ICMS

Instituto de Ciencia de Materiales de Sevilla

Memoria de Actividades
Annual Report

Consejo
Superior de
Investigaciones
Científicas

Universidad
de Sevilla

Junta de
Andalucía

2017

Consejo Superior de Investigaciones Científicas
Universidad de Sevilla
Junta de Andalucía

INSTITUTO DE CIENCIA DE MATERIALES DE SEVILLA

Memoria de Actividades
Annual Report
2017

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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
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Diseño y Maquetación
José Carlos Rivero Cabello

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

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El ICMS en 2017
ICMS in 2017

Presentación Presentation

A través de esta Memoria 2017, el Instituto de Ciencia de Materiales de Sevilla (ICMS) pretende comunicar lo mejor de las actividades científicas realizadas a lo largo del periodo.

El ICMS es un Centro Mixto del Consejo Superior de Investigaciones Científicas (CSIC) y la Universidad de Sevilla (US), cofinanciado asimismo por la Junta de Andalucía. Está incluido en el Área de Ciencia y Tecnología de Materiales, una de las 8 en las que el CSIC estructura sus actividades científicas. Desde su creación en 1986, está integrado por personal científico de la Universidad de Sevilla y el CSIC, cuenta en la actualidad con 122 personas, 45 de las cuales son científicos permanentes.

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

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

Through this Activity Report 2017, the Institute of Materials Science of Seville (ICMS) is trying to communicate the best of its scientific activities for the period.

The ICMS is a joint Center of the Spanish Research Council (CSIC) and the University of Seville (US), also funded by the Junta de Andalucia. It is included in the "Field of Science and Technology of Materials", one of the eight within the scientific framework of the CSIC. It was founded in 1986, and integrated by scientific staff of the University of Seville and the CSIC, including today more than 122 people, 45 of which are permanent scientific staff.

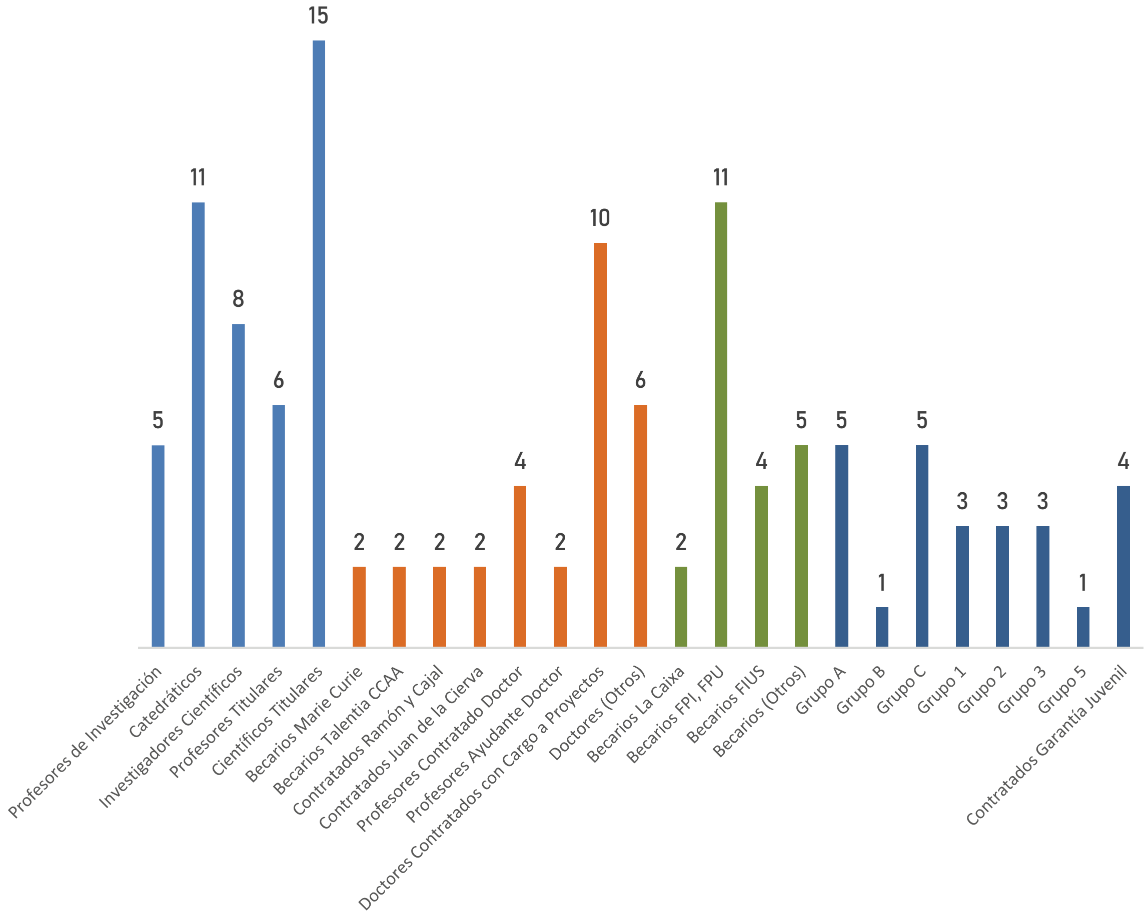
The current Strategic Plan 2014-2017 organizes our Center in 5 lines of research, which related with disciplines of solid state chemistry and physic, as well as materials engineering subjects, are intended to cover the most modern and innovative aspects of the current material science. Our basic commitment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

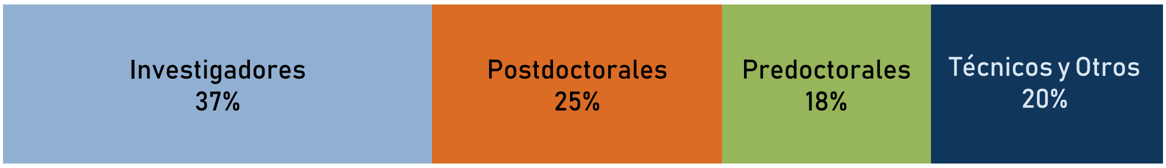
Dr. Alfonso Caballero Martínez
Director del Instituto de Ciencia de Materiales de Sevilla

Datos Estadísticos del ICMS
Statistical Data of ICMS

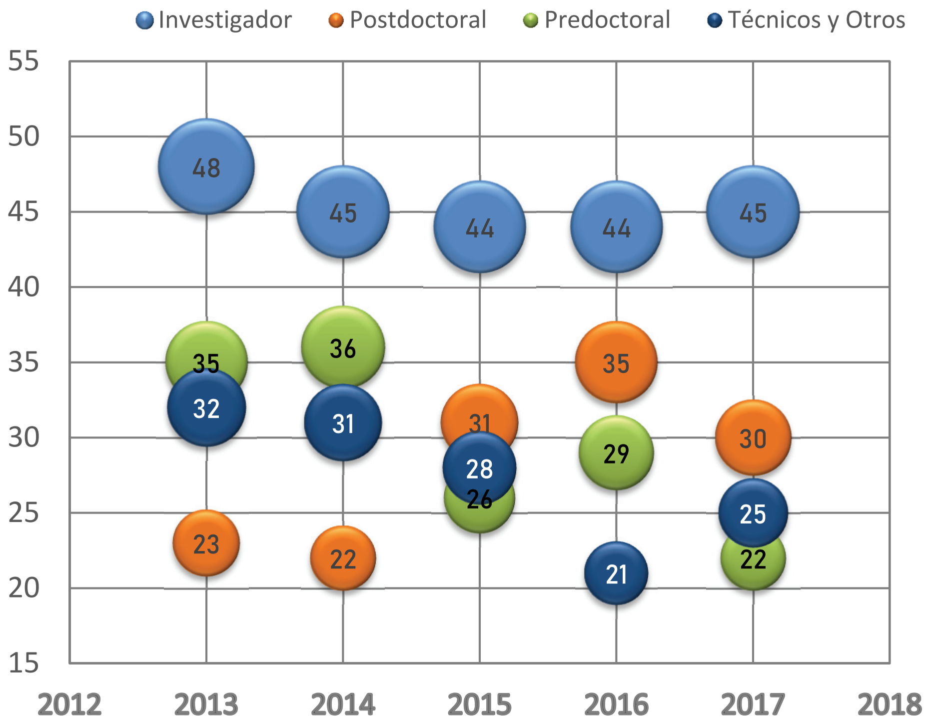
RECURSOS HUMANOS / HUMAN RESOURCES

Distribución del personal por categorías - 2017
Distribution by professional category - 2017

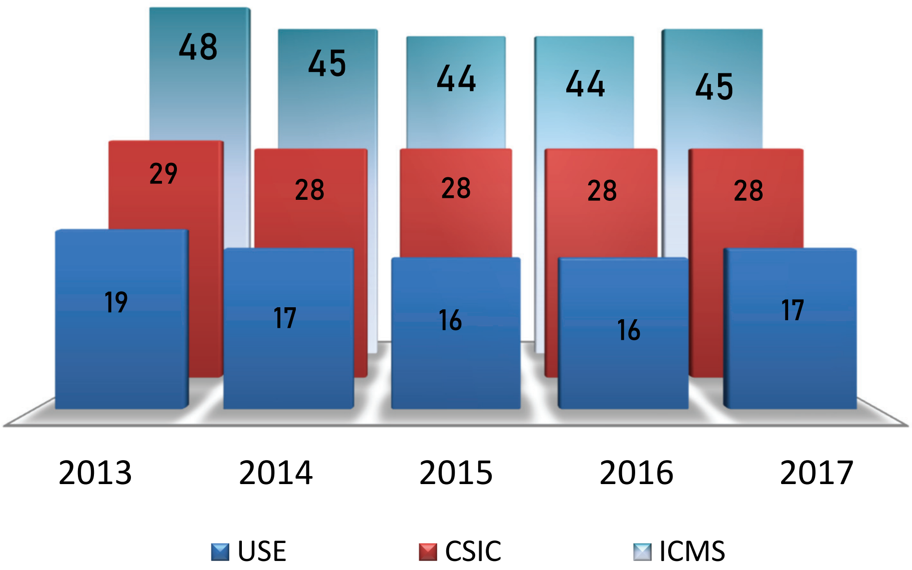




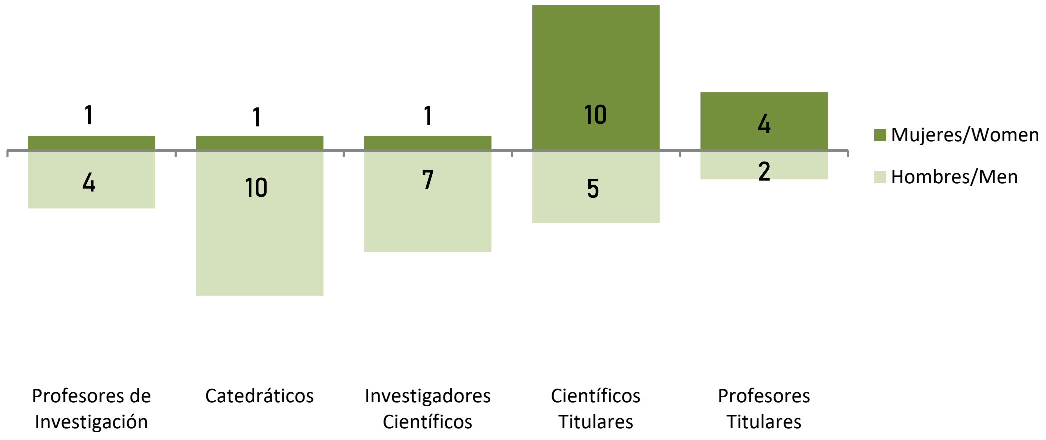
Evolución 2013-2017 del personal
Evolution of Staff

