para abordar con rigor este problema es indispensable conocer la ley de movilidad de las fronteras de grano en función de la temperatura y de las tensiones locales. En presencia de impurezas, esta ley depende crucialmente de la concentración de especies atómicas segregadas en dichas fronteras y su evolución durante el régimen dinámico (i. e. durante la deformación). A su vez, la segregación se ve alterada por el propio movimiento de la frontera de grano, de modo que ambos fenómenos están acoplados entre sí. El estudio de la segregación se realizará mediante simulación por Dinámica Molecular (DM); asimismo, se utilizará la DM para caracterizar la movilidad de una única frontera de grano conteniendo impurezas. Estos datos serán empleados en un modelo mesoscópico que se usará para estudiar la dinámica de un conjunto de granos de tamaño nanométrico y, por ende, la plasticidad de este sistema policristalino modelo.

El objetivo último de este proyecto es el de determinar la ley de evolución de los centros de masas de los granos para obtener, previo tratamiento estadístico, la ley constitutiva para la plasticidad en un policristal nanométrico. Esta ley macroscópica se contrastará finalmente con resultados experimentales en el sistema itria-zirconia policristalino (YTZP) nanométrico, en el que el equipo ha adquirido amplia experiencia en los últimos años.

This project aims to study, by computer simulation at different length scales, the microstructural evolution of a polycrystal at elevated temperature and under an applied mechanical stress field, with an emphasis on nanometric systems. For this study it is essential to know the law of mobility of the grain boundaries as a function of the temperature and the local stresses. When impurities are present, this law depends critically upon the concentration of segregated chemical species at these boundaries and upon their evolution during the dynamic regime (i.e., during deformation). As segregation itself is altered by the movement of the grain boundary, the two phenomena are coupled. The study of segregation will be carried out by Molecular Dynamics (MD) simulations; MD will also be used to characterize the mobility of a single grain boundary containing impurities. These data will be used as input in a mesoscopic model, which will allow the study of the dynamics of an ensemble of nanometric grains and, consequently, plasticity in this model polycrystalline system.

The final objective of this project is to determine the law of evolution of the centers of mass of the grains in order to get, via a statistical treatment, the constitutive law for plasticity in a nanometric polycrystal. This macroscopic law will then be compared with experimental results in nanometric YTZPa system in which the research team has wide experience in recent years.

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.

OTROS PROYECTOS / OTHER PROJECTS

Nanomateriales y catalizadores para la generación y uso del hidrógeno en aplicaciones portátiles

Periodo/Period:	01-01-2012 / 31-12-2013
Código/Code:	201260E006
Oganismo Financiador/Financial source:	CSIC (Intramural)
Importe total/Total amount:	100.315 €
Investigador responsable/Research head:	Asunción Fernández Camacho

TRANSFERENCIA / TECHNOLOGICAL TRANSFER

CONVENIOS Y CONTRATOS / CONTRACTS AND AGREEMENTS

Caracterización microestructural y química de materiales para avisadores sonoros

Periodo/Period:	19-01-2010 / 19-01-2012
Organismo Financiador/Financial source:	Clarton Horn
Investigador responsable/Research head:	Asunción Fernández Camacho
Componentes/Research group:	Vanda C. Fortio Godinho, Inmaculada Rosa Cejudo

Nuevas tecnologías para la obtención, funcionalización y manipulación de carbonato de calcio derivados del mármol blanco, de tamaño de partícula submicrónica y sus derivados - NONOCARBOFUN

Periodo/Period:	01-04-2011 / 31-07-2012
Organismo Financiador/Financial source:	REVERTE MINERALS
Investigador responsable/Research head:	T. Cristina Rojas Ruiz
Componentes/Research group:	Asunción Fernández Camacho, M. Carmen Jiménez de Haro, T. Cristina Rojas Ruiz

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

PUBLICACIONES / PUBLICATIONS

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

Water-dependent micromechanical and rheological properties of silica colloidal crystals studied by nanoindentation
Gallego-Gomez, Francisco; Morales-Florez, Victor; Blanco, Alvaro; de la Rosa-Fox, Nicolas; Lopez, Cefe
Nano Letters, **12** (2012) 4920-4924
August, 2012 | **DOI:** 10.1021/nl3024998

Here we show the suitability of nanoindentation to study in detail the micromechanical response of silica colloidal crystals (CCs). The sensitivity to displacements smaller than the submicrometer spheres size, even resolving discrete events and superficial features, revealed particulate features with analogies to atomic crystals. Significant robustness, long-range structural deformation, and large energy dissipation were found. Easily implemented temperature/rate-dependent nanoindentation quantified the paramount role of adsorbed water endowing silica CCs with properties of wet granular materials like viscoplasticity. A novel "nongranular" CC was fabricated by substituting capillary bridges with silica necks to directly test water-independent mechanical response. Silica CCs, as specific (nanometric, ordered) wet granular assemblies with well-defined configuration, may be useful model systems for granular science and capillary cohesion at the nanoscale.

Nanoecotoxicity effects of engineered silver and gold nanoparticles in aquatic organisms
Lapresta-Fernandez, A; Fernandez, A; Blasco, J
TrAC Trends in Analytical Chemistry, **32** (2012) 40-59
February, 2012 | **DOI:** 10.1016/j.trac.2011.09.007

Engineered nanoparticles (ENPs) are increasingly being incorporated into commercial products. A better understanding is required of their environmental impacts in aquatic ecosystems.
This review deals with the ecotoxicity effects of silver and gold ENPs (AgNPs and AuNPs) in aquatic organisms, and considers the means by which these ENPs enter aquatic environments, their aggregation status and their toxicity. Since ENPs are transported horizontally and vertically in the water column, we discuss certain factors (e.g., salinity and the presence of natural organic materials), as they cause variations in the degree of aggregation, size range and ENP toxicity. We pay special attention to oxidative stress induced in organisms by ENPs.
We describe some of the main analytical methods used to determine reactive oxygen species, antioxidant enzyme activity, DNA damage, protein modifications, lipid peroxidation and relevant metabolic activities. We offer an overview of the mechanisms of action of AgNPs and AuNPs and the ways that relevant environmental factors can affect their speciation, agglomeration or aggregation, and ultimately their bio-availability to aquatic organisms.
Finally, we discuss similarities and differences in the adverse effects of ENPs in freshwater and salt-water systems.

How Important is Working with an Ordered Electrode to Improve the Charge Collection Efficiency in Nanostructured Solar Cells?

Gonzalez-Vazquez, JP; Morales-Florez, V; Anta, JA
Journal of Physical Chemistry Letters, **3** (2012) 386-393
February, 2012 | **DOI:** 10.1021/jz2015988

The collection efficiency of carriers in solar cells based on nanostructured electrodes is determined for different degrees or morphological one-dimensional order. The transport process is modeled by random walk numerical simulation in a mesoporous electrode that resembles the morphology of nanostructured TiO2 electrodes typically used in dye-sensitized solar cells and related systems. By applying an energy relaxation procedure in the presence of an external potential, a preferential direction is induced in the system. It is found that the partially ordered electrode can almost double the collection efficiency with respect to the disordered electrode. However, this improvement depends strongly on the probability of recombination. For too rapid or too slow recombination, working with partially ordered electrodes will not be beneficial. The computational method utilized here makes it possible to relate the charge collection efficiency with morphology. The collection efficiency is found to reach very rapidly a saturation value, meaning that, in the region of interest, a slight degree of ordering might be sufficient to induce a large improvement in collection efficiency.

New insights into the synergistic effect in bimetallic-boron catalysts for hydrogen generation: The Co–Ru–B system as a case study

Arzac, G. M.; Rojas, T. C.; Fernandez, A.
Applied Catalysis B-Environmental, **128** (2012) 39-47
November, 2012 | **DOI:** 10.1016/j.apcatb.2012.02.013

Catalysed sodium borohydride hydrolysis is a high-potential method to produce hydrogen for portable applications. Co–B catalysts are the most chosen because they are easily prepared, cheap and efficient. The addition of small amounts of Ru produces a significant enhancement in catalytic activity.

In the present work a series of Co–Ru–B catalysts with variable Ru content was prepared, isolated and characterized. The comprehension of the synergistic effect was achieved trough the incorporation of the nanostructural dimension to the study of surface and bulk chemical states of the involved atoms along the series. It was found that up to 70% (of total metal) atomic content of Ru the catalysts can be considered isostructural to the single Co–B catalyst in the nanoscale. A structural transition occurs in the case of the pure Ru–B material to produce a boron deficient material with higher nanoparticle size. This structural transition together with Co segregation and Ru dispersion play a key role when explaining a [OH−] dependent effect.

The inexistence of borate layers in Ru rich catalysts is suggestive in the research for non deactivating catalysts.

Public concern over ecotoxicology risks from nanomaterials: Pressing need for research-based information

Lapresta-Fernandez, A; Fernandez, A; Blasco, J
Environment International, **39** (2012) 148-149
February, 2012 | **DOI:** 10.1016/j.envint.2011.10.012

[No abstract]

Electrons in the Band Gap: Spectroscopic Characterization of Anatase TiO2 Nanocrystal Electrodes under Fermi Level Control

Berger, T; Anta, JA; Morales-Florez, V
Journal of Physical Chemistry C, **116** (2012) 11444-11455
May, 2012 | **DOI:** 10.1021/p212436b

Macroscopic properties of semiconductor nanoparticle networks in functional devices strongly depend on the electronic structure of the material. Analytical methods allowing for the characterization of the electronic structure in situ, i.e., in the presence of an application-relevant medium, are therefore highly desirable. Here, we present the first spectral data obtained under Fermi level control of electrons accumulated in anatase TiO 2 electrodes in the energy range from the MIR to the UV (0.1-3.3 eV). Band gap states were electrochemically populated in mesoporous TiO 2 films in contact with an aqueous electrolyte. The combination of electrochemical and spectroscopic measurements allows us for the first time to determine both the energetic location of the electronic ground states as well as the energies of the associated optical transitions in the energetic range between the fundamental absorption threshold and the onset of lattice absorption. On the basis of our observations, we attribute spectral contributions in the vis/NIR to d-d transitions of Ti 3+ species and a broad MIR absorption, monotonically increasing toward lower wavenumbers, to a quasi-delocalization of electrons. Importantly, signal intensities in the vis/NIR and MIR are linearly correlated. Absorbance and extractable charge show the same exponential dependence on electrode potential. Our results demonstrate that signals in the vis/NIR and MIR are associated with an exponential distribution of band gap states.