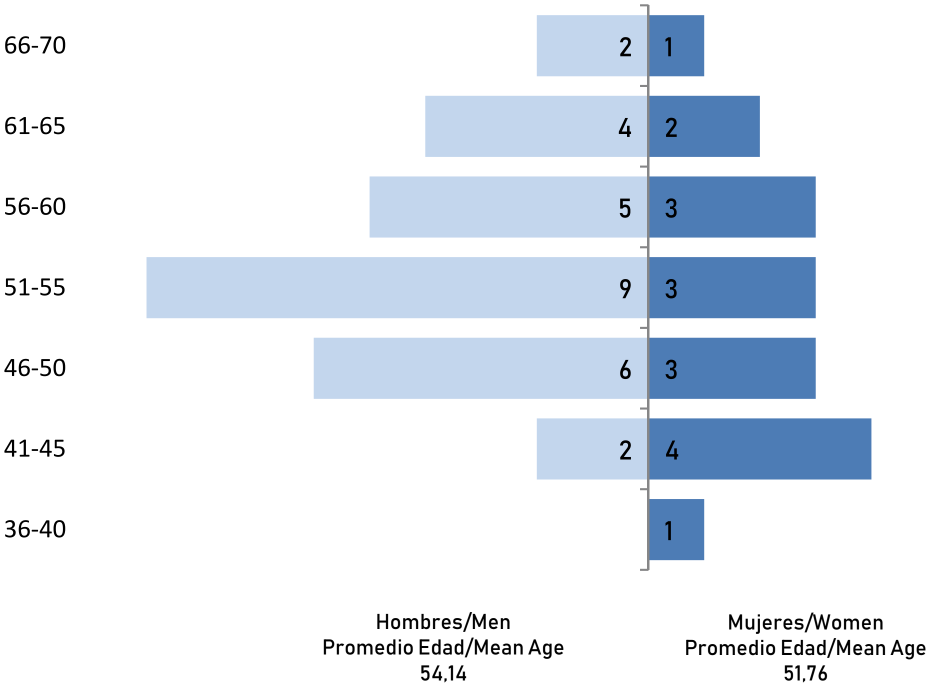


Evolución 2013-2017 del personal investigador
Evolution of Research Staff



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





Financiación / Funding

Evolución 2013-2017 de la Financiación conseguida por año (miles€)
Evolution of the funding of the ICMS

PRODUCCIÓN CIENTÍFICA / SCIENTIFIC PRODUCTION

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

REVISTA JOURNAL	ARTÍCULOS PAPERS	FACTOR DE IMPACTO IMPACT FACTOR (*)
ACS NANO	1	13,709
ADVANCED FUNCTIONAL MATERIALS	1	13,325
NANO ENERGY	1	13,120
INTERNATIONAL MATERIALS REVIEWS	1	12,703
APPLIED CATALYSIS B-ENVIRONMENTAL	6	11,698
ACS CATALYSIS	2	11,384
JOURNAL OF MATERIALS CHEMISTRY A	2	9,931
CHEMISTRY OF MATERIALS	1	9,890
ACS APPLIED MATERIALS & INTERFACES	3	8,097
APPLIED ENERGY	1	7,900
ADVANCED OPTICAL MATERIALS	5	7,430
CHEMSUSCHEM	3	7,411
NANOSCALE	2	7,233
JOURNAL OF POWER SOURCES	1	6,945
CHEMICAL ENGINEERING JOURNAL	1	6,735
ACS SUSTAINABLE CHEMISTRY & ENGINEERING	1	6,140
NANOPHOTONICS	1	6,014
JOURNAL OF MATERIALS CHEMISTRY C	2	5,976
SENSORS AND ACTUATORS B-CHEMICAL	1	5,667
JOURNAL OF CO2 UTILIZATION	2	5,503
CATALYSIS SCIENCE & TECHNOLOGY	1	5,365
JOURNAL OF EXPERIMENTAL BOTANY	1	5,354
CARBOHYDRATE POLYMERS	1	5,158
ELECTROCHIMICA ACTA	1	5,116
SOLAR ENERGY MATERIALS AND SOLAR CELLS	1	5,018
FUEL	1	4,908
RENEWABLE ENERGY	1	4,900
JOURNAL OF INDUSTRIAL AND ENGINEERING CHEMISTRY	1	4,841
ADVANCED MATERIALS INTERFACES	1	4,834
SCIENCE AND TECHNOLOGY OF ADVANCED MATERIALES	1	4,787

REVISTA JOURNAL	ARTÍCULOS PAPERS	FACTOR DE IMPACTO IMPACT FACTOR (*)
INORGANIC CHEMISTRY	1	4,700
GEOCHIMICA ET COSMOCHIMICA ACTA	1	4,690
CATALYSIS TODAY	2	4,667
BIOCONJUGATE CHEMISTRY	1	4,485
JOURNAL OF PHYSICAL CHEMISTRY C	2	4,484
APPLIED SURFACE SCIENCE	2	4,439
PARTICLE & PARTICLE SYSTEMS CHARACTERIZATION	1	4,384
SOLAR ENERGY	1	4,374
SCRIPTA MATERIALA	1	4,163
SCIENTIFIC REPORTS	3	4,122
DALTON TRANSACTIONS	2	4,099
JOURNAL OF ENVIRONMENTAL MANAGEMENT	1	4,005
LANGMUIR	1	3,789
JOURNAL OF ALLOYS AND COMPOUNDS	3	3,779
APPLIED CLAY SCIENCE	1	3,641
APPLIED PHYSICS LETTERS	1	3,495
CATALYSIS COMMUNICATIONS	1	3,463
JOURNAL OF APPLIED CRYSTALLOGRAPHY	1	3,422
NANOTECHNOLOGY	2	3,404
CRYSTENGCOMM	1	3,304
POWDER TECHNOLOGY	1	3,230
CHEMNANOMAT	1	3,173
NANOSCALE RESEARCH LETTERS	1	3,125
CERAMICS INTERNATIONAL	5	3,057
ENERGY & FUELS	1	3,024
JOURNAL OF MATERIALS SCIENCE	2	2,993
BEILSTEIN JOURNAL OF NANOTECHNOLOGY	1	2,968
JOURNAL OF THE AMERICAN CERAMIC SOCIETY	2	2,956
JOURNAL OF THE EUROPEAN CERAMIC SOCIETY	3	2,956
POLYMERS	2	2,935
SURFACE & COATINGS TECHNOLOGY	1	2,906
JOURNAL OF PHOTOCHEMISTRY AND PHOTOBIOLOGY A-CHEMISTRY	2	2,891
JOURNAL OF ENDODONTICS	1	2,886
SPECTROCHIMICA ACTA PART A-MOLECULAR AND BIOMOLECULAR SPECTROSCOPY	1	2,880
SOLID STATE IONICS	1	2,751

REVISTA **JOURNAL**	ARTÍCULOS PAPERS	FACTOR DE IMPACTO IMPACT FACTOR (*)
PLASMA PROCESSES AND POLYMERS	2	2,700
INTERNATIONAL JOURNAL OF REFRACTORY METALS & HARD MATERIALS	1	2,606
EUROPEAN JOURNAL OF INORGANIC CHEMISTRY	1	2,507
SENSORS	1	2,475
MATERIALS	1	2,467
TOPICS IN CATALYSIS	1	2,439
JOURNAL OF PHYSICS D-APPLIED PHYSICS	1	2,373
JOURNAL OF THERMAL ANALYSIS AND CALORIMETRY	2	2,209
ENGINEERING FAILURE ANALYSIS	1	2,157
CRYSTALS	1	2,144
JOURNAL OF APPLIED POLYMER SCIENCE	1	1,901
METALLURGICAL AND MATERIALS TRANSACTIONS A-PHYSICAL METALLURGICAL AND MATERIALS SCIENCE	1	1,887
APPLIED OPTICS	1	1,791
JOURNAL OF SOL-GEL SCIENCE AND TECHNOLOGY	1	1,745
MEDICINA ORAL PATOLOGIA ORAL Y CIRUGIA BOCAL	1	1,671
ANTIQUITY	1	1,656
REACTION KINETICS MECHANISMS AND CATALYSIS	1	1,515
APPLIED RHEOLOGY	1	1,442
MATERIALS RESEARCH EXPRESS	1	1,151
X-RAY SPECTROMETRY	1	1,077
JOURNAL OF NATURAL FIBERS	1	1,076
JOURNAL OF NANOPHOTONICS	1	1,060
AUSTRALIAN JOURNAL OF CHEMISTRY	1	1,059
PHYSICS OF THE SOLID STATE	1	0,925
ACTA PHYSICSA POLONICA A	1	0,857
JOURNAL OF NUCLEAR SCIENCE AND TECHNOLOGY	1	0,774
BIOMEDICAL PHYSICS & ENGINEERING EXPRESS	1	0
MOLECULAR CATALYSIS	1	0
Total	**131**	**4,767**

(*) Factor de Impacto correspondiente al año 2017
Journal Citation Reports of 2017

COMPOSICIÓN Y ESTRUCTURA

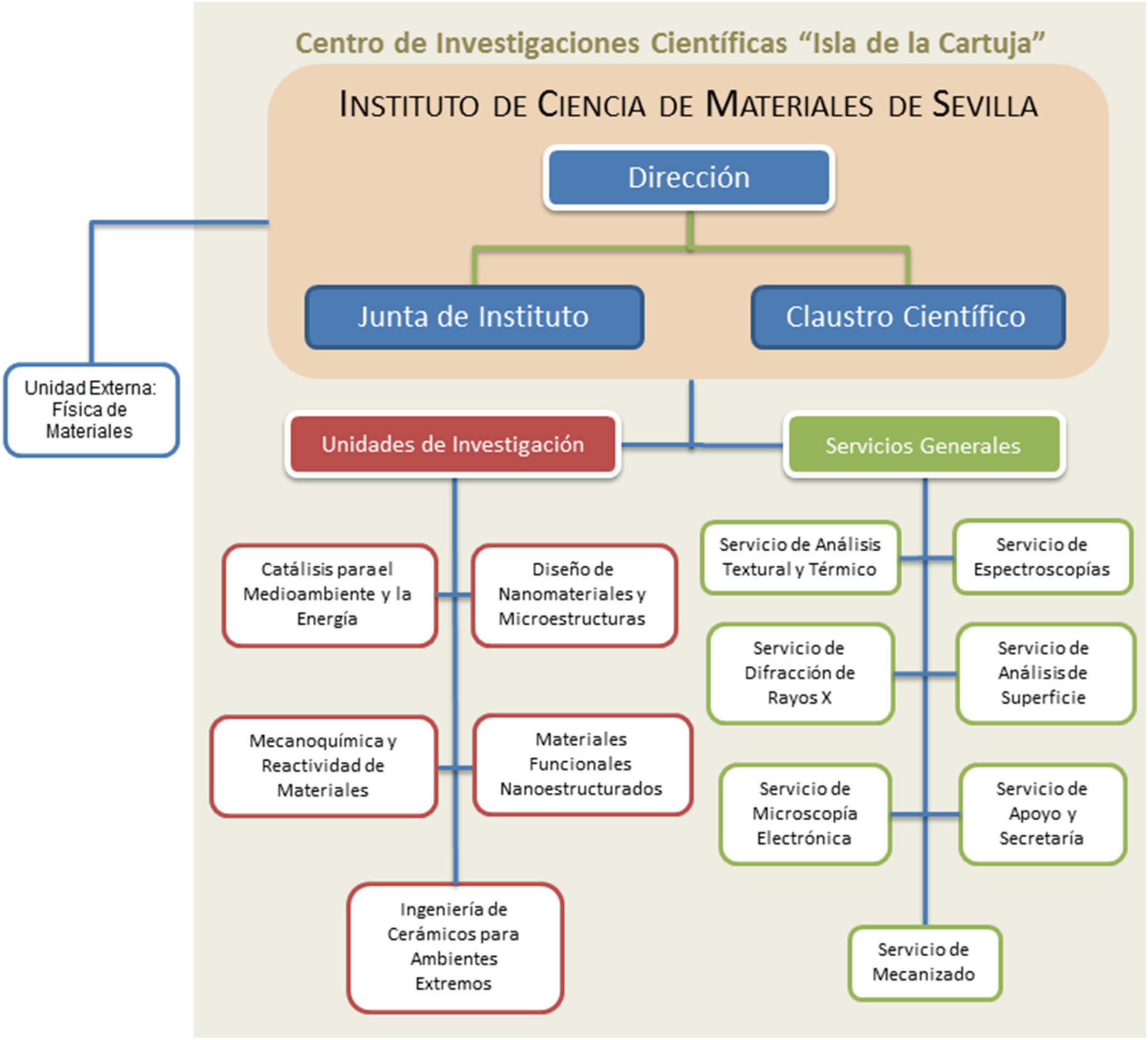
STRUCTURE AND ORGANISATION

EL INSTITUTO / THE INSTITUTE

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

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

ORGANIGRAMA / ORGANIZATION CHART



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UNIDADES DE INVESTIGACIÓN
RESEARCH UNITS

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

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Proyectos de Investigación / Research Projects

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Ministerio de Economía y Competitividad

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

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

RESUMEN / ABSTRACT

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

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

Entre los materiales que se pretenden sintetizar en el Subproyecto#2 (empleando métodos no-hidrotermales, hidrotermales y sol-gel) se contemplan: óxidos binarios (TiO2, ZnO, ZnO2, Fe_2O_3, WO_3, Bi_2O_3, Ta_2O_5 , La_2O_3), óxidos binarios acoplados (TiO_2-WO_3, TiO_2-ZnO, TiO_2-ZnO_2; TiO_2-Ta_2O_5, TiO_2-La_2O_3, ZnO-Fe_2O_3 y ZnO_2-Fe_2O_3), óxidos ternarios (Bi_2WO_6, Bi_2WO_6-ZnO, Bi_2WO_6-ZnO_2, Bi_2WO_6-Fe_2O_3, $Bi_2Ti_2O_7$, $ZnWO_4$, $La_2Ti_2O_7$), contemplando la foto-deposición de metales (Pt, Ag, Au) en los sistemas que muestren una actividad fotocatalítica considerable (Semiconductores/Metal).

Los mejores sistemas evaluados se remitirán al grupo del Subproyecto#1 para el estudio de la viabilidad fotocatalítica en la eliminación de NOx,COVs, CO, CO_2 y SO_2 presentes en emisiones gaseosas.

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

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

Among the materials that are to be synthesized in the Subproject # 2 (by using non-hydrothermal, hydrothermal and sol-gel methods) are contemplated: binary oxides (TiO_2, ZnO, WO_3, Fe_2O_3, Bi_2O_3, Ta_2O_5, La_2O_3), coupled binary oxides (TiO_2-WO_3, TiO_2-ZnO, TiO_2-ZnO_2, TiO_2-Ta_2O_5, TiO_2-La_2O_3, ZnO-Fe_2O_3 y ZnO_2-Fe_2O_3), ternary oxides (Bi_2WO_6, Bi_2WO_6-ZnO, Bi_2WO_6-ZnO_2, Bi_2WO_6-Fe_2O_3, $Bi_2Ti_2O_7$, $ZnWO_4$,$La_2Ti_2O_7$) studying the photo-deposition of single metals (Pt, Ag, Au) on those prepared systems that exhibit significant photocatalytic activity (Semiconductor /Metal). Best evaluated systems will be forwarded to the Subprojet 1 for the feasibility study on the photocatalytic removal of NOx, VOCs, CO, CO_2 and SO_2 present in gaseous emissions.

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

Código/Code:	ENE2015-66975-C3-2-R
Periodo/Period:	01-01-2016 / 31-12-2018
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	302.500 €
Investigador responsable/Research head:	José Antonio Odriozola Gordón

Componentes/Research group: Anna Dimitrova Penkova, María Isabel Domínguez Leal, Francisca Romero Sarria

RESUMEN / ABSTRACT

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

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

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

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

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

The dependence of our current energy system on fossil fuels and their harmful effects on the environment are strengthen the development of renewable energy sources. This is the

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

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

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

RESUMEN / ABSTRACT

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

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

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

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

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

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

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

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

Ibáñez, Rosa María Pereñiguez Rodríguez, Alberto Rodríguez Gómez

RESUMEN / ABSTRACT

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

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

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

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

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

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

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

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

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

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

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

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

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

RESUMEN / ABSTRACT

Hoy en día no existen dudas acerca de que la concentración de gases de efecto invernadero, en particular la de CO_2, está aumentando de manera considerable en la atmósfera terrestre. Para evitar este aumento continuado se debe aumentar la eficiencia en la producción de energía, disminuir la intensidad del uso de las fuentes fósiles y, finalmente, potenciar la captura y secuestro del CO_2. Todo esto debe conseguirse manteniendo el crecimiento económico y la calidad de vida. En consecuencia, si tenemos en cuenta el desarrollo de las naciones menos industrializadas y el intensivo consumo energético necesario para aumentar su nivel de vida, la captura y secuestro de CO_2 parece la alternativa más favorable.

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

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

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

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

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

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

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

OTROS PROYECTOS / OTHER PROJECTS

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

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

Ensayos de laboratorio y elaboración de informe acerca de la inflamabilidad de neumáticos usados

Periodo/Period:	02-12-2016 / 02-03-2017
Oganismo Financiador/Financial source:	Universidad de Sevilla
Código/Code:	020165180011
Investigador responsable/Research head:	Alfonso Caballero Martínez

UNIDAD ASOCIADA / ASSOCIATED UNIT

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

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

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

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

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

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

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

The main research lines of working are:

"Photocatalysis for environmental processes"
"Spectroscopical-electrochemistry applied to environment"
"Natural processes for the treatment of waste water"

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

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

Gold promoted Cu/ZnO/Al2O3 catalysts prepared from hydrotalcite precursors: Advanced materials for the WGS reaction

Santos, JL; Reina, TR; Ivanova, S; Centeno, MA; Odriozola, JA
Applied Catalysis B: Enviromental, **201** (2017) 310-317
Enero, 2017 | **DOI:** 10.1016/j.apcatb.2016.08.017

Outstanding catalysts for the water was shift reaction are reported in this work. The combination of gold nanoparticles with Cu/ZnO/Al2O3 prepared from hydrotalcite-like precursors leads to very promising systems for pure hydrogen production. Full CO conversion is reached at temperatures as low as 180 degrees C. The key point seems to be the cooperation of Au and Cu and the optimal metal-oxide contact derived from the synthesis method. The high activity of gold for low temperature CO oxidation and the suitability of copper for the WGS results in a perfect synergy. Moreover the materials developed in this work present good stability and tolerance towards start/stop cycles an indispensable requisite for a realistic application in an integrated hydrogen fuel processor.