Electrostatic Induced Molecular Tilting in Self-Assembled Monolayers of n-Octadecylamine on Mica

Oviedo, J; San-Miguel, MA; Heredia-Guerrero, JA; Benitez, JJ
Journal of Physical Chemistry C, **116** (2012) 7099-7105
March, 2012 | **DOI:** 10.1021/jp300829g

Self-assembled monolayers of n-octadecylamine on mica (ODA/mica SAMs) have been investigated by atomic force microscopy (AFM) and by attenuated total reflectance infrared (ATR-FTIR) and X-ray photoelectron (XPS) spectroscopies. Topographic data characterizes a stable configuration with the alkyl skeleton tilted approximate to 46 degrees from the surface normal that is rationalized according to a well established structural alkyl chain packing model. Extended contact with air increases molecular tilting up to approximate to 58 degrees. ATR-FTIR and XPS reveal the presence of protonated amino groups within the monolayer and its increment upon exposure to air. The transition between both tilted states is explained assuming the protonation reaction as the driving force and introducing a model to evaluate an electrostatic repulsions term in the overall cohesive energy balance of the system. ODA molecules in the self-assembled monolayer respond to their spontaneous protonation by atmospheric water by tilting as a mechanism to relax the repulsions between -NH3+ heads.

Deactivation, reactivation and memory effect on Co–B catalyst for sodium borohydride hydrolysis operating in high conversion conditions
Arzac, GM; Hufschmidt, D; De Haro, MCJ; Fernandez, A; Sarmiento, B; Jimenez, MA; Jimenez, MM
International Journal of Hydrogen Energy, **37** (2012) 14373-14381
October, 2012 | **DOI:** 10.1016/j.ijhydene.2012.06.117

A system with a continuous reactor to produce hydrogen by sodium borohydride hydrolysis was designed and built. The purpose was to test a supported Co–B catalyst durability upon cycling and long life experiments in high conversion conditions. A Stainless Steel monolith was built and calcined to improve adherence. For comparison a Ru–B catalyst was tested upon cycling. Both Co–B and Ru–B catalysts are durable during 6 cycles and then deactivate. A known reactivation procedure has proven to be more effective for the Co–B than for the Ru–B catalyst. This is related to stronger adsorption of B–O based compounds on the Co–B catalyst which is reversible upon acid washing. For the Ru–B catalyst deactivation may be more related to particle agglomeration than to the adsorption of B–O based species. The continuous system enlarges the catalysts durability because of the continuous borate elimination at elevated temperatures.

Three-dimensional fabrication and characterisation of core-shell nano-columns using electron beam patterning of Ge-doped SiO2
Gontard, LC; Jinschek, JR; Ou, HY; Verbeeck, J; Dunin-Borkowski, RE
Applied Physics Letters, **100** (2012) 263113
June, 2012 | **DOI:** 10.1063/1.4731765

A focused electron beam in a scanning transmission electron microscope (STEM) is used to create arrays of core-shell structures in a specimen of amorphous SiO2 doped with Ge. The same electron microscope is then used to measure the changes that occurred in the specimen in three dimensions using electron tomography. The results show that transformations in insulators that have been subjected to intense irradiation using charged particles can be studied directly in three dimensions. The fabricated structures include core-shell nano-columns, sputtered regions, voids, and clusters.

Magnetron sputtered a-SiO xN y thin films: A closed porous nanostructure with controlled optical and mechanical properties
Godinho, V; Rojas, TC; Fernandez, A
Microporous and Mesoporous Materials, **149** (2012) 142-146
February, 2012 | **DOI:** 10.1016/j.micromeso.2011.08.018

Amorphous silicon oxynitride coatings with similar composition and different closed porosity were prepared by magnetron sputtering. Pores size, shape and distribution were evaluated by scanning electron microscopy and transmission electron microscopy. Raman and EELS analysis proved that the pores are filled with molecular nitrogen trapped during deposition. The mechanical properties evaluated by nanoindentation shows that the presence of closed nano-porosity does not compromise the mechanical integrity of these coatings. The introduction of closed porosity is shown as a good strategy for obtaining lower dielectric constant silicon oxynitride coatings with similar composition while keeping the good mechanical properties (~13 GPa) characteristic of this type of coatings. The presence of close porosity gives also a good stability of coatings properties as compared to open porosity microstructures where gas phase in contact with the coatings can affect coatings properties.

Microstructural and chemical characterization of nanostructured Tialsin coatings with nanoscale resolution

Godinho, V; Rojas, TC; Trasobares, S; Ferrer, FJ; Delplancke-Ogletree, MP; Fernandez, A
Microscopy and Microanalysis, **18** (2012) 568-581
June, 2012 | **DOI:** 10.1017/S1431927612000384

Nanoscale resolution electron microscopy analysis combined with ion beam assisted techniques are presented here, to give answers to full characterization of morphology, growth mode, phase formation, and compositional distribution in nanocomposite TiAlSiN coatings deposited under different energetic conditions. Samples were prepared by magnetron sputtering, and the effects of substrate temperature and bias were investigated. The nanocomposite microstructure was demonstrated by the formation of a face-centered cubic (Ti,Al)N phase, obtained by substitution of Al in the cubic titanium nitride (c-TiN) phase, and an amorphous matrix at the column boundary regions mainly composed of Si, N (and O for the samples with higher oxygen contents). Oxygen impurities, predicted as the principal responsible for the degradation of properties, were identified, particularly in nonbiased samples and confirmed to occupy preferentially nitrogen positions at the column boundaries, being mainly associated to silicon forming oxynitride phases. It has been found that the columnar growth mode is not the most adequate to improve mechanical properties. Only the combination of moderate bias and additional substrate heating was able to reduce the oxygen content and eliminate the columnar microstructure leading to the nanocomposite structure with higher hardness (>30 GPa).

Chemical–physical characterization of isolated plant cuticles subjected to low-dose γ-irradiation

Heredia-Guerrero, Jose A; de Lara, Rocio; Dominguez, Eva; Heredia, Antonio; Benavente, Juana; Benitez, Jose J
Chemistry and physics of lipids, *165* (2012) 803-808
December, 2012 | **DOI:** 10.1016/j.chemphyslip.2012.10.003

Isolated tomato fruit cuticles were subjected to low dose (80 Gy) γ-irradiation, as a potential methodology to prevent harvested fruit and vegetables spoilage. Both irradiated and non-irradiated samples have been morphologically and chemically characterized by scanning electron (SEM), atomic force (AFM), attenuated total reflectance Fourier transform infrared (ATR-FTIR) and X-ray photoelectron (XPS) spectroscopies. Additionally, electrochemical measurements comprising membrane potential and diffusive permeability were carried out to detect modifications in transport properties of the cuticle as the fruit primary protective membrane. It has been found that low dose γ-irradiation causes some textural changes on the surface but no significant chemical modification. Texture modification is found to be due to a partial removal of outermost (epicuticular) waxes which is accompanied by mild changes of electrochemical parameters such as the membrane fixed charge, cation transport number and salt permeability. The modification of such parameters indicates a slight reduction of the barrier properties of the cuticle upon low dose γ-irradiation.

Phase composition and tribomechanical properties of Ti-B-C nanocomposite coatings prepared by magnetron sputtering

Sanchez-Lopez, JC; Abad, MD; Justo, A; Gago, R; Endrino, JL; Garcia-Luis, A; Brizuela, M
Journal of Physics D: Applied Physics, **45** (2012) 375401
September, 2012 | **DOI:** 10.1088/0022-3727/45/37/375401

Protective nanocomposite coatings based on hard ceramic phases (TiC, TiB2) combined with amorphous carbon (a-C) are of interest because of their adequate balance between mechanical and tribological performances. In this work, Ti–B–C nanocomposite coatings were prepared by co-sputtering of graphite and TiB2 targets. Varying the discharge power ratio applied to the graphite and TiB2 targets from 0 to 2, the a-C content in the coatings could be tuned from 0 to 60%, as observed by means of Raman and x-ray photoelectron spectroscopy (XPS). The microstructural characterization demonstrated a progressive decrease in crystallinity from an initial nanocrystalline (nc) TiB2-like structure to a distorted TiBxCy ternary compound with increasing C concentration. X-ray absorption near-edge structure measurements on the B K-edge helped to determine a hexagonal arrangement around the B atoms in the ternary TiBxCy phase. A fitting analysis of the C 1s XPS peak allowed us to evaluate the relative amount of a-C and TiBxCy components. A drastic change in hardness (from 52 to 13 GPa) and friction coefficient values (from 0.8 to 0.2) is noticed when moving from nc-TiB2 to TiBC/a-C nanocomposites. The fraction of a-C necessary to decrease the friction below 0.2 was found to be 45%. Raman observation of the wear tracks determined the presence of disordered sp2-bonded carbon phase associated with the diminution of the friction level.

Microstructural characterization of hydrophobic Ti1−xAlxN coatings with moth-eye-like surface morphology

Godinho, V; Lopez-Santos, C; Rojas, TC; Philippon, D; de Haro, MCJ; Lucas, S; Fernandeza, A
Journal of Alloys and Compounds, **536** (2012) S398-S406
September, 2012 | **DOI:** 10.1016/j.jallcom.2012.02.178

Ti1−xAlxN thin films with different Al content were deposited by magnetron sputtering. The combination of electron energy loss spectroscopy (EELS) and energy dispersive spectroscopy (EDS) was used to evaluate the composition of the coatings. The effect of Al content on the morphology and properties of the coatings was investigated. High resolution electron microscopy and related techniques revealed the formation of a pillared moth-eye-like nanostructure with variable size and distribution of meso- and nano-columns and different degree of open porosity that depends on the Al content on the coating. For low Al content ($x \leq 0.21$) c-(Ti,Al)N highly porous columns ending in a sharp pyramidal shape present low reflectivity and high hydrophobicity. While the precipitation of h-AlN phase at the column boundaries for $x = 0.71$ suppresses the c-(Ti,Al)N columnar growth and produces a smother surface, with higher reflectivity and less hydrophobic character.

Rapid carbothermic synthesis of silicon carbide nano powders by using microwave heating
Moshtaghioun, BM; Poyato, R; Cumbrera, FL; de Bernardi-Martin, S; Monshi, A; Abbasi, MH; Karimzadeh, F; Dominguez-Rodriguez, A
Journal of the European Ceramic Society, **32** (2012) 1787-1794
July, 2012 | **DOI:** 10.1016/j.jeurceramsoc.2011.12.021

This paper reports an improved procedure for synthesis of silicon carbide nanopowders from silica by carbothermic reduction under fast microwave-induced heating. The powders have been prepared by direct solid-state reaction in a 2.45 GHz microwave field in nitrogen atmosphere after 40 h milling. For the first time, the formation of silicon carbide (beta-SiC) as a major phase can be achieved at 1200 degrees C in 5 min of microwave exposure, resulting in nano sized particles ranging from 10 to 40 nm under optimized synthesis condition. The Rietveld quantitative phase-composition analysis confirmed that the major SiC polytype is cubic SiC (beta-SiC) with 98.5(4) weight fraction and the remained is minor hexagonal SiC polytypic (alpha-SiC) phases. Therefore this method is the most efficient one for SiC powder synthesis in terms of energy and time saving as well as preparation of SiC nano powders.

Nanostructured Spark Plasma Sintered Ce-TZP Ceramics
Cruz, SA; Poyato, R; Cumbrera, FL; Odriozola, JA
Journal of the American Ceramic Society, **95** (2012) 901-906
March, 2012 | **DOI:** 10.1111/j.1551-2916.2011.04978.x

In this work, spark plasma sintering (SPS) of 10 mol% CeO_2-doped ZrO_2 nanocrystalline powders, obtained by a two-step synthesis procedure, allows the preparation of fully densified nanostructured ceramics. The CeO_2-ZrO_2 powders with particle size below 100 nm are obtained after CeO_2 deposition on hydrothermally synthesized ZrO_2 particles by the impregnation method. Tetragonal CeO_2-ZrO_2 ceramics are obtained when sintering at 1200°C without holding time. A graded material containing tetragonal, monoclinic, and pyrochlore phases are obtained when sintering at 1200°C and for 5 min holding time. This is explained in terms of the gradual reduction of Ce^{4+} to Ce^{3+} species by carbon in the graphite environment during SPS. With the successful combination of the stabilizer coating technique and SPS, we achieve not only the stabilization of the tetragonal phase in the ceramics, but also good control of the grain size, by producing nanostructured ceramics with 40-70 nm grain sizes.

Nanoscale mechanically induced structural and electrical changes in Ge 2Sb 2Te 5 films
Cecchini, R; Benitez, JJ; Sanchez-Lopez, JC; Fernandez, A
Journal of Applied Physics, **111** (2012) 016101
January, 2012 | **DOI:** 10.1063/1.3673592

We demonstrate that the microstructure and electrical properties of Ge2Sb2Te5 films can be changed by a nanoscale mechanical process. Nanoscratching is used to define modified areas onto an as-deposited crystalline Ge2Sb2Te5 film. Scanning tunneling microscopy measurements show that the modified areas have a very low electrical conductivity. Micro-Raman measurements indicate that the mechanically induced microstructural changes are consistent with a phase transformation from crystalline to amorphous, which can be reversed by laser irradiation.

Changes on the nanostructure of cementitius calcium silicate hydrates (C-S-H) induced by aqueous carbonation
Morales-Florez, V; Findling, N; Brunet, F
Journal of Materials Science, **47** (2012) 764-771
January, 2012 | **DOI:** 10.1007/s10853-011-5852-6

The nanostructure of the main binding phase of the hydrated cements, the calcium silicate hydrates (C–S–H), and their structural changes due to aqueous carbonation have been characterized using TEM, nitrogen physisorption, and SAXS. Synthetic C–S–H has been used for this purpose. Two different morphologies were identified, similar to the high density and low density C–S–H types. When submitting the sample to a CO2 flux, the low density phase was completely carbonated. The carbonation by-products, calcium carbonate, and silica gel were also identified and characterized. The precipitation of the silica gel increased the specific surface area from 95 to 132 m2/g, and its structure, formed by particles of ~5 nm typical radius, was observed by small angle X-ray scattering. In addition, the resistance of the high density C–S–H to carbonation is reported, and the passivating effect of the precipitated calcium carbonate is also discussed. Finally, the results have been compared with carbonation features observed in Portland cement carbonated experimentally at downhole conditions.

Chemical and microstructural characterization of (Y or Zr)-doped CrAlN coatings
Rojas, T. C.; El Mrabet, S.; Dominguez-Meister, S.; Brizuela, M.; Garcia-Luis, A.; Sanchez-Lopez, J. C.
Surface and Coatings Technology, **211** (2012) 104-110
October, 2012 | **DOI:** 10.1016/j.surfcoat.2011.07.071

Magnetron sputtered chromium aluminium nitride films are excellent candidates for advanced machining and protection for high temperature applications. In this work CrAlN-based coatings including Y or Zr as dopants (≈ 2 at.%) are deposited by d.c. reactive magnetron sputtering on silicon substrates using metallic targets and Ar/N2 mixtures. The hardness properties are found in the range of 22–33 GPa with H/E ratios close to 0.1. The influence of the dopant element in terms of oxidation resistance after heating in air at 1000 °C is studied by means of X-ray diffraction (XRD), cross-sectional scanning electron microscopy (X-SEM) and energy dispersive X-ray analysis (EDX). The microstructure and chemical bonding are investigated using a transmission electron microscope (TEM) and electron energy-loss spectroscopy (EELS) respectively. The improvement in oxidation resistance as compared to pure CrN coating is manifested in the formation of a Al-rich outer layer that protects the underneath coating from oxygen diffusion. The best performance obtained with the CrAlYN film is investigated by in situ annealing of this sample inside the TEM in order to gain knowledge about the structural and chemical transformations induced during heating.

Influence of silver content on the tribomechanical behavior on Ag-TiCN bioactive coatings
Sanchez-Lopez, JC; Abad, MD; Carvalho, I; Galindo, RE; Benito, N; Ribeiro, S; Henriques, M; Cavaleiro, A; Carvalho, S
Surface and Coatings Technology, **206** (2012) 2192-2198
January, 2012 | **DOI:** 10.1016/j.surfcoat.2011.09.059

Surface modification of bulk materials used in biomedical applications has become an important prerequisite for better biocompatibility. In particular, to overcome the particle generation, low-wear coatings based on carbon (nitrogen) and containing antimicrobial elements such as silver are promising candidates. Thus, the present work explores the potentialities of silver-containing carbonitride-based (Ag-TiCN) thin films prepared by direct current unbalanced reactive magnetron sputtering. The silver content in the coatings was varied from 0 to 26.7at.% by changing the targets and the fraction of C 2H 2 and N 2 in the gas mixture with Ar. The obtained Ag-TiCN based coatings were characterized in terms of composition and microstructure. Mechanical and tribological properties of the films were studied by nanoindentation and reciprocating pin-on disk testing in a fetal bovine serum solution, respectively. Raman, scanning electron microscope and energy dispersive X-ray analysis was carried out in the contact region after tribological tests to obtain information about the friction mechanism. The cytotoxicity of the coatings was assessed by in vitro tests using fibroblast cells. The coatings comprised a mixture of TiC xN 1-x, Ag and a-C(N) x phases whose relative proportion varied depending on the Ag/Ti ratio. The mechanical, tribological and cytotoxicity properties were correlated with the chemical and phase composition. When the Ag/Ti ratios were below 0.20 (Ag contents <6.3at.%) the films resulted harder (~18GPa) with higher wear resistance (~10 -6mm 3/Nm), showing similar friction coefficient (~0.3) and good biocompatibility.

An international round-robin calibration protocol for nanoindentation measurements
Cabibbo, M; Ricci, P; Cecchini, R; Rymuza, Z; Sullivan, J; Dub, S; Cohen, S
Micron, **43** (2012) 215-222
February, 2012 | **DOI:** 10.1016/j.micron.2011.07.016

Nanoindentation has become a common technique for measuring the hardness and elastic–plastic properties of materials, including coatings and thin films. In recent years, different nanoindenter instruments have been commercialised and used for this purpose. Each instrument is equipped with its own analysis software for the derivation of the hardness and reduced Young's modulus from the raw data. These data are mostly analysed through the Oliver and Pharr method. In all cases, the calibration of compliance and area function is mandatory. The present work illustrates and describes a calibration procedure and an approach to raw data analysis carried out for six different nanoindentation instruments through several round-robin experiments. Three different indenters were used, Berkovich, cube corner, spherical, and three standardised reference samples were chosen, hard fused quartz, soft polycarbonate, and sapphire. It was clearly shown that the use of these common procedures consistently limited the hardness and reduced the Young's modulus data spread compared to the same measurements performed using instrument-specific procedures. The following recommendations for nanoindentation calibration must be followed: (a) use only sharp indenters, (b) set an upper cut-off value for the penetration depth below which measurements must be considered unreliable, (c) perform nanoindentation measurements with limited thermal drift, (d) ensure that the load–displacement curves are as smooth as possible, (e) perform stiffness measurements specific to each instrument/indenter couple, (f) use Fq and Sa as calibration reference samples for stiffness and area function determination, (g) use a function, rather than a single value, for the stiffness and (h) adopt a unique protocol and software for raw data analysis in order to limit the data spread related to the instruments (i.e. the level of drift or noise, defects of a given probe) and to make the H and Er data intercomparable.

Processing of swnt-reinforced yttria stabilized zirconia by spark plasma sintering and microstructure characterization

Martín, SD; Poyato, R.; Garcia, D.G.; Rodríguez, A.D.
Journal of Nano Research, **18-19** (2012) 317-323
July, 2012 | **DOI:** 10.4028/www.scientific.net/JNanoR.18-19.317

Single wall carbon nanotube reinforced yttria stabilized zirconia ceramic materials have been obtained by means of spark plasma sintering technique. Single wall carbon nanotubes were treated in an acid solution before mixing with zirconia powders to obtain a uniform distribution of both powders. This method allows obtaining ceramic materials with a grain size between 200 nanometers and 1 micron and with a grain size distribution which depends on processing conditions. This new route opens a new perspective for new ceramic composites tailoring with enhanced mechanical properties as structural materials.