NO photooxidation with TiO2 photocatalysts modified with gold and platinum

Rodriguez, MJH; Melian, EP; Santiago, DG; Diaz, OG; Navio, JA; Rodriguez, JMD
Applied Catalysis B: Environmental, **205** (2017) 148-157
Mayo, 2017 | **DOI:** 10.1016/j.apcatb.2016.12.006

In this study, a comparative analysis is made of TiO2 modified with Pt or Au in NO photoxidation under different radiation and humidity conditions. The metals were deposited on the TiO2 surface using two methods, photodeposition and chemical reduction. All catalysts were supported on borosilicate 3.3 plates using a dip-coating technique. These modified photocatalysts were characterized by X-ray diffraction analysis (XRD), UV-vis diffuse reflectance spectra (DRS), Brunauer-Emmett-Teller measurements (BET), transmission electron microscopy (TEM) and X-ray photoelectron spectrum analysis (XPS). It was found from the XPS results that Pt and oxidized Pt species coexist on the samples obtained by photodeposition and chemical reduction. In the case of Au, though other oxidation states were also detected the dominant oxidation state for both catalysts is Au. TEM results showed most Au-C particles are below 5 nm, whereas for Au-P the nanoparticles are slightly bigger. With UV irradiation, the Pt modified catalysts do not show any significant improvement in NO photocatalytic oxidation in comparison with the unmodified P25. For Au, both modified photocatalysts (Au-P and Au-C) exceed the photocatalytic efficiency of the unmodified P25, with Au-C giving slightly better results. The incorporation of metals on the TiO2 increases its activity in the visible region.

Structuring Pt/CeO_2/Al_2O_3 WGS catalyst: Introduction of buffer layer

Gonzalez-Castano, M; Ivanova, S; Laguna, OH; Martinez, LM; Centeno, MA; Odriozola, JA
Applied Catalysis B: Environmental, **200** (2017) 420-427
Enero, 2017 | **DOI:** 10.1016/j.apcatb.2016.07.039

This work is devoted to the development of novel structured catalytic system for WGS reaction. The new concept is related to the presence of a pre-catalytic "buffer" layer formed by WGS-inert oxide, i.e. not involved in CO conversion, but able to increase the number of participating sites in water dissociation step during the reaction. The performance of the proposed systems appears to depend strongly on the stream composition, being its effect beneficial in highly reducing atmospheres making it ideal for cleanup application. An increment of the partial kinetic order for water species is observed and reveals the key role of the water activation for superior catalytic behavior.

Study of the E. coli elimination from urban wastewater over photocatalysts based on metallized TiO_2

Murcia, JJ; Avila-Martinez, EG; Rojas, H; Navio, JA; Hidalgo, MC
Applied Catalysis B: Environmental, **200** (2017) 469-476
Enero, 2017 | **DOI:** 10.1016/j.apcatb.2016.07.045

In this study, a series of photocatalysts based on TiO_2 was tested in the elimination of Escherichia coli (E. coli) from urban wastewater. Firstly, TiO_2 obtained by sol-gel method was modified by sulfation, and then gold or platinum nanoparticles were photodeposited on sulfated titania surface. Platinized samples were also prepared with different Pt content of 0.5 and 2 wt.%. The samples thus obtained were extensively characterized and it was found that sulfation considerably increases the S-BET value of TiO_2 and promotes the anatase phase formation; it was also found that 0.5 wt.% Pt-TiO_2 sample presents the lowest noble metal particle size and the best particle dispersion. All the photocatalysts synthesized have shown bactericidal effect and the results obtained by using bare and metalized TiO_2 were considerably better than the results obtained with the commercial TiO_2 P25 Evonic. Different light intensities were also evaluated in the photocatalytic tests and it was found that 120 W/m(2) leads to obtain the highest E. coli elimination from wastewater samples; however no total elimination of E. coli or other species of bacteria was achieved even after 5 h of photocatalytic treatment without catalyst. Total elimination of the E. coli was achieved after 3 h of photocatalytic reaction by using 120 Wim(2) of light intensity and 2 wt.% Pt-TiO_2 as photocatalyst; no bacterial regrowth was observed even after 72 h.

Cobalt Carbide Identified as Catalytic Site for the Dehydrogenation of Ethanol to Acetaldehyde

A. Rodríguez-Gómez; J.P. Holgado; A. Caballero
ACS Catalysis, **7** (2017) 5243-5247
Julio, 2017 | **DOI:** 10.1021/acscatal.7b01348

Two cobalt catalysts, Co/SBA-15 and Co/SiO_2, have been studied in steam reforming of ethanol (SRE). Besides the steam reforming products, ethoxide dehydrogenation to acetaldehyde is observed as one of the main reactions. Although by hydrogen treatment cobalt is reduced to the metallic state, under SRE conditions, a phase appears that has been identified as cobalt carbide and correlates with acetaldehyde production. These findings provide insights about the catalytic sites, for SRE, in cobalt catalysts. Comparison with previous results shows that these conclusions are not translatable to other cobalt catalysts, stressing the importance of the support on the catalytic behavior of cobalt.

Monitoring the Reaction Mechanism in Model Biogas Reforming by InSitu Transient and Steady-State DRIFTS Measurements

Bobadilla, LF; Garcilaso, V; Centeno, MA; Odriozola, JA
Chemsuschem, **10** (2017) 1193-1201
Marzo, 2017 | **DOI:** 10.1002/cssc.201601379

In this work, the reforming of model biogas was investigated on a Rh/$MgAl_2O_4$ catalyst. In situ transient and steady-state diffuse reflectance infrared Fourier transform spectroscopy (DRIFTS) measurements were used to gain insight into the reaction mechanism involved in the activation of CH_4 and CO_2. It was found that the reaction proceeds through of an initial pathway in which methane and CO_2 are both dissociated on Rh metallic sites and additionally a bifunctional mechanism in which methane is activated on Rh sites and CO_2 is activated on the basic sites of the support surface via a formate intermediate by H-assisted CO_2 decomposition. Moreover, this plausible mechanism is able to explain why the observed apparent activation energy of CO_2 is much lower than that of CH_4. Our results suggest that CO_2 dissociation facilitates CH_4 activation, because the oxygen-adsorbed species formed in the decomposition of CO_2 are capable of reacting with the CH_x species derived from methane decomposition.

Deep insight into Zr/Fe combination for successful Pt/CeO_2/Al_2O_3 WGS catalyst doping

Gonzalez-Castano, M; Ivanova, S; Ioannides, T; Centeno, MA; Odriozola, JA
Catalysis Science & Technology, **7** (2017) 1556-1564
Abril, 2017 | **DOI:** 10.1039/c6cy02551j

Efficient promotion of the $Pt/CeO_2/Al_2O_3$ catalytic system was achieved by the addition of two different ceria promoters, Zr and Fe. From the exhaustive data analysis, the key features for enhanced catalytic performance and the roles of each doping metal are established. The combination of both doping agents manifests a synergistic effect reflected in noteworthy improvements in H_2 reducibility. In addition, the catalyst's doping influences its chemisorptive properties, which is reflected in an increase of the easiness of carbonaceous species desorption, thus leading to superior catalyst resistance toward deactivation.

Gold catalysts screening in base-free aerobic oxidation of glucose to gluconic acid

Megias-Sayago, C.; Ivanova, S.; Lopez-Cartes, C.; Centeno, M.A.; Odriozola, J.A.
Catalysis Today, **279** (2017) 148-154
Enero, 2017 | DOI: 10.1016/j.cattod.2016.06.046

Base-free aerobic oxidation of glucose in presence of Au/Al_2O_3, Au/CeO_2, Au/CeO_2(20 wt%)/Al_2O3, Au/CeO_2(25 wt%)/ZrO_2 and Au/CeO_2(50 wt%)/ZrO_2 catalysts using molecular oxygen at atmospheric pressure is studied. Within the whole series high conversion and selectivity to gluconic acid are observed after 18 h of reaction at 120 degrees C. The activity and especially the selectivity changes are related to the support nature in a way that the higher the Lewis acidity of the support the lower the selectivity to gluconic acid and the higher the production of lactic acid. The highest yield to gluconic acid is obtained over Au/Al_2O_3 for which the influence of the reaction time, temperature and stirring rate are further evaluated and discussed.

High UV-photocatalytic activity of ZnO and Ag/ZnO synthesized by a facile method

C. Jaramillo-Páez; J.A. Navío; M.C. Hidalgo; M. Macías
Catalysis Today, **284** (2017) 121-128
Abril, 2017 | **DOI:** 10.1016/j.cattod.2016.11.021

ZnO nanoparticles have been successfully synthesized by a facile precipitation procedure by mixing aqueous solutions of Zn(II) acetate and dissolved Na_2CO_3 at pH ca. 7.0 without template addition. We have investigated the effect of annealing temperature in the final surface and structural properties. Photocatalytic studies were performed using two selected substrates, Methyl Orange and Phenol, both as single model substrates and in mixtures of them.
It has been stated that calcination treatments lead to a significant improvement in the photocatalytic properties of the studied samples, even better than TiO_2(P25). As expected, the addition of Ag^+ during the photocatalytic degradation of MO increases the reaction rate of the degradation of MO, giving a resultant Ag/ZnO photocatalyst which, after recovery, can be reused at least 18 times for the MO degradation tests, being even more photoactive than ZnO.

Redox and Catalytic Properties of Promoted NiO Catalysts for the Oxidative Dehydrogenation of Ethane

Delgado, D; Solsona, B; Ykrelef, A; Rodriguez-Gomez, A; Caballero, A; Rodriguez-Aguado, E; Rodriguez-Castellon, E; Nieto, JML
Journal of Physical Chemistry C, **121** (2017) 25132-25142
Noviembre, 2017 | **DOI:** 10.1021/acs.jpcc.7b07066

NiO and metal-promoted NiO catalysts (M-NiO, with a M/(M+Ni) atomic ratio of 0.08, with M = Nb, Sn, or La) have been prepared, tested in the oxidative dehydrogenation (ODH) of ethane, and characterized by means of XRD, TPR, HRTEM, Raman, XPS, and in situ XAS (using H-2/He, air or C_2H_6/He mixtures). The selectivity to ethylene during the ODH of ethane decreases according to the following trend: Nb NiO Sn NiO > La NiO > NiO, whereas the catalyst reducibility (determined by both TPR and XAS using H-2/He mixtures) shows the opposite trend. However, different reducibility and catalytic behavior in the absence of oxygen (ethane/He mixtures) have been observed, especially when comparing Nb- and Sn-promoted NiO samples. These differences can be ascribed mainly to a different phase distribution of the promoter. The results presented here are discussed in terms of the nature of active and selective sites for ODH of ethane in selective and unselective catalysts, but also the role of promoters and the importance of their phase distribution.