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

COMUNICACIONES EN CONGRESOS Y REUNIONES / COMMUNICATIONS IN CONGRESS AND MEETINGS

NanoSpain Conference

27 febrero – 1 marzo [Santander, España]

Assesing toxicity of citrate-gold nanoparticles at different marine trophic levels (microalgae, copepods and bivalve mollusks)
Julián Blasco; Ignacio Moreno-Garrido; Miriam Hampel; Jorge Otero; Gonzalo Quiroga; Moritz Volland; Carlos A. García-Negrete; A. Lapresta-Fernández; Asunción Fernández
Comunicación oral

Exposure of the bivalve RUDITAPES PHILIPPINARUM to gold nanoparticles: Location study by electron microscopy
C. A. García-Negrete; T.C. Rojas Ruíz; M.C. Jimenez de Haro; A. Lapresta-Fernández; A. Fernández; M. Volland; M. Hampel; J. Blasco.
Poster

The 39th International Conference on Metallurgical Coatings and Thin Films ICMCTF 2012

23 – 27 abril [San Diego, Estados Unidos de América]

High temperature protection of ferritic steels by nano-structured coatings: supercritical steam turbines applications
S. Mato; P. Hierro; I. Castañeda; A. Alcalá; I. Lasanta; M. Tejero; J. Sánchez; M. Brizuela; J. Pérez
Comunicación oral

Tribological behaviour at high temperature of hard CrAlN coatings doped with Y or Zr.
J.C. Sánchez-López; A. Contreras; A. García-Luis; M. Brizuela
Comunicación oral

E-MRS 2012 Spring Meeting

27 - 31 Mayo [Estrasburgo, Francia]

Magnetron sputtered amorphous silicon films with closed porosity: microstructure and optical properties
V. Godinho, D. Jamon, T.C. Rojas, J. García-López, A. Fernández
Poster

ANQUE International congress of chemical engineering

24 - 27 junio [Sevilla, España]

Deactivation, Reactivation and memory effect on Co-B Catalyst for sodium borohydride hydrolysis operating in harsh conditions
G.M. Arzac; D. Hufschmidt; A. Fernández; B. Sarmiento; M.A. Jiménez
Poster

Bimetallic-Boron nanoalloys as catalysts for hydrogen generation. New insights into the Co-Ru-BSystem
G.M. Arzac; T. C. Rojas; A. Fernández
Poster

Continuous reactor based on sodium borohydride hydrolysis for stable and versatile hydrogen generation
D. Hufschmidt; G.M. Arzac; A. Fernández; B. Sarmiento; M.A. Jiménez
Poster

Synthesis and Characterization of supported Co Catalysts for Hydrogen Generation by Magnetron sputtering
M. Paladini; G.M. Arzac; V. Godinho; A. Fernández
Poster

1st Al-Nanofunc Workshop: Advanced Microstructural Characterization of Nanomaterials

5 - 6 julio [Sevilla, España]

Determination of nanostructure and chemical composition by TEM techniques in a complex CrAl(Y)N multilayered
T.C. Rojas; S. Domínguez-Meister; M. Brizuela; A. García-Luis; A. Fernández; J.C. Sánchez-López
Comunicación oral

TEM of hybrid Au nanoparticles capped with allylamine
Lionel Cervera Gontard; Vanessa Valembois; Asunción Fernández; Takeshi Kasama; Rafal Dunin-Borkowski; Stéphane Lucas
Comunicación oral

Comparative oxidation resistance of CrAlN, CrAlYN and CrAlZrN films by electron microscopies and EELS techniques
T.C. Rojas; S. Domínguez-Meister; S. El Mrabet; M. Brizuela; A. García-Luis; J.C. Sánchez-López
Poster

Microstructural characterization of magnetron sputtered porous silicon coatings
Jaime Caballero-Hernandez; Vanda Godinho; Roland Schierholz; Asunción Fernández
Poster

Synthesis and characterization of supported Co catalysts for hydrogen generation by magnetron sputtering
M. Paladini; V. Godinho; G.M. Arzac; A. Fernández
Poster

Exposure of a filter feeding bivalve to gold nanoparticles: Location study by the STEM mode in a SEM-FEG microscope
M. Paladini; V. Godinho; G.M. Arzac; A. Fernández
Poster

European Conference on Nano Films ECNF

16 – 17 junio [Ancona, Italia]

Microstructural changes in chalcogenides films induced by Nanoscratching
Cecchini, R.; Benitez, J. J.; Sánchez-López, J. C.; Fernández, A.
Comunicación oral

Preparation of Nanostructured Co Catalysts for Hydrogen Generation by Magnetron sputtering
M. Paladini; G.M. Arzac; V. Godinho; A. Fernández
Comunicación oral

16th International Symposium on Small Particles and Inorganic Clusters (ISSPIC XVI)

8 – 13 julio [Leuven, Bélgica]

Microwave-assisted synthesis and luminescence of mesoporous Ln-doped YPO4 (Ln=Eu, Ce, Tb and Ce Tb) nanophosphors with lenticular shape
S. Rodriguez-Liviano; F. J. Aparicio; T.C. Rojas; A.B. Hungría; L.E. Chinchilla; M. Ocaña
Poster

IUVSTA WS-66: Friction Under Controlled Environments

9 – 12 julio [Avila, España]

Correlation between (micro)structure and tribo-mechanical behaviour of nitride and carbo-nitride coatings
R. Escobar-Galindo; J.C. Sánchez-López; N.K. Manninen; M. Henriques; S. Carvalho
Comunicación oral

Tribological properties of WSex films prepared by magnetron sputtering
J.C. Sánchez-López; S. Domínguez-Meister
Comunicación oral

13th International Conference on Plasma Surface Engineering (PSE 2012)

15 – 19 septiembre [Garmisch-Partenkinchen, Alemania]

Synthesis and tribological properties of WSex films prepared by magnetron sputtering
J.C. Sánchez-López; S. Dominguez-Meister
Poster

15th European Microscopy Congress (EMC2012)

16 – 21 septiembre [Manchester, Gran Bretaña]

The Co-Ru-B series as catalysts for hydrogen generation: synergistic effect, chemistry and nanostructure
G.M. Arzac; T.C. Rojas; L.C. Gontard; A. B. Hungría; L. E. Chinchilla; A.Fernández

Comunicación oral

A nanoscale characterization with electron microscopies techniques of CrAlYN coatings prepared by magnetron
T.C. Rojas; M. Brizuela; A. García-Luis; A. Fernández; J.C. Sánchez-López
Poster

Materials and Coatings for Extreme Performances: Investigations, Applications, Ecologically Safe Technologies for Their Production and Utilization. Seventh International Conference
24 – 28 septiembre [Crimea, Ucrania]

Hard Heat-Resistant multyalloyed nanocomposite coatings produced by hams-technology
Kiryukhantsev-Korneev Ph. V; Kuptsov K.A.; Sheveyko A.N.; T.C. Rojas; A. Fernández; Shtansky D.V.; Levashov E.A.
Comunicación oral

ASME/STLE International Joint Tribology Conference
7 – 10 octubre [Colorado, Estados Unidos de América]

Tribological Properties of Tialsin Coating: Effect of Silicon and Deposition Conditions on Wear and Adhesion
D. Philippon; V. Godinho; P. Nagy; M.P. Delplancke-Ogletree; A. Fernández
Comunicación oral

CONGRESOS Y REUNIONES NACIONALES / NATIONAL CONGRESSES AND MEETINGS

COMUNICACIONES EN CONGRESOS Y REUNIONES / COMMUNICATIONS IN CONGRESS AND MEETINGS

16° Seminario Ibérico de Química Marina
25-28 enero [Cádiz, España]

Bioensayos de toxicidad para Au disuelto y para nanopartículas de Au-Citrato sobre cinco especies microalgales marinas
I. Moreno-Garrido, C. Trombini, C. García Negrete, A. Lapresta-Fernández, A. Fernández, J. Blasco
Poster

VII Reunión Grupo Especializado de Física del Estado Sólido (GEFES)

23 - 27 enero [Sevilla, España]

3D characterization of electron irradiation effects on Ge
L. Cervera-Gontard, H. Ou, JR Jinschuck, J. Verbeeda, RE Dunir Berkowski
Comunicación Oral

Sintering behavior and microstructure of boron carbide ceramics processed by spark plasma sintering
B.M. Moshtaghioun, R. Poyato, F. Gutiérrez-Mora, A. Muñoz, A. Domínguez-Rodríguez
Comunicación Oral

Amorphous SiOxNy thin films with controlled closed porosity and its effects on refractive index and mechanical properties
V.Godinho; T.C.Rojas; A. Fernández Camacho
Poster

Processing and microstructure of spark plasma sintered 8Y-CSZ/5 vol% SWNT composites
E. Carrillo, R. Poyato, A. Gallardo-López, A. Muñoz
Poster

Opto-Coat 2012. Coatings and surface functionalization of materials for optical applications

31 mayo [Alicante, España]

Microstructural characterization of magnetron sputtered porous silicon coatings
Jaime Caballero-Hernández; Vanda Godinho; Roland Schierholz; Asunción Fernández
Poster

XIII Congreso Nacional Propiedades Mecánicas de Sólidos

26 – 28 septiembre [Alcoy, Alicante, España]

Procesado, microestructura y comportamiento de fractura de compuestos de 3Y-TZP/nanotubos de carbon mono-capa con una dispersión óptima de nanotubos preparados mediante “Spark Plasma Sintering”
R. Poyato, A. Gallardo-López, F. Gutiérrez-Mora, A. Muñoz, A. Domínguez-Rodríguez
Comunicación oral

Estudio de la influencia del contenido de nanotubos de carbono (SWCNTs) en el comportamiento tribológico de compuestos de 3Y-TZP
F. Gutiérrez-Mora, R. Poyato, A. Gallardo-López, A. Muñoz, A. Domínguez Rodríguez
Comunicación oral

Estudio de la influencia del contenido de nanotubos de carbono (SWCNTs) en el comportamiento tribológico de compuestos de 3Y-TZP

F. Gutiérrez-Mora, R. Poyato, A. Gallardo-López, A. Muñoz, A. Domínguez Rodríguez
Comunicación oral

Procesado y microestructura de composites de alúmina y nanotubos de pared simple dispersos con tamaña de grano fino
A. Gallardo-López, R. Poyato, A. Morales-Rodríguez, F. Gutiérrez-Mora, A. Muñoz, A. Domínguez-Rodríguez
Poster

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

PROYECTOS DE INVESTIGACIÓN / RESEARCH PROJECTS

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

Compuestos cerámico/SWNT: procesado, caracterización y propiedades mecánicas Ceramic/SWNT composites: processing, characterization and mechanical properties

Código/Code:	MAT2009-11078
Periodo/Period:	01-10-2009 / 30-09-2012
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	211.750,01 €
Investigador responsable/Research head:	Antonio Muñoz Bernabé
Componentes/Research group:	Arturo Domínguez Rodríguez, Ángela Gallardo López, Felipe Gutiérrez Mora, Ana Morales Rodríguez, Nitin P. Padture, Rosalía Poyato Galán

RESUMEN / ABSTRACT

Con este proyecto se pretende, en primer lugar, la fabricación de materiales compuestos nanoestructurados con matrices cerámicas de Al_2O_3 y ZrO_2 estabilizada con Ytria (8Y-SZP), reforzadas con diferentes concentraciones de nanotubos de carbono (CNTs). En ambos tipos de materiales se partirá de polvos cerámicos de alta pureza (> 99 %) y diferentes tamaños de grano, entre 30 y 40 nm para el Al_2O_3 y entre 20 y 300 nm para el 8Y-SZP. Se utilizarán diferentes técnicas tendentes a lograr la dispersión uniforme de los CNTs en la matriz cerámica, uno de los principales problemas que persisten en la actualidad para la fabricación de este tipo de materiales con características reproducibles y propiedades mecánicas mejoradas respecto de la matriz. En segundo lugar, se pretende estudiar la influencia de la microestructura (segregación en los bordes de grano, distribución de nanotubos, tamaño de grano,...) en las propiedades mecánicas de los materiales obtenidos, a baja temperatura (resistencia a la fractura y al desgaste) y también a alta temperatura (resistencia a la fluencia).

La sinterización se realizará usando técnicas de nueva generación como el horno de microondas (MW) y el spark plasma sintering (SPS), equipos de los que dispone nuestro grupo de investigación y que actualmente se encuentran plenamente operativos. Estas técnicas permiten el sinterizado de las matrices cerámicas en un tiempo corto y, en el caso del SPS, a temperaturas relativamente bajas en comparación con los métodos tradicionales lo que permite la densificación del material conservando el tamaño nanométrico de los polvos cerámicos de partida y sin producir daños importantes en los CNTs.

Los policristales obtenidos serán caracterizados microestructuralmente mediante técnicas de microscopia electrónica de transmisión tales como TEM, HRTEM, EDX y PEELS, microscopia electrónica de barrido de alta resolución (HRSEM), difracción de rayos X y espectroscopia Raman. La caracterización mecánica incluye ensayos de resistencia a la fractura y al desgaste a temperatura ambiente y temperaturas moderadas y ensayos de deformación a alta temperatura, controlando tensión, temperatura y atmósfera de trabajo.

Los parámetros macroscópicos y microscópicos obtenidos se analizarán en términos de los modelos existentes y en caso de no encontrar en ellos la justificación adecuada se intentará

avanzar en la modelización de los mecanismos que contribuyen a la deformación de estos materiales compuestos nanoestructurados y con segundas fases de CNTs.

The aim of this Project is two fold: firstly the fabrication of nanostructured composites of alumina and yttria stabilized zirconia polycrystals (8Y-SZP) reinforced with different concentrations of carbon nanotubes (CNTs). In both materials, the powder used will have a high purity (> 99%) and different grain sizes; between 30 and 40 nm in the case of alumina and between 20 and 300 nm in the case of 8Y-SZP, and secondly to study the influence of the microstructure (grain boundary segregation and nanotubes distribution) on the mechanical properties at low temperature (fracture and wear resistance) as well as at high temperature (creep resistance).

To obtain the polycrystals nanostructured, two techniques of new generation (both equipments belong to our research group) will be used, such as the microwave furnace (MW) and the spark plasma sintering (SPS); this techniques allow to sinter the powder in a short time and at low temperature in the case of SPS which is a fundamental requirement to keep the nanostructure of the as-received powder without destroying the CNTs.

The obtained polycrystals will be microstructurally characterized using techniques such as SEM, TEM, HRTEM, EDX, PEELS, X-Ray diffraction and Raman spectroscopy. The mechanical characterization includes fracture and wear resistance tests at moderate temperature and creep test at high temperature as a function of stress, temperature and controlled atmosphere.

The macroscopic and microscopic parameters will be analyzed using the existent models and, if needed, by formulating new models in order to establish the master chart of the mechanical behaviour of these ceramics composites.

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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 / 31-12-2013
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

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 / 15-03-2014
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

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. En cuanto a la técnica de aleado mecánico continuaremos el análisis de los complejos procesos de

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

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

TRANSFERENCIA / TECHNOLOGICAL TRANSFER

PATENTES / PATENTS

Chemistries and processes for tuning the Curie temperature of gamma-ferri nanoparticles for magnetocaloric applications by controling the oxidation kinetics
Inventores: Ipus-Bados, Jhon Jairo; Ucar, Huseyin; McHenry, Michael E.; Laughlin, David E.
Tipo de Patente: Patente Estadounidense
Fecha Solicitud: 3 febrero 2012
Entidad/es Titular/es: Center for Technology Transfer and Enterprises Creation, Carnegie Mellon University

CONVENIOS Y CONTRATOS / CONTRACTS AND AGREEMENTS

Characterization of magnetocaloric materials

Organismo Financiador/Financial source:	Empresa ERASTEEL (Francia)
Investigador responsable/Research head:	Victorino Franco García

PUBLICACIONES / PUBLICATIONS

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

The Magnetocaloric Effect and Magnetic Refrigeration Near Room Temperature: Materials and Models
V. Franco, J.S. Blázquez, B. Ingale, and A. Conde
Annual Review of Materials Research, **42** (2012) 305-342
DOI: 10.1146/annurev-matsci-062910-100356

In the past 20 years, there has been a surge in research on the magnetocaloric response of materials, due mainly to the possibility of applying this effect for magnetic refrigeration close to room temperature. This review is devoted to the main families of materials suitable for this application and to the procedures proposed to predict their response. Apart from the possible technological applications, we also discuss the use of magnetocaloric characterization to gain fundamental insight into the nature of the underlying phase transition.

Excellent magnetocaloric properties of melt-extracted Gd-based amorphous microwires

N.S. Bingham, H. Wang, F. Qin, H.X. Peng, J.F. Sun, V. Franco, H. Srikanth and M.H. Phan
Applied Physics Letters, **101** (2012) 102407 (5 pages)
DOI: 10.1063/1.4751038

We report upon the excellent magnetocaloric properties of Gd53Al24Co20Zr3 amorphous microwires. In addition to obtaining the large magnetic entropy change (−ΔSM ∼ 10.3 J/kg K at TC ∼ 95 K), an extremely large value of refrigerant capacity (RC ∼ 733.4 J/kg) has been achieved for a field change of 5 T in an array of forty microwires arranged in parallel. This value of RC is about 79% and 103% larger than those of Gd (∼410 J/kg) and Gd5Si2Ge1.9Fe0.1 (∼360 J/kg) regardless of their magnetic ordering temperatures. The design and fabrication of a magnetic bed made of these parallel-arranged microwires would thus be a very promising approach for active magnetic refrigeration for nitrogen liquefaction. Since these microwires can easily be assembled as laminate structures, they have potential applications as a cooling device for micro electro mechanical systems and nano electro mechanical systems.

Magnetic refrigerants with continuous phase transitions: Amorphous and nanostructured materials

V. Franco, A. Conde
Scripta Materialia, **67** (2012) 594-599
DOI: 10.1016/j.scriptamat.2012.05.004

An overview of the magnetocaloric properties of amorphous and nanostructured materials which undergo second-order phase transitions is presented, paying special attention to open problems which need to be solved to enhance the applicability of these materials for magnetic refrigeration. The potential of composites is also highlighted, as well as the current status of modeling these particular subset of magnetocaloric materials.

Extension of the classical theory of crystallization to non-isothermal regimes: Application to nanocrystallization processes

J.S. Blázquez, J.M. Borrego, C.F. Conde, A. Conde, S. Lozano-Pérez
Journal of Alloys and Compounds, **544** (2012) 73-81
DOI: 10.1016/j.jallcom.2012.08.002

The non-isothermal kinetics of primary crystallization processes is studied from numerically generated curves and their predictions have been tested in several nanocrystallization processes. Single processes and transformations involving two overlapped processes in a non-isothermal regime have been generated and deviations from isokinetic behavior are found when the overlapped processes have different activation energies. In the case of overlapped processes competing for the same type of atoms, the heating rate dependence of the obtained Avrami exponent can supply information on the activation energies of each individual processes. The application to experimental data of nanocrystallization processes is consistent with a limited growth approximation. In the case of preexisting crystallites in the as-cast samples, predictions on the heating rate dependence of the obtained Avrami exponents of multiple processes have been confirmed.

Analysis of nanocrystallization kinetics and crystal size distribution under limited growth approach
J.S. Blázquez, C.F. Conde, A. Conde
Journal of Alloys and Compounds, **536S1** (2012) 550-553
DOI: 10.1016/j.jallcom.2011.11.109

Two different simulation approaches have been used to describe nanocrystallization processes: a limited growth approach, which is an extension from instantaneous growth approximation, and an average soft impingement simulation where spherical crystallites grow to a size for which the corresponding region depleted in Fe (or the element enriched in crystalline phase) is comparable to the average distance between crystallites. Both simulations agree describing a local Avrami exponent which decreases down to ~1 as crystallization fraction increases. Experimental data for evolution of crystal size and crystal size distribution are reproduced.

Comparison of equivalent ball milling processes on $Fe_{70}Zr_{30}$ and $Fe_{70}Nb_{30}$
J.S. Blázquez, J.J. Ipus, C.F. Conde, A. Conde
Journal of Alloys and Compounds, **536S-1** (2012) S9-S12
DOI: 10.1016/j.jallcom.2011.11.084
2,289
Dynamical analysis of the movement of a single ball in a planetary ball mill yields a cubic law with the frequency of the mill for the power released during the process. This fact has been explored for two binary compositions (Fe70Zr30 and Fe70Nb30) using two different milling frequencies. The experimental techniques used, in general, support the predictions of the equivalent milling time model. However, some deviations appear which could be ascribed to differences in the temperature inside the vial during milling.