Outstanding visible photocatalytic activity of a new mixed bismuth titanatate material

Zambrano, P; Sayagues, MJ; Navio, JA; Hidalgo, MC
Applied Surface Science, **394** (2017) 16-24
Enero, 2017 | DOI: 10.1016/j.apsusc.2016.10.042

In this work, a new photocatalyst based on bismuth titanates with outstanding visible photocatalytic activity was prepared by a facile hydrothermal method. The synthesised material showed visible activity as high as UV activity of commercial TiO_2 P25 under the same experimental conditions for phenol degradation. A wide characterisation of the photocatalyst was performed. The material was composed of three phases; majority of $Bi_{2}0TiO_{32}$ closely interconnected to $Bi_4Ti_3O_{12}$ and amorphous TiO_2. The high visible activity showed by this material could be ascribed to a combination of several features; i.e. low band gap energy value (2.1 eV), a structure allowing a good separation path for visible photogenerated electron-holes pairs and a relatively high surface area. This photocatalyst appeared as a promising material for solar and visible applications of photocatalysis.

Preferential oxidation of CO on a La-Co-Ru perovskite-type oxide catalyst

Pereniguez, R; Caballero, A; Ferri, D
Catalysis Communication, **92** (2017) 75-79
Marzo, 2017 | **DOI:** 10.1016/j.catcom.2016.12.020

A Ru-containing perovskite-type oxide La(Co,Ru)O_3 of nominal composition $LaCo_{0.8}Ru_{0.2}O_3$ was prepared by ultrasonic spray combustion and tested for the preferential oxidation of CO (PROX). EXAFS indicated that Ru adopted the coordination environment of Co in $LaCoO_3$ while Co was present as $LaCoO_3$ and Co_3O_4. PROX activity was replaced by CO hydrogenation activity above 250 °C. Short oxidation at 500 °C between temperature programmed reaction ramps did not restore the initial $La(Co,Ru)O_3$ structure but generated a catalyst with improved PROX activity compared to the initial $La(Co,Ru)O_3$. Under reductive PROX conditions the material experienced structural changes that improved its overall catalytic activity only if the catalyst was oxidized after each temperature programmed ramp.

Identification of Outer and Inner Nickel Particles in a Mesoporous Support: How the Channels Modify the Reducibility of Ni/SBA-15 Catalysts

Rodriguez-Gomez, A; Caballero, A
Chemnanomat, **3** (2017) 94-97
Febrero, 2017 | **DOI:** 10.1002/cnma.201600297

Two different nickel supported on SBA-15 catalytic systems have been prepared by means of impregnation (Ni/SBA-15-ImU) and deposition-precipitation (Ni/SBA-15-DP) methodologies. Upon calcination, Ni/SBA-15DP presents a well-developed nickel phyllosilicate phase, which after reduction gives rise to a dispersed and homogeneous metallic phase, mainly located inside the 5 nm in diameter mesoporous structure of the support. On the contrary, as evidenced by XRD and a double temperature programmed reduction (TPR) peak, the Ni/SBA-15-ImU catalyst presents two different NiO phases, which after reduction in hydrogen generate nickel particles in a wide range of sizes. In situ XAS and XPS have unambiguously showed that the distinct TPR profiles obtained for each system are related with particles located in and out the mesoporous structure of the SBA-15 channels. The particles inside the porous are more difficult to reduce, clearly showing a kind of confinement effect of the SBA-15 mesostructure, modifying the reducibility of the NiO phase.

Structural and catalytic properties of Au/MgO-type catalysts prepared in aqueous or methanol phase: application in the CO oxidation reaction

Hernandez, Willinton Y.; Alic, Funda; Navarro-Jaen, Sara; Centeno, Miguel A.; Vermeir, Pieter; Van der Voort, Pascal; Verberckmoes, An
Journal of Materials Science, **52** (2017) 4727-4741
Abril, 2017 | **DOI:** 10.1007/s10853-016-0715-9

Au/MgO and Au/Mg(OH)(2)-type catalysts for CO oxidation reaction were prepared by using two different synthesis methods in presence of either an aqueous or methanol phase. The influence of the porous and morphological properties of the starting magnesium oxide supports was analyzed and correlated with the catalytic performances of the final gold-supported catalysts. It was found that the deposition of gold in the presence of methanol as a solvent avoids the total rehydration of the MgO support and maintains the textural and morphological properties of the starting oxides. The support synthesized by a surfactant-assisted hydrothermal route, having a combined meso-macroporous structure (i.e., MgO-P) showed a positive influence on the CO oxidation reaction as it favored the dispersion of gold and the surface-to-gas phase interaction during the catalytic process.

Mixed alpha-Fe_2O_3/Bi_2WO_6 oxides for photoassisted hetero-Fenton degradation of Methyl Orange and Phenol

Jaramillo-Paez, C; Navio, JA; Hidalgo, MC; Bouziani, A; El Azzouzi, M
Journal of Photochemistry and Photobiology A-Chemistry, **332** (2017) 521-533
Enero, 2017 | **DOI:** 10.1016/j.jphotochem.2016.09.031

Mixed oxides, alpha-Fe_2O_3/Bi_2WO_6, were prepared using a mechanical mixing procedure by adding to the Bi_2WO_6 previously obtained by hydrothermal method the corresponding amount of a prepared alpha-Fe_2O_3, the latter obtained by thermal decomposition of Fe(NO_3)center dot 9H(2)O. The physicochemical surface, structural, morphological characteristics and optical properties of the samples, single and mixed, were determined by BET, XRD, FE-SEM, XPS and UV-vis diffuse reflectance spectroscopy. UV-vis diffuse reflectance spectra showed that incorporating a 5%wt. of alpha-Fe_2O_3 to the corresponding amount of Bi_2WO_6 sample broadened the visible light absorption of Bi_2WO_6 as expected. The photocatalytic activity, of single and mixed catalysts, to degrade a selected dye such as Methyl Orange (MO) as well as the transparent substrate Phenol (Ph) was studied, in aqueous medium (pH 5.5) under UV and sun-like illumination conditions in the absence and presence of H_2O_2. In the present study the use of a alpha-Fe_2O_3/Bi_2WO_6/H_2O_2 system demonstrate much higher photocatalytic efficiency to degrade both MO and Ph than pristine Bi_2WO_6 or alpha-Fe_2O_3, single or mixed. Using the system alpha-Fe_2O_3/Bi_2WO_6/H_2O_2, around 85% of MO was degraded in 60 min under sun-like illumination whereas 100% was degraded in 60 min under UV-illumination. However, just around 30% of Ph was degraded in 120 min in the alpha-Fe_2O_3/Bi_2WO_6/H_2O_2 system under sun-like illumination whereas around a 95% was degraded in 90 min under UV-illumination. Under UV illumination, the generation of hydroxyl radicals is favorable; whereas under sun-like illumination, only the small fraction of the UV can produces the center dot OH. Under illumination, the H_2O_2 could react with photoinduced electrons from the photocatalysts leading to the production of hydroxyl radicals (center dot OH).

Photochemical methane partial oxidation to methanol assisted by H_2O_2

Lopez-Martin, Angeles; Caballero, Alfonso; Colon, Gerardo
Journal of Photochemistry and Photobiology A: Chemistry, **349** (2017) 216-223
Diciembre, 2017 | **DOI:** 10.1016/j.jphotochem.2017.09.039

The photochemical conversion of methane into methanol from H_2O_2 aqueous solution as well as the effect of the addition mode were studied. Direct addition of different amounts H_2O_2 leads to increasing methanol production at the first stage of the reaction. The excess of H_2O_2 would lead to the reactive oxygen species scavenging and the subsequent O_2 production. It was also corroborated that extra hydroxyl radicals in the aqueous medium do not improve the formation of methanol but a noticeable increase in the formation of HCOOH with respect to methanol was evidenced. In contrast, dosing addition at relatively low rates leads to constant methane consumption towards methanol. Methanol formation would be in this case in equilibrium with further oxidation to HCOOH or CO_2. This suggests that only a controlled constant availability of HO's at low concentration can enhance the performance of methanol generation in the photochemical process.

Fischer-Tropsch Synthesis Over Zr-Promoted Co/gamma-Al_2O_3 Catalysts

Barrientos, J; Garcilaso, V; Venezia, B; Aho, A; Odriozola, JA; Boutonnet, M; Jaras, S
Topics in Catalysis, **60** (2017) 1285-1298
Noviembre, 2017 | **DOI:** 10.1007/s11244-017-0813-1

Two Zr-modified alumina supports were synthetized containing the same amount of Zr but a different distribution of this modifier over the alumina surface. These supports, together with the unmodified alumina carrier, were used to prepare three cobalt-based catalysts which were characterized and tested under relevant Fischer-Tropsch conditions. The three catalysts presented very similar porosity and cobalt dispersion. The addition of Zr nor its distribution enhanced the catalyst reducibility. The catalyst activity was superior when using a carrier consisting of large ZrO_2 islands over the alumina surface. The use of a carrier with a homogeneous Zr distribution had however, a detrimental effect. Moreover, a faster initial deactivation rate was observed for the Zr-promoted catalysts, fact that may explain this contradictory effect of Zr on activity. Finally, the addition of Zr showed a clear enhancement of the selectivity to long chain hydrocarbons and ethylene, especially when Zr was well dispersed.

Analysis of Ni species formed on zeolites, mesoporous silica and alumina supports and their catalytic behavior in the dry reforming of methane

Drobna, Helena; Kout, Martin; Soltysek, Agnieszka; Gonzalez-Delacruz, Victor M.; Caballero, Alfonso; Capek, Libor
Reaction Kinetics Mechanisms and Catalysis, **121** (2017) 255-274
Junio, 2017 | **DOI:** 10.1007/s11144-017-1149-3

The presented investigation is focused on the analysis of Ni species formed on microporous (zeolites MFI and FAU) and mesoporous materials (Al-MCM-41 and SBA-15) and alumina supports and their catalytic behavior in the dry reforming of methane. The paper lays emphasis on the relationship between the catalytic behavior of Ni-based catalysts and their textural/structural properties. Ni-based catalysts were prepared by wet impregnation (11 wt% of Ni) followed by calcination in air and reduction in hydrogen. The properties of Ni-based catalysts were also compared prior and after the catalytic tests. The critical role was played by the high value of the specific surface area and the high strength of the interaction between the Ni species and the support, which both determined the high dispersion and stability of metal Ni-0 particles. Ni-Al-MCM-41 and Ni-SBA-15 showed the values of the conversion of CO_2 and CH_4 above 90% (stable during 12 h). Slightly lower values of the conversion of CO_2 and CH_4 were observed over Ni-Al_2O_3 (also stable during 12 h). In contrast to these materials, Ni-MFI and Ni-FAU exhibited the worse metallic Ni-0 particles dispersion and very bad catalytic behavior.