Influence of isochronal annealing on the microstructure and magnetic properties of Cu-free HITPERM $Fe_{40.5}Co_{40.5}Nb_7B_{12}$ alloy
P. Gupta, Tapas Ganguli, A. Gupta, A.K. Sinha, S.K. Deb, P. Svec, Jr., and V. Franco
Journal of Applied Physics, **111** (2012) 113518 (10 pages)
DOI: 10.1063/1.4728161

Systematic study of the effect of isochronal annealing on the structure and magnetic properties of Cu-free HITPERM alloy (Fe40.5Co40.5Nb7B12) is described herein. Mössbauer spectroscopy (MS) and anomalous x-ray diffraction (XRD) measurement at Fe K-edge (7.112 keV) jointly provide clear evidence for the presence of atomically ordered α'-FeCo (B2 structure) phase as a nanocrystalline ferromagnetic phase. Being a short range order probe, Mössbauer spectroscopy also confirms the development of an additional non-magnetic Nb-rich phase in the nanocrystalline specimens (annealed above 723 K) with simultaneous lowering of the volume fraction of ferromagnetic phases. The fraction of Fe atoms in the non-magnetic phase is ~15% upon annealing at 773 K for 1 h, which increases gradually and reaches to as high as ∼19% after annealing at 923 K. This phase was not detected by XRD and transmission electron microscopy (TEM) measurements, which may be attributed to tiny crystallite size and/or high degree of disorder. In the second stage of crystallization, i.e., above 923 K, the alloy becomes fully crystalline and a stable, hard magnetic fcc-(FeCo)23B6 type phase was observed as a main boride phase along with soft magnetic α′-FeCo phase and Nb rich non-magnetic phase. Thermo magnetic measurement evidenced re-crystallization process as a considerable decrease in magnetization at the second transformation stage. Simultaneous lowering of the volume fraction of magnetic phases with the formation of non-magnetic phase provides convincing origin for the decrease in magnetization at the second crystallization stage.

The magnetocaloric effect of partially crystalline Fe-B-Cr-Gd alloys

J.Y. Law, V. Franco and R.V. Ramanujan

Journal of Applied Physics, **111** (2012) 113919 (6 pages)

DOI: 10.1063/1.4751038

We report upon the excellent magnetocaloric properties of Gd53Al24Co20Zr3 amorphous microwires. In addition to obtaining the large magnetic entropy change (−ΔSM ∼ 10.3 J/kg K at TC ∼ 95 K), an extremely large value of refrigerant capacity (RC ∼ 733.4 J/kg) has been achieved for a field change of 5 T in an array of forty microwires arranged in parallel. This value of RC is about 79% and 103% larger than those of Gd (∼410 J/kg) and Gd5Si2Ge1.9Fe0.1 (∼360 J/kg) regardless of their magnetic ordering temperatures. The design and fabrication of a magnetic bed made of these parallel-arranged microwires would thus be a very promising approach for active magnetic refrigeration for nitrogen liquefaction. Since these microwires can easily be assembled as laminate structures, they have potential applications as a cooling device for micro electro mechanical systems and nano electro mechanical systems.

The magnetocaloric effect and critical behavior in amorphous $Gd_{60}Co_{40-x}Mn_x$ alloys

Z.G. Zheng, X.C. Zhong, H.Y. Yu, V. Franco, Z.W. Liu and D.C. Zeng

Journal of Applied Physics, **111** (2012) 07A922 (3 pages)

DOI: 10.1063/1.3673860

The amorphous alloys Gd60Co40−xMnx (x = 0, 5, 10, 15) were prepared by melt spinning. The Curie temperature, Tc, increases monotonously with Mn addition, ranging from 198 K for x = 0 to 205 K for x = 15, while the maximum values of −ΔSM under the applied field change from 0 to 5 T are 7.7, 7.1, 6.2 and 5.4 J·kg−1·K−1 for x = 0, 5, 10, and 15, respectively. All samples undergo a second order ferri-paramagnetic phase transition. The critical behavior around the transition temperature is investigated in detail, using both the standard Kouvel-Fisher procedure as well as the study of the field dependence of the magnetocaloric effect. Results indicate that the obtained critical exponents are reliable, and that the present alloys exhibit local magnetic interaction.

Enhancement of the magnetocaloric effect in composites: Experimental validation

S.C. Paticopoulos, R. Caballero-Flores, V. Franco, J.S. Blázquez, A. Conde, K.E. Knipling, M.A. Willard
Solid State Communications, **152** (2012) 1590-1594
DOI: 10.1016/j.ssc.2012.05.015

Recent calculations have shown that the refrigerant capacity (RC) of magnetic refrigerants can be enhanced using multiphase materials or composites, which expand the temperature range over which a significant magnetic entropy change can be obtained. This work is a systematic experimental validation of the improvement of RC (RCI) using layered composites comprised of two Fe88−2yCoyNiyZr7B4Cu1 amorphous alloy constituents, with y=8.25 and y=11 compositions. RCI has a nonmonotonic dependence on the applied magnetic field H and the fraction x of the two constituent phases. In contrast to common assumptions, the composite has a smaller RCI than its constituent phases for small values of H and x, and there are critical values of each for which RCI is maximized. This work demonstrates the outstanding agreement between the experimental results and the continuous curves predicted by numerical calculations, indicating that this approach can be used to design magnetic refrigerant materials with enhanced magnetocaloric response for moderate magnetic fields.

Enhancement of the magnetic refrigerant capacity in partially amorphous Fe70Zr30 powders obtained by mechanical alloying

J.S. Blázquez, V. Franco, A. Conde
Intermetallics, **26** (2012) 52-56
DOI: 10.1016/j.intermet.2012.03.011

After mechanical alloying Fe70Zr30 composition from pure starting powders, an amorphous phase with Curie temperature TC = 244 K and an intermetallic compound (that should be non-stoichiometric Zr-rich fcc Fe2Zr phase) with TC = 355 K are formed. Residual α-Fe crystallites are also found. The multiphase character of this system yields a non-monotonic dependence of the magnetocaloric effect (characterized by the refrigerant capacity, RC) on the fraction of phases. Among the samples studied in this work, RC is enhanced for samples with the highest fraction of intermetallic compound, although the maximum magnetic entropy change monotonically decreases with the increase of the fraction of this phase. This behaviour agrees with the predicted one for biphasic systems.

Overview of Amorphous and Nanocrystalline Magnetocaloric Materials Operating Near Room Temperature

Huseyin Ucar, John J. Ipus, V. Franco, M.E. McHenry, D.E. Laughlin
JOM, **64** (2012) 782-788
DOI: 10.1007/s11837-012-0349-6

The observation of a giant magnetocaloric effect in Gd5Ge1.9Si2Fe0.1 has stimulated the magnetocaloric research in the last two decades. However, the high price of Gd and its proclivity to corrosion of these compounds have prevented their commercial use. To reduce raw materials cost, transition metal-based alloys are investigated to replace rare earth-based materials. Environmental considerations, substitution for scarce and strategic elements, and cost considerations all speak to potential contributions of these new materials to sustainability. Fe-based soft amorphous alloys are believed to be promising magnetic refrigerants. Efforts in improving the refrigeration capacity (RC) of refrigerants mainly rely on broadening the magnetic entropy change. One promising technique is to couple two phases of magnetic materials with desirable properties. Second is the investigation of nanoparticle synthesis routes, with ball milling being the most widely used one. The motivation for the nanoparticles synthesis is rooted in their inherent tendency to have distributed exchange coupling, which will broaden the magnetic entropy curve. As proven with the cost analysis, the focus is believed to shift from improving the RC of refrigerants toward finding the most economically advantageous magnetic refrigerant with the highest performance.

Square-modulated differential thermal analysis: measuring method
J. del Cerro, J. Manchado, F.J. Romero and M.C. Gallardo
Measurement Science and Technology, **23** (2012) 035003
DOI: 10.1088/0957-0233/23/3/035003

A conduction calorimetry technique has been used to characterize qualitatively and quantitatively first-order phase transitions. A series of long-period square thermal pulses superimposed on a temperature ramp (square-modulated differential thermal analysis) is applied to the sample and the heat flux is measured by highly sensitive fluxmeters. In a single run, the specific heat behavior is determined and the heat flux due to the temperature dependence of the specific heat is obtained. This contribution is subtracted from the measured heat flux and the value of the latent heat is extracted. The analysis of the fluxmeter's response allows us to know if it is necessary to carry out a second experiment in order to study the kinetics of the transformation in depth. In this second run, the temperature ramp is only used to obtain the DTA trace. Four materials with first-order phase transitions, ranging from narrow coexistence intervals to very large ones and exhibiting smooth or very abrupt transformations, have been studied using this technique.

Magnetic multilayers as a way to increase the magnetic field responsiveness of magnetocaloric materials
R. Caballero-Flores, V. Franco, A. Conde, L.F. Kiss, L. Péter, I. Bakonyi
J. Nanosci Nanotechnolol., **12** (2012) 7432-6

The magnetocaloric response of Ni-Cu based multilayers has been studied with the aim of optimizing their magnetic field dependence. In contrast to the behavior of single phase materials, whose peak magnetic entropy change follows a power law with exponents close to 0.75, multilayering leads to exponents of -1 for an extended temperature span close to the transition temperature. This demonstrates that nanostructuring can be a good strategy to enhance the magnetic field responsiveness of magnetocaloric materials.

Analysis of the magnetic anisotropy in Fe-Co-Nb-B alloys partially amorphized by mechanical alloying
J.J. Ipus, J.S. Blázquez, V. Franco, A. Conde, S. Lozano-Pérez

Physics Express, **2** (2012) 1-7

Effective magnetic anisotropy evolution of mechanically alloyed (Fe100-xCox)0.75Nb10B15 powders has been analyzed using three contributions: average magnetocrystalline, average short range magnetoelastic and long range magnetoelastic anisotropies. The effect of the presence of amorphous phase on the average magnetocrystalline contribution has been taken into account modifying previous models. Once microstrains can be considered saturated, the analysis supplies information about the relative values of exchange stiffness constant, which increases as the Curie temperature increases.

CONGRESOS Y REUNIONES / CONGRESS AND MEETINGS

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

COMUNICACIONES EN CONGRESOS Y REUNIONES / COMMUNICATIONS IN CONGRESS AND MEETINGS

VII Reunión Grupo Especializado de Física del Estado Sólido (GEFES)
23 – 23 enero [Sevilla, España]

V. Franco, A. Conde
Conferencia invitada

B. Ingale, V. Franco, A. Conde, K.E. Knipling, M.A. Willard
Poster

C. Romero-Muñiz, V. Franco, A. Conde
Poster

D. Doblas, V. Franco, A. Conde, A.V. Svalov, G.V. Kurlyandskaya
Poster

J.S. Blázquez, C.F. Conde, A. Conde
Poster

European Symposium of Martensitic Transformations, ESOMAT 2012
9 – 16 septiembre [Saint-Petersburg, Rusia]

“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.K.H. Salje
Poster

TMS 2012, Orlando-USA,
2012. Oral. V. Franco, R. Caballero-Flores, A. Conde, L.F. Kiss, L. Péter, I. Bakonyi.