Synthesis and characterization of Rh/MnO_2-CeO_2/Al_2O_3 catalysts for CO-PrOx reaction

Martinez, TLM; Laguna, OH; Lopez-Cartes, C; Centeno, MA
Molecular Catalysis, **440** (2017) 9-18
Octubre, 2017 | **DOI:** 10.1016/j.mcat.2017.06.018

Rh/MnO_2-CeO_2/Al_2O_3 catalysts with different manganese-to-ceria ratios have been synthesized, characterized and tested in CO-PrOx reaction. The physicochemical properties of the solids were studied by XRD, Raman spectroscopy, BET surface area, H-2-TPR, TGA-DTG and TEM. The differences observed in the textural, structural and redox properties were related to the Mn-to-ceria ratio of the samples. The segregation of Mn species was observed at high Mn-to-Ce ratios. In opposite way, MnO_2-CeO_2 solid solutions were obtained at low Mn to Ce ones. In this last case, the physicochemical properties of the solids were favored by the intimate Rh-Ce-Mn contact. The effect of the Mn-Ce presence on Rh catalysts which promotes the catalytic behavior towards selective CO oxidation was observed to be better at low temperatures. At higher temperatures, Mn species promote the Reverse Water Gas Shift reaction, whilst ceria promotes the H-2 oxidation in the whole range of working temperatures.

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

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

COMUNICACIONES / COMMUNICATIONS

10th International Conference on Chemical Kinetics | ICCK2017
21 – 25 mayo [Chicago, Estados Unidos]

Kinetics and mechanism of catalytic coke gasification by air/CO_2
M.A.N.D.A. Lemos; S.A.C. Carabineiro; L.F. Bobadilla; M.A. Centeno; E. Santos; F. Lemos; L.S. Lobo

5th European Conference on Environmental Applications of Advanced Oxidation Processes
25 – 29 junio [Praga, República Checa]

ZnO and Pt-ZnO photocatalysts: Characterization and Photo-Catalytic Activity Assessing by mean of Three Substrates
C.A. Jaramillo-Páez; J.A. Navío; M.C. Hidalgo; M. Macias
Poster

13th European Congress on Catalysis | EUROPACAT 2017

27 – 31 agosto [Florencia, Italia]

Operando near ambient pressure XPS (NAP-XPS) study of a model Pt-Cu catalyst for the PROX reaction
R. Castillo; F. Romero-Sarria; J.A. Odriozola
Comunicación Oral

Time-resolved operando DRIFTS/MS study of CO_2 reforming of methane over an efficient Ni-Ru supported catalyst
L.F. Bobadilla; A. Álvarez; V. Garcilaso; M.A. Centeno; J.A. Odriozola
Comunicación Oral

Highly active and selective Ru nanoparticles as catalysts for the CO and CO_2 methanation
S. Navarro; O.H. Laguna; M.A. Centeno; J.A. Odriozola
Poster

Kinetic study of the dry reforming of methane using monolithic Ni-Ru structured catalysts
I. Reyero; V. Garcilaso; A. Álvarez; L.M. Martínez; F. Bimbela; M.A. Centeno; L.M. Gandía
Poster

Water gas shift reaction on ionic conductor modified platinum catalyst
N. García-Moncada; L. Jurado; A. Beretta; F. Romero-Sarria; G. Groppi; J.A. Odriozola
Poster

Assessing the Photo-catalytic properties of Bi_2WO_6 catalysts synthesized at different pHs values
C.A. Jaramillo-Páez; J.A. Navío; M.C. Hidalgo; M. Macias
Poster

Facile synthesis of ZnWO4. Characterization and Photo-catalytic properties
C.A. Jaramillo-Páez; J.A. Navío; M.C. Hidalgo; M. Macias
Poster

The Third International Congress of Nanoscience and Nanotechnology ICNN'2017

28 agosto – 1 septiembre [Quito, Ecuador]

Gold Nanoparticles for Energy Production and Biomass Valorization
S. Ivanova
Conferencia Invitada

8th World Congress on Oxidation Catalysis WCOC 2017 and XII European Wordkshop Meeting on Innovation in Selective Oxidation Catalysis | ISO'17

3 – 8 septiembre [Cracovia, Polonia]

Which is the support contribution to gold catalyst activity for FDCA production?
C. Megías-Sayago; A. Lolli; S. Ivanova; S. Albonetti; F. Cavani; J.A. Odriozola
Comunicación Oral

CONGRESOS Y REUNIONES NACIONALES / NATIONAL CONGRESSES AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

Avances de la Nanotecnología en Biomedicina y Medio Ambiente en la Región Sur-Suroeste

29 mayo – 2 junio [Villahermosa, México]

Nanomateriales como fotocatalizadores para aplicaciones ambientales
M.C. Hidalgo
Conferencia Invitada

XXXVI Reunión Bienal de la Real Sociedad Española de Química

25 – 29 junio [Sitges, España]

New insights on the reaction mechanism of model biogas reforming by operando DRIFTS measurements
Luis F. Bobadilla; Victoria Garcilaso; Miguel A. Centeno; José A. Odriozola
Comunicación oral

Reunión de la Sociedad Española de Catálisis | SECAT'17

26 – 28 junio [Oviedo, España]

Cinética de hidrogenación de ciclohexeno en fase líquida usando catalizadores de Pd-Al/Carbón biomórfico
F. Cazaña; N. Latorre; E. Romeo; C. Royo; M.A. Centeno; A. Monzón
Comunicación oral

Influence of support to gold catalyst activity for teh production of 2,5-furandicarboxilic acid
C. Mejías-Sayago; A. Lolli; S. Ivanova; S. Albonetti; F. Cavani; J.A. Odriozola
Comunicación oral

Multicomponent catalytic system: versatile ionic conductor for diverse type WGS catalysts
N. García-Moncada; M. González Castaño; S. Ivanova; M.A. Centeno; F. Romero Sarria; J.A. Odriozola
Comunicación oral

Reformado catalítico de biogás usando catalizadores Rh/$MgO-Al_2O_3$
V. Garcilaso; M. A. Centeno; O.H. Laguna; J.A. Odriozola
Comunicación oral

Actividad catalítica de nanopartículas de TiO_2 facetado en la condensación de compuestos oxigenados presentes en fracciones acuosas derivadas de la biomasa
A.Fernández-Arroyo; M.A. Lara; M.E. Domine; M.J. Sayagués; J.A. Navío; M.C.Hidalgo
Comunicación oral

Effect of rice starch as binder in carbon xerogel preparation
Nicolás Rodríguez Riaño; Miguel Ángel Centeno; Yazmin Agamez-Pertuz; Jose Antonio Odriozola
Poster

Estrategias para el depósito de carbón activo en micromonolitos metálicos para su potencial uso como soportes catalíticos estructurados
J. L Santos-Muñoz; L. M. Martínez T; A. Monzón; J. A. Odriozola; M. A. Centeno
Poster

Estudio cinético de la reacción de reformado seco de metano con catalizadores estructurados de Ni-Ru
Inés Reyero; Victoria Garcilaso; Andrea Álvarez; Leidy Marcela Martínez; Fernando Bimbela; Miguel Ángel Centeno; Luis M. Gandía
Poster

Estudios operando DRIFTS-MS del proceso de Fischer-Tropsch
E. M. Jimenez-Barrera; F. Romero-Sarria; J. A. Odriozola
Poster

Near Ambient Pressure XPS (NAP-XPS): Estudio de un catalizador modelo para la reacción de PrOx
R.Castillo; S. Navarro-Jaén; V. Perez-Dieste; C. Escudero; F. Romero-Sarria; J.A. Odriozola
Poster

Producción de H_2 a partir de ácido fórmico usando catalizadores soportados de Pd: influencia del soporte y de la carga metálica
José Luis Santos; Luis Francisco Bobadilla; José Antonio Odriozola; Miguel Ángel Centeno
Poster

Pt-supported catalysts over proton conductor solids. Highly active catalysts for the WGS reaction
S. Navarro-Jaén; O.H. Laguna; M.A. Centeno; J.A. Odriozola
Poster

Recubrimiento de SAPO-34 sobre soportes cerámicos
M. Romero; I. Suarez; M.I. Domínguez; M.A. Centeno; J.A. Odriozola
Poster

Acoplamiento de WO_3 con un TiO_2 pre-sintetizado con exposición preferente de la cara {001}: Efecto de la incorporación del WO_3 en la actividad foto-catalítica
M.A. Lara; M.C. Hidalgo; J.A. Navío
Poster

Actividad foto-catalítica en el visible del TiO_2 dopado con nitrógeno preparado por un procedimiento combinado Sol-Gel/Solvotermal
J.A. Navío; M.C. Hidalgo; G. Restrepo; J.M. Marín
Poster

Alta actividad foto-catalítica en el UV de óxidos de cinc sintetizados por tres procedimientos diferentes: Una evaluación comparativa
C.A. Jaramillo-Páez; J.A. Navío; M.C. Hidalgo; M. Macias
Poster

6th International Conference on Semiconductor Photochemistry

11 – 14 septiembre [Oldenburg, Alemania]

Outstanding visible photocatalytic activity of a new mixed Bismuth Titanate material
M.C. Hidalgo; P. Zambrano; M.J. Sayagués; J.A. Navío
Poster

X Simposio Colombiano de Catálisis

25 – 29 septiembre [Boyacá, Colombia]

Catalizadores basados en titania metalizada con Au y Pt para la oxidación de CO
J.J. Murcia Mesa; Oscar H. Laguna; César Jaramillo-Páez; Hugo A. Rojas; María del Carmen Hidalgo; José Antonio Navío
Poster

XIV Reunión del Grupo Español del Carbón

22 – 25 octubre [Málaga, España]

Catalizadores de oro, platino y paladio sobre carbón para la producción de ácido 2,5-furandicarboxílico
C. Megías-Sayago; J. L. Santos; A. Lolli; S. Ivanova; S. Albonetti; M. A. Centeno; F. Cavani; J. A. Odriozola
Comunicación Oral

XX Congreso Argentino de Catálisis

1 – 3 noviembre [Córdoba, Argentina]

Valorización de suero de quesería mediante oxidación selectiva de lactosa empleando monolitos metálicos recubiertos con Au/Al_2O_3

Silvina Regenhardt;Camilo Meyer;Oihane Sanz;S. Ivanova;M.A. Centeno;J.A. Odriozola;Mario Montes;Teresita Garetto;Alberto Marchi
Poster

3rd Iberoamerican Conference on Advanced Oxidation Technologies | III CIPOA

14 – 17 noviembre [Antioquia, Colombia]

Photocatalytic treatment of commercial dyes used in handicraft factories
J.J. Murcia; C.C. Macías; A.C. Fernández; H. Rojas; C. Jaramillo-Páez; M.A. Lara; J.A. Navío; M.C. Hidalgo
Comunicación oral

Urban wastewater treatment by using Ag/ZnO and Pt/TiO2 photocatalysts
J.J. Murcia; L. G. Arias; H. Rojas; E.G. Ávila-Martínez; C. Jaramillo-Páez; M.A. Lara; J.A. Navío; M.C. Hidalgo
Poster

FORMACION / TRAINING

TESIS DOCTORALES/ DOCTOR DEGREE THESIS

Título:	**Desarrollo de materiales basados en TiO_2 modificado con funcionalidad fotocatalítica para aplicaciones medioambientales**
Autor:	Mayra Anabel Lara Angulo
Directores:	María del Carmen Hidalgo López, José Antonio Navío Santos
Calificación:	Sobresaliente “Cum Laude” por Unanimidad
Centro:	Universidad de Sevilla
Fecha:	7 de julio de 2017

Título:	**Desarrollo de materiales fotocataizadores basados en Bi, Ti y O con alta actividad en el visible para la eliminación de contaminantes en agua**
Autor:	Paola Zambrano Chacón
Directores:	María del Carmen Hidalgo López, José Antonio Navío Santos
Calificación:	Sobresaliente “Cum Laude” por Unanimidad
Centro:	Universidad de Sevilla
Fecha:	22 de septiembre de 2017

Título: **Catalizadores híbridos bifuncionales para la valorización química de biomasa**
Autor: Cristina Megías Sayago
Directores: Svetlana Lyubomirova Ivanova, José Antonio Odriozola Gordón
Calificación: Sobresaliente “Cum Laude” por Unanimidad
Centro: Universidad de Sevilla
Fecha: 29 de septiembre de 2017

Título: **On the rate-limiting step of the WGs reaction: design of the model catalyst**
Autor: Nuria García Moncada
Directores: Francisca Romero Sarria, José Antonio Odriozola Gordón
Calificación: Sobresaliente “Cum Laude” por Unanimidad
Centro: Universidad de Sevilla
Fecha: 15 de diciembre de 2017

Formación de Graduados / Master Degree Thesis

Título: **Materiales para la purificación de biogas**
Autor: Juan Carlos Navarro de Miguel
Directores: Svetlana Lyubomirova Ivanova, María Isabel Domínguez Leal
Grado: Trabajo Fin de Master
Centro: Universidad de Sevilla
Año Académico: 2017-2018 (30 junio 2017)

Título: **Estudios In operando de la reacción de Water Gas Shift (WGS)**
Autor: Dolores Jurado Fuentes
Directores: Francisca Romero Sarria, José Antonio Odriozola Gordón
Grado: Trabajo Fin de Master
Centro: Universidad de Sevilla
Año Académico: 2017-2018 (30 junio 2017)

Título: **Corrosión en biomateriales influenciada por microorganismos**
Autor: Blanca María Benítez Zamora
Directora: Leidy Marcela Martínez Tejada
Grado: Trabajo Fin de Grado
Centro: Universidad de Sevilla
Año Académico: 2017-2018 (5 julio 2017)
Título: **Complejos que liberan monóxido de carbono como agentes terapeuticos**

Autor: Alba Rodriguez González
Directora: Svetlana Lyubomirova Ivanova
Grado: Trabajo Fin de Grado
Centro: Universidad de Sevilla
Año Académico: 2017-2018 (21 julio 2017)

Título: **Diseño de catalizadores eficientes a partir de residuos metalúrgicos para procesos de reformado**
Autor: Pablo Navarro Vicente
Directores: Luis Francisco Bobadilla Baladrón, José Antonio Odriozola Gordón
Grado: Trabajo Fin de Master
Centro: Universidad de Sevilla
Año Académico: 2017-2018 (22 septiembre 2017)

Título: **Estudios de las aplicaciones biomédicas de las nanopartículas de plata**
Autor: Teresa Fernández Bueno
Directores: María Isabel Domínguez Leal, Svetlana Lyubomirova Ivanova
Grado: Trabajo Fin de Grado
Centro: Universidad de Sevilla
Año Académico: 2017-2018 (29 septiembre 2017)

DOCENCIA / TEACHING

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

EQUIPAMIENTO CIENTÍFICO AVAILABLE EQUIPMENT

- Molino de bolas de movimiento planetario PM100 de RETSCH
- Espectrofotómetro Infrarrojos: Thermo-Nicolet Nexus FT-IR; Thermo-Nicolet 380 FT-IR
- Accesorio DRIFTS, celdas de alta y baja temperatura
- Sistema de vacío cuarzo/vidrio para adsorción de moléculas sonda seguido por FT-IR
- Espectrómetro de masas: Balzers Thermostar

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

- Cromatografos HPLC
- Horno de soldadura: Microtest Máquina de ensayos EM2/200/FR
- Baño de ultrasonidos
- Hornos
- Horno para tratamiento de aceros a alta temperatura equipado con medidores de flujo e inyector de agua.
- Planta Piloto de Integración de reacciones catalíticas Reference PID Eng&Tech
- Espectrómetro Uv-Vis, con esfera integradora para muestras sólidas)
- Analizador de Carbón Orgánico
- Dip-Coater con cámara de temperatura
- Reactores catalíticos de gases con detección por cromatografía de gases y espectrometría de masas.
- Reactores catalíticos de líquidos que permiten el seguimiento de hasta 8 reacciones de forma simultánea con control de temperatura y flujo de gases.
- Reactores catalíticos de líquidos de alta presión y temperatura con agitación interna y control de flujo de gases.
- Reactores fotocatalíticos con lámparas de Xe y Hg.

INGENIERÍA DE CERÁMICOS PARA AMBIENTES EXTREMOS

ENGINEERED CERAMICS FOR EXTREME ENVIRONMENTS

PERSONAL / PERSONNEL

Catedráticos

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

Científicos Titulares

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

Profesores Titulares

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

Investigadores Contratados

Dr. Miguel Antonio Cruz Carrillo (h. junio)
Dra. Esperanza Pavón González (h. junio)

Profesor Contratado Doctor

Dr. Joaquin Ramírez Rico

Becarios Predoctorales

Ldo. Rafael Cabezas Rodríguez
Lda. María del Pilar Orihuela Espina
Ldo. Francisco J. Osuna Barroso

PROYECTOS DE INVESTIGACIÓN / RESEARCH PROJECTS

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

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

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

RESUMEN / ABSTRACT

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

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

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

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

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

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

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

RESUMEN / ABSTRACT

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

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

EPOs) y reconocer y promover la I+D como un elemento esencial para la generación de nuevos conocimientos de excelencia.

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

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

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

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

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

Código/Code: TAHUB-082. Programa Talent HUB
Periodo/Period: 01-02-2015 / 20-06-2017
Organismo Financiador/Financial source: Junta de Andalucía
Importe total/Total amount: 142642,80 €
Investigador responsable/Research head: Esperanza Pavón González

RESUMEN / ABSTRACT

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

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

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

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

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

An immobilization protocol of harmful anionic products like AsO_4^{2-}, SO_4^{2-} will be established, using the best adsorbent in function of both the structure and the funcionalization of the

highly charged swelling mica. Afterwards, the applicability of these adsorption reactions will be tested in actual contaminated soils from Chili and Spain.

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JUNTA DE ANDALUCIA

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

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

RESUMEN / ABSTRACT

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

Desde la segunda mitad del siglo XX la Humanidad se ha enfrentado a un enorme desarrollo científico y tecnológico que es el responsable de un incremento de la contaminación mediambiental. Como ejemplo podemos mencionar dos problemas que en la actualidad son motivos de preocupación y actuación de la Junta de Andalucía: contaminación de los litorales andaluces y las aguas residuales urbanas. Por tanto, estamos ante un problema complejo en el que los agentes contaminantes son variados, las fuentes de procedencia son diversas y las vías o rutas seguidas por los distintos contaminantes, frecuentemente, escapan al control necesario para evitar efectos indeseados sobre el entorno natural y urbano. Es por ello, que se demanda una investigación a nivel básico y aplicado de los mecanismos necesarios para la inmovilización de dichos cationes nocivos.

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

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

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

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

OTROS PROYECTOS / OTHER PROJECTS

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

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

CONVENIOS Y CONTRATOS / CONTRACTS AND AGREEMENTS

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

Periodo/Period:	01-07-2015 / 30-06-2017
Organismo Financiador/Financial source:	ENRESA

importe total/Total amount:	139.755 €
Investigador responsable/Research head:	Miguel Angel Castro Arroyo
Componentes/Research group:	María del Mar Orta Cuevas, M. Dolores Alba Carranza

Artículos Publicados en Revistas SCI / Papers In SCI Journals

Biomorphic ceramics from wood-derived precursors

Ramirez-Rico, J.; Martinez-Fernandez, J.; Singh, M.
International Materials Reviews, **62** (2017) Issue 8
Agosto, 2017 | **DOI:** 10.1080/09506608.2017.1354429

Materials development is driven by microstructural complexity and, in many cases, inspired by biological systems such as bones, shells and wood. In one approach, one selects the main microstructural features responsible for improved properties and design processes to obtain materials with such microstructures (continuous-fibre-reinforced ceramics, porous ceramics, fibrous ceramic monoliths, etc.). In a different approach, it is possible to use natural materials directly as microstructural templates. Biomorphic ceramics are produced from natural and renewable resources (wood or wood-derived products). A wide variety of SiC-based ceramics can be fabricated by infiltration of silicon or silicon alloys into cellulose-derived carbonaceous templates, providing a low-cost route to advanced ceramic materials with near-net shape potential and amenable to rapid prototyping. These materials have tailorable microstructure and properties, and behave like ceramic materials manufactured by advanced ceramic processing approaches. This review aims to be a comprehensive description of the development of bioSiC ceramics: from wood templates and their microstructure to potential applications of bioSiC materials.