Thermag V, Grenoble-Francia,
2012. Oral. D. Doblas, V. Franco, A. Conde, A.V. Svalov, G.V. Kurlyandskaya.

International Conference on Superconductivity and Magnetism (ICSM2012), Estambul-Turquía,
2012. Póster. L. González, T. Sánchez, J.S. Blázquez, V. Franco, J.J. Suñol, B. Hernando.

ICFMD 2012, Pune-India, 2012. Póster. B. Ingale, V. Franco, A. Conde.

FORMACION / TRAINING

TESIS DOCTORALES/ DOCTOR DEGREE THESIS

Título:	**Estudio Calorimétrico de Transiciones de Fase con un amplio intervalo de Coexistencia**
Autor:	Julia Manchado Ligioiz
Directores:	Jaime del Cerro González y Francisco Javier Romero Landa
Calificación:	Apto “Cum Laude”
Centro:	Universidad de Sevilla

FORMACIÓN DE GRADUADOS / MASTER DEGREE THESIS

Título:	**Simulación del efecto magnetocalórico en materiales multifásicos**
Autor:	Carlos Romero Muñiz
Directores:	Victorino Franco García y Alejandro Conde Amiano
Grado:	Ingeniería de Materials
Centro:	Facultad de Física. Universidad de Sevilla
Año Académico:	2011-12

Título:	**Efecto magnetocalórico en materiales multifásicos**
Autor:	David Doblas Jiménez
Directores:	Victorino Franco García y Alejandro Conde Amiano
Grado:	Ingeniería de Materials
Centro:	Facultad de Física. Universidad de Sevilla
Año Académico:	2011-12

Título:	**Desarrollo de un trazador de histéresis para materiales magnéticos blandos**
Autor:	David Doblas Jiménez
Directores:	Victorino Franco García
Grado:	Ingeniería de Materials
Centro:	Facultad de Física. Universidad de Sevilla
Año Académico:	2012-13

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.
- 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)

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: Lda. Olga Montes Amorín (CicCartuja) y 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: Ldo. 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 super-

ficie 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: Ldo. 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: Dr. 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:

- Torno de control numérico computerizado, modelo HASS TM1
  A computer numeric control turing machine, model HASS TM1
- Taladro vertical, modelo ERLO TSAR32
  A vertical drill, model ERLO TSAR32
- Un par de tornos manuales o semiautomáticos, modelos PINACHO TURN180 y SUPER 11 CD EMCO
  A couple of turning machines: models PINACHO TURN180 and SUPER 11 CD EMCO

Responsable/ Responsible: Ing. Enrique Jiménez Roca
Personal Técnico: 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)
Espectroscopias de Fotoemisión (XPS, UPS)
Microscopias Electrónicas de Transmisión y Barrido (TEM y SEM) Espectroscopias Electrónicas (Auger, REELS, EDX, PEELS)
Difracción de Rayos X
Absorción de Rayos X (EXAFS, XANES)
Dispersión de Iones (ISS, RBS)
Microscopias de Aproximación de puntas (AFM, STM)

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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: 27 junio al 1 de julio de 2012
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. Métodos Experimentales de Análisis de Sólidos | Dr. Richard Lambert
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
Deposición de Láminas Delgadas mediante la Utilización de Plasmas| 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. Asunción Fernández Camacho
Microscopía Electrónica: PEELS y EFTEM| Dra. Cristina Rojas Ruiz
Microscopía Electrónica de Barrido| Dra. María del Carmen Jiménez de Haro
Métodos de Determinación de Espesores de Capas Finas | Dr. Juan Pedro Espinós Manzorro
La Difracción de Rayos X: Generalidades | Dr. Angel Justo Erbez
Análisis de Texturas: Difracción de Rayos X y Espectroscopía Raman | Dr. Juan Carlos González González
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 | Lda. 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 | Dra. Ana Borrás | Dr. Carlos Sánchez | Dr. Rafael Alvarez
Lda. Antonia Terriza

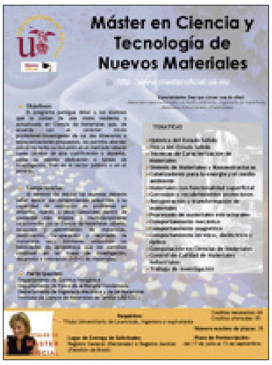
MÁSTER / MASTER

MÁSTER EN ESTUDIOS AVANZADOS EN QUÍMICA

Organizado por la Universidad de Sevilla
Coordinadora: Dra. Mª Angeles Alvarez Rodríguez
Fecha de Celebración: Curso Académico 2011-12
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

Seminario-Laboratorio de espectroscopía Avanzada (Créditos: 8) |Dr. Ernesto Carmona (IIQ) | Dra. María Dolores Alba | Dr. Antonio M. Gil (US) | Dra. Maria Angeles Pradera (US) | Dr. José M. Fernández-Bolaños (US)
Catálisis Homogénea y Heterogénea (Créditos: 4) | Dr. José Antonio Odriozola Gordón | Dra. Francisca Romero Sarriá

MÁSTER EN CIENCIA Y TECNOLOGÍA DE NUEVOS MATERIALES


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 2011-12
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 Blázquez Gámez | Dr. Victorino Franco García
Comportamiento Térmico, Dieléctrico y Óptico (Créditos: 2.85) | Dr. Jaime del Cerro González | Dr. Francisco Javier Romero Landa

Computación en Ciencia de Materiales (Créditos: 3.25) | Dra. Regla Ayala Espinar | Dr. Antonio Córdoba Zurita (US) | Dr. Felipe Gutiérrez Mora

OTROS / OTHER

Máster Oficial y Doctorado Interuniversitario en "Biotecnología Avanzada"

Módulo de Nanotecnología
Dra. Aránzazu Díaz Cuenca, Dr. José Jesús Benítez Jiménez
Lugar: Universidad de Málaga
Organizado por la Universidad de Málaga y la Universidad Internacional de Andalucía

Máster Universitario en Biotecnología Sanitaria

Curso de Nanotecnología
Dra. María Jesús Sayagués de Vega
Lugar: Universidad Pablo de Olavide

Máster en Formación del Profesorado de Educación Secundaria

Ciencia, Tecnología y Sociedad
Dra. Adela Muñoz Páez
Lugar: Universidad de Sevilla

Máster Profesorado de ESO y Bachillerato, FP

Complementos de formación disciplinar en Física y Química
Dr. J. Sebastián Blázquez
Lugar: Universidad de Sevilla

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

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.

CONFERENCIAS INVITADAS IMPARTIDAS POR PERSONAL DEL ICMS
INVITED CONFERENCES BY PERSONNEL OF THE ICMS

Sample Controlled Thermal Analysis: A Challenge for the Kinetics analysis of Solid State reactions and the synthesis of materials
José Manuel Criado Luque
Academia de Ciencias de Rumania
Bucarest (Rumania), Febrero

Kinetic analysis of solid state reactions. Some experimental and mathematical issues
Luis A. Pérez Maqueda
Ecole Nationale Supéreure des Mines
Saint Etienne (Francia), Marzo

Gold nanoparticles in heterogeneous oxidation catalysis: structured catalysts
Miguel Angel Centeno Gallego
Conferencia Plenaria invitada en la Réunion Plénière du GDR OR NANO
Universidad de Poitiers (Francia), 20 Marzo

Desrrollo, optimización y caracterización de materiales foto-funcionales para aplicaciones medioambientales
José Antonio Navío Santos
Instituto de Ciencia de Materiales de Madrid (ICMM)
Madrid (España), 27 abril

Kinetic analysis and kinetic control of solid-state reactions of interest in materials science
Luis A. Pérez Maqueda
Universidad de Pardubide
Pardubice (República Checa), Junio

CONFERENCIAS Y SEMINARIOS IMPARTIDOS EN EL ICMS
CONFERENCES AND SEMINAR IN THE ICMS

7 de marzo| **Mechanism of Propylene Epoxidation on Gold/Titanosilicate Catalysts**
Prof. S. Ted Oyama
The University of Tokyo Department of Chemical Systems Engineering 7-3-1 Hongo, Bunkyo-ku, Tokyo 113-8656, Japan

19 de marzo| **Intercalation of Hydrated Fe^{2+} and Fe^{3+} in the Vermiculites from Santa Olalla and Ojén. A Mössbauer spectroscopic investigation**
Prof. Anton Lerf

Walther-Meißner-Institut der Bayerischen Akademie der Wissenschaften Walther-Meißner-Straße 8
85748 Garching

8 de noviembre| **Fluorescent noble metal nanoclusters for biomedical applications**
Dr. Xavier Le Guebel
Centro Andaluz de Nanomedicina y Biotecnología

Estancias y Visitas de Personal del ICMS en otros Centros

Personnel of the ICMS in Other Laboratories

Universidad de Barcelona. Departamento de Estructura y Constituyentes de la Materia		
Barcelona, España	**M. Carmen Gallardo Cruz**	[2 días]
	José María Martín Olalla	[2 días]
	Francisco Javier Romero Landa	[2 días]
Romanian National Research Council, CNCS		
Bucarest, Rumanía	**Miguel Angel Centeno Gallego**	[2 días]
Instituto de Ciencia de Materiales de Madrid (ICMM)		
Madrid, España	**José Antonio Navío Santos**	[3 días]
Universidad Nacional		
Bogotá, Colombia	**Miguel Angel Centeno Gallego**	[4 días]
Instituto de Catálisis. Academia de Ciencias de Bulgaria		
Sofia, Bulgaria	**Tomás Ramírez Reina**	[1 semana]
	Svetlana Ivanova	[1 semana]
Laboratoire de Archeologie Moleculaire et Structurale		
Paris, Francia	**José Luis Pérez Rodríguez**	[1 semana]
Ernst Ruska-Centre (ER-C) for Microscopy and Spectroscopy with Electrons		
Alemania	**Lionel Cervera Gontard**	[1 semana]
	Roland Schierholz	[1 mes]
Cavendish Laboratory. Department of Physics, University of Cambridge		
Reino Unido	**Mauricio Calvo Roggiani**	[1 semana]
	Carlos García Negrete	[6 meses]
Canadian Light Synchrotron		
Saaskatoon, Canadá	**Juan Carlos Sánchez López**	[1 semana]