Cutin from agro-waste as a raw material for the production of bioplastics

Heredia-Guerrero, J.A.; Heredia, A.; Dominguez, E.; Cingolani, R.; Bayer, I.S.; Athanassiou, A.; Benitez, J.J.
Journal of Experimental Botany, **68** (2017) 5401-5410
Agosto, 2017 | **DOI:** 10.1093/jxb/erx272

Cutin is the main component of plant cuticles constituting the framework that supports the rest of the cuticle components. This biopolymer is composed of esterified bi- and trifunctional fatty acids. Despite its ubiquity in terrestrial plants, it has been underutilized as raw material due to its insolubility and lack of melting point. However, in recent years, a few technologies have been developed to obtain cutin monomers from several agro-wastes at an industrial scale. This review is focused on the description of cutin properties, biodegradability, chemical composition, processability, abundance, and the state of art of the fabrication of cutin-based materials in order to evaluate whether this biopolymer can be considered a source for the production of renewable materials.

Cellulose-polyhydroxylated fatty acid ester-based bioplastics with tuning properties: Acylation via a mixed anhydride system

Heredia-Guerrero, JA; Goldoni, L; Benitez, JJ; Davis, A; Ceseracciu, L; Cingolani, R; Bayer, IS; Heinze, T; Koschella, A; Heredia, A; Athanassiou, A
Carbohydrate Polymers, **173** (2017) 312-320
Octubre, 2017 | **DOI:** 10.1016/j.carbpol.2017.05.068

The synthesis of microcrystalline cellulose (MCC) and 9,10,16-hydroxyhexadecanoic (aleuritic) acid ester-based bioplastics was investigated through acylation in a mixed anhydride (trifluoroacetic acid (TFA)/trifluoroacetic acid anhydride (TFAA)), chloroform co-solvent system. The effects of chemical interactions and the molar ratio of aleuritic acid to the anhydroglucose unit (AGU) of cellulose were investigated. The degree of substitution (DS) of new polymers were characterized by two-dimensional solution-state NMR and ranged from 0.51 to 2.60. The chemical analysis by attenuated total reflection Fourier transform infrared spectroscopy (ATR-FTIR) confirmed the presence of aleuritate groups in the structure induces the formation of new H-bond networks. The tensile analysis and the contact angle measurement confirmed the ductile behavior and the hydrophobicity of the prepared bioplastics. By increasing the aleuritate amounts, the glass transition temperature decreased and the solubility of bioplastic films in most common solvents was improved. Furthermore, this new polymer exhibits similar properties compared to commercial cellulose derivatives.

Front contact optimization of industrial scale CIGS solar cells for low solar concentration using 2D physical modeling

Delgado-Sanchez, JM; Lopez-Gonzalez, JM; Orpella, A; Sanchez-Cortezon, E; Alba, MD; Lopez-Lopez, C; Alcubilla, R
Renewable Energy, **101** (2017) 90-95
Enero, 2017 | **DOI:** 10.1016/j.renene.2016.08.046

Cu(In,Ga)Se-2 (CIGS) technology is one of the best absorber materials with record efficiencies among photovoltaic thin-film technologies (22.3% at lab scale and 16% at large commercial module). Although research on this material was originally motivated by low-cost, glass-glass applications focusing to fixed photovoltaic structures, the high efficiency values make CIGS an interesting alternative for low concentration systems. In this paper a 2D model for Cu(In,Ga)Se-2 (CIGS) solar cells under low solar concentration is described and contrasted with experimental data. Using simulation, the effect of front electric contact design parameters: finger width, finger separation, and number of buses are analyzed for solar concentrations from 1 up to 10 suns. Efficiency maps allowing front contact grid optimization are shown and analyzed for each concentration factor (Cx), assessing the viability of CIGS solar cells for low concentration applications, where commercial CIGS solar cells may exhibit 35% of electrical power increases with proper front grid optimization under low concentration respect to conventional grid design.

New insights into surface-functionalized swelling high charged micas: Their adsorption performance for non-ionic organic pollutants

Pazos, MC; Castro, MA; Cota, A; Osuna, FJ; Pavon, E; Alba, MD
Journal of Industrial and Engineering Chemistry, **52** (2017) 179-186

Agosto, 2017 | **DOI:** 10.1016/j.jiec.2017.03.042

The major components of the wastewater from the petroleum refineries are benzene, toluene and phenol and one of the techniques applied to the treatment of effluents is sorption using organo-functionalized clay. The materials exploited in the present study are a family of surface-functionalized synthetic micas and their sorption capacities for non-ionic organic pollutants are analyzed. The organo-functionalization of their surface provides them the capacity to sorb effectively non-ionic pollutants in the interface. Their adsorption performance is a function of the alkylamonium properties such as the chain length, the mass fraction and the organization of the organic cation in the interlayer space of the micas.

Effect of the crystal chemistry on the hydration mechanism of swelling micas

Pavon, E; Alba, MD; Castro, MA; Cota, A; Osuna, FJ; Pazos, MC
Geochimica et Cosmochimica Acta, **217** (2017) 231-239
Noviembre, 2017 | **DOI:** 10.1016/j.gca.2017.08.028

Swelling and dehydration under minor changes in temperature and water vapor pressure is an important property that clays and clay minerals exhibit. In particular, their interlayer space, the solid-water interface and the layers' collapse and re-expansion have received much attention because it affects to the dynamical properties of interlayer cations and thus the transfer and fate of water and pollutants. In this contribution, the dehydration and rehydration mechanism of a swelling high-charge mica family is examined by in situ X-ray Diffraction. The effect of the aluminosilicate layer charge and the physicochemical properties of the interlayer cations on these processes are analyzed. The results showed that the dehydration temperature and the number of steps involved in this process are related to the layer charge of the silicate and the physicochemical properties of the interlayer cations. Moreover, the ability to adsorb water molecules in a confined space with high electric field by the interlayer cations does not only depend on their hydration enthalpy but also on the electrostatic parameters of these cations.

Performance of biomorphic Silicon Carbide as particulate filter in diesel boilers

Orihuela, M Pilar; Gomez-Martin, Aurora; Becerra, Jose A; Chacartegui, Ricardo; Ramirez-Rico, Joaquin
Journal of Environmental Management, **203** (2017) 907-919
Diciembre, 2017 | **DOI:** 10.1016/j.jenvman.2017.05.003

Biomorphic Silicon Carbide (bioSiC) is a novel porous ceramic material with excellent mechanical and thermal properties. Previous studies have demonstrated that it may be a good candidate for its use as particle filter media of exhaust gases at medium or high temperature. In order to determine the filtration efficiency of biomorphic Silicon Carbide, and its adequacy as substrate for diesel particulate filters, different bioSiC-samples have been tested in the flue gases of a diesel boiler. For this purpose, an experimental facility to extract a fraction of the boiler exhaust flow and filter it under controlled conditions has been designed and built. Several filter samples with different microstructures, obtained from different precursors, have been tested in this bench. The experimental campaign was focused on the measurement of the number and size of particles before and after placing the samples. Results show that the initial efficiency of filters made from natural precursors is severely determined by the cutting direction and associated microstructure. In biomorphic Silicon Carbide derived from radially cut wood, the initial efficiency of the filter is higher than 95%. Nevertheless, when the cut of the wood is axial, the efficiency depends on the pore size and the permeability, reaching in some cases values in the range 70–90%. In this case, the presence of macropores in some of the samples reduces their efficiency as particle traps. In continuous operation, the accumulation of particles within the porous media leads to the formation of a soot cake, which improves the efficiency except in the case when extra-large pores exist. For all the samples, after a few operation cycles, capture efficiency was higher than 95%. These experimental results show the potential for developing filters for diesel boilers based on biomorphic Silicon Carbide.

High temperature compressive strength and creep behavior of Si-Ti-C-O fiber-bonded ceramics

Vera, MC; Martinez-Fernandez, J; Singh, M; Ramirez-Rico, J
Journal of the European Ceramic Society, **37** (2017) 4442-4448
Diciembre, 2017 | **DOI:** 10.1016/j.jeurceramsoc.2017.06.037

Fiber bonded silicon carbide ceramic materials provide cost-advantage over traditional ceramic matrix composites and require fewer processing steps. Despite their interest in extreme environment thermostructural applications no data on long term mechanical reliability other than static fatigue is available for them. We studied the high temperature compressive strength and creep behavior of a fiber bonded SiC material obtained by hot-pressing of Si Ti-C-O fibers. The deformation mechanism and onset of plasticity was evaluated and compared with other commercial SiC materials. Up to 1400 degrees C, plasticity is very limited and any macroscopic deformation proceeds by crack formation and damage propagation. A transient viscous creep stage is observed due to flow in the silica matrix and once steady state is established, a stress exponent n similar to 4 and an activation energy Q similar to 700 kJ mol(-1) are found. These results are consistent with previous data on creep of polymer derived SiC fibers and polycrystals.

Cs+ immobilization by designed micaceous adsorbent under subcritical conditions

Osuna, FJ; Cota, A; Pavon, E; Pazos, MC; Alba, MD
Applied Clay Science, **143** (2017) 293-299
Julio, 2017 | **DOI:** 10.1016/j.clay.2017.03.041

The adsorption of Cs+ by clay minerals is a complicate process, being cation exchange and frayed-edge sites the major mechanisms that govern it. However, environmental variables have a significant impact on the process. In this work, the influence of the temperature and time in the cesium adsorption capacity of Na-Mica-n (n = 2 and 4) have been explored under subcritical conditions. Those synthetic micas were able to immobilize cations Cs+ combining adsorption at nonspecific sites, at specific sites and chemical reaction. The distribution constant of Cs+ was larger in the Na-Mica-2 denoting a higher concentration of specific adsorption sites when layer charge decreased.

High-temperature thermal conductivity of biomorphic SiC/Si ceramics

Ramirez-Rico, J.; Singh, M