Facultad de Ciencias Físicas y Matemáticas, Departamento de Ciencia de los Materiales, Universidad de Chile

Santiago, Chile	**José Manuel Criado Luque**	[2 semanas]
	María Jesús Diánez Millán	[2 semanas]

Walther-Meissner- Institut (Academia de Ciencias de Baviera)

Garching, Alemania	**Juan Poyato Ferrera**	[3 semanas]

Center for Electron Nanoscopy, Danish Technical University

Dinamarca	**Lionel Cervera Gontard**	[3 semanas]

Departamento de Ciencia de Materiales e Ingeniería. Universidad de Sheffield

Sheffield, Reino Unido	**Luis A. Pérez Maqueda**	[1 mes]
	Antonio Perejón Pazo	[4 meses]

Departamento de Ciencia de Materiales e Ingeniería. Universidad de Sheffield

Sheffield, Reino Unido	**Luis A. Pérez Maqueda**	[1 mes]

Institute of Experimental Physics. Slovak Academy of Sciences

Kosice, Eslovaquia	**Victorino Franco uis A. Pérez Maqueda**	[1 mes]

Université Louis Pasteur

Estrasburgo, Francia	**José Antonio Odriozola Gordón**	[1 mes]
	Leidy Marcela Martínez Tejada	[3 semanas]

Universitè de Namur

Bélgica	**Vanda Godinho Fortio**	[2 meses]

Universidad de Salerno

Salerno, Italia	**Sebastián Murcia López**	[3 meses]

Universidad de las Palmas de Gran Canarias

Las Palmas de Gran Canarias, España	**Julie J. Murcia Mesa**	[3 meses]

Institute of Chemical Engineering Sciences, FORTH/ICE-HT

Patras, Grecia	**Tomás Ramírez Reina**	[3 meses]

Laboratorio de Catálisis y Espectroquímica, CNRS-Universidad de Caen

Caen, Francia	**Sandra Palma del Valle**	[3 meses]

Materials Department of the University of Oxford

Oxford, Reino Unido	**Adela Muñoz Páez**	[3 meses]

ESTANCIAS Y VISITAS DE INVESTIGADORES EN EL ICMS

PERSONNEL OF THE OTHER LABORATORIES IN THE ICMS

Grupo de Ingeniería Química, Departamento de Química Aplicada, Facultad de Ciencias Químicas de San Sebastián, UPV/EHU		
San Sebastián, España	**Oihane Sanz Iturralde**	[1 semana]
Universidad de McMaster		
Hamilton, Canadá	**Manuel David Abad Roldán**	[1 semana]
National University of Science and Tecnology (MISIS)		
Rusia	**Andrey Bondarev**	[1 semana]
University of Oxford		
Reino Unido	**Sergio Lozano Pérez**	[2 semanas]
Instituto de Catálisis, Academia de Ciencias de Bulgaria		
Sofia, Bulgaria	**Tatyana Tabakova**	[2 semanas]
	Vasko Idakiev	[2 semanas]
Walther-Meissner-Institute. Bayerischen Akademie der Wissenschaften		
Múnich, Alemania	**Anton Lerf**	[8 días]
Fitzwilliam College, Department of Chemistry, University of Cambridge		
Reino Unido	**Adam Earl Fraser**	[1 mes]
Universitè de Namur		
Bélgica	**Benjamín Bera**	[1 mes]
l'Institut de Recherche en Sciences de la vie NARILIS		
Bélgica	**Pavel Moskovkin**	[1 mes]
	Stéphane Lucas	[1 mes]
Instituto Nacional del Carbón (INCAR)-CSIC		
Oviedo, España	**Rocío Jiménez Carmona**	[1 mes]
Universidad Nacional de Colombia		
Colombia	**Natalia Jaramillo Gómez**	[2 meses]
Universidad de Antioquia		
Medellin, Colombia	**Laila Galiano Botero**	[3 meses]
Universidad de Cagliari		
Cagliari, Italia	**Laura Abis**	[6 meses]

OTRAS ACTIVIDADES / OTHER ACTIVITIES

FERIA DE LA CIENCIA / FAIR OF SCIENCE

La Feria de la Ciencia (10 al 12 de mayo de 2012, 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 (10 to 12 May 2012, 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 12 al 16 de Noviembre de 2012, 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. Juan Carlos Sánchez López, A. Ruiz Conde y P.J. Sánchez Soto

12 de Enero. I.E.S. Ruiz Gijón (Utrera)

Charlas

12 de Noviembre de 2012. Dra. Ana Isabel Becerro Nieto

Titulo: "Ciencia de la luz y color”

15 de Noviembre de 2012. Dr. Juan Pedro Holgado Vázquez

Titulo: “Química ecosostenible (verde): nuevos productos y menos residuos”

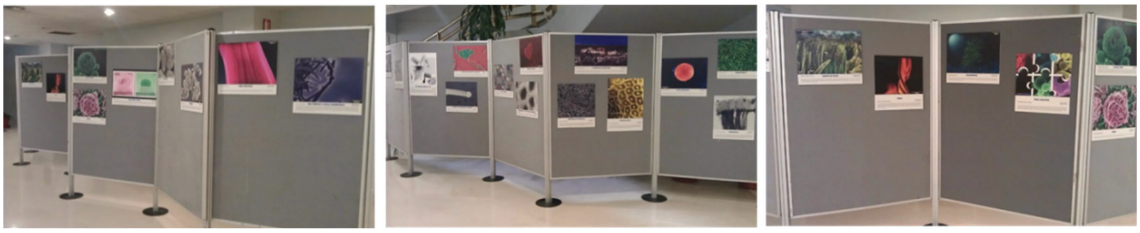
Café con Ciencia

5 de noviembre de 2012. Dra. Asunción Fernández Camacho. Coloquio con estudiantes. Tema: “Hidrógeno, el vector de transporte para la energía sostenible del futuro: un reto científico”

5 de noviembre de 2012. Dr. Alfonso Caballero Martínez. Coloquio con estudiantes. Tema: “Energía sin emisiones: desafíos para la producción económica de Hidrógeno”

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).



Las fotografías corresponden a materiales con variada funcionalidad: desde catalizadores hasta dispositivos ópticos, pasando por sensores de diversas sustancias. Dichos materiales han sido preparados previamente por científicos (en su mayoría, del ICMS) como parte de su trabajo de investigación. Las micrografías expuestas, retocadas y coloreadas con fines artísticos, han sido seleccionadas teniendo en cuenta su apariencia y la relevancia del material que representan.

ACTO DE HOMENAJE / TRIBUTE

En el marco del **I Workshop Al-Nanofunc**, organizado por las doctoras **Asunción Fernández Camacho** y **Vanda Godinho** y celebrado en la Casa de la Ciencia, del CSIC, la Universidad de Sevilla ha rendido homenaje al profesor **Guillermo Munuera Contreras**. Ponente en este workshop sobre nanotecnología, Munuera recibió tras su conferencia una placa de manos del vicerrector de Investigación, Manuel García León. Con este homenaje, la US reconoce la excelente trayectoria de este catedrático de Química Inorgánica, jubilado el pasado mes de septiembre.

Licenciado en 1962 por la Universidad de Sevilla, Guillermo Munuera realizó un posgrado en la Universidad de Bristol (Reino Unido). Tras su paso por las universidades de Murcia, Córdoba y Santander, en 1981 se incorpora como catedrático en la recién creada Facultad de Química, donde dirige el departamento de Química General. En 1986 se convierte en el primer director del Instituto de Ciencia de Materiales de Sevilla (ICMS), centro mixto CSIC-US, cargo que desempeña hasta 1991.

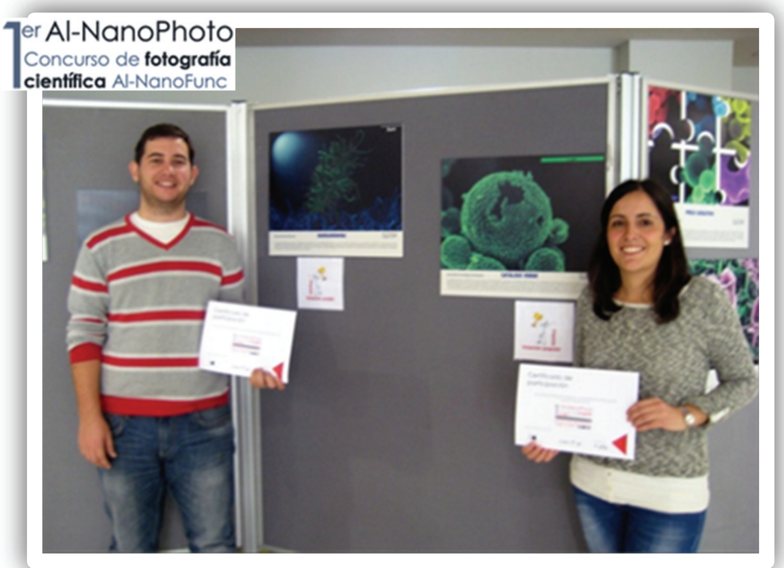
De 1988 a 2011 ha dirigido el Servicio de Espectroscopía de Fotoelectrones de Rayos X (XPS) con el que aún colabora activamente en el Citius (Centro de Investigación, Tecnología e Innovación de la Universidad de Sevilla). Autor de numerosas publicaciones, ha sido uno de los químicos más citados entre 1987 y 1997, es un referente en el tema de la Catálisis y ha ganado numerosos premios, como el de la Real Sociedad Química Española (1968); Alfonso X el Sabio, CSIC (1977) o Ramón Areces (1983), entre otros.

Desde 2005 es miembro de la Real Academia Sevillana de Ciencias, y a partir de 2011 forma parte de su Consejo de Gobierno.

Los catedráticos Manuel García León (Vicerrector de Investigación la US), Guillermo Munuera Contreras, Julián Martínez Fernández y Asunción Fernández Camacho. Foto: Victoria Ramírez. Fuente: Diario de Sevilla

CONCURSO DE FOTOGRAFÍA / PHOTO CONTEST

Al-NanoPhoto es un certamen de fotografía científica convocado por el proyecto europeo **Al-NanoFunc** cuyo objetivo es acercar la ciencia y la tecnología a los ciudadanos mediante una visión artística y estética a través de imágenes científicas.



Manuel Macías y Rosa Pereñíguez, autores de las fotografías ganadoras

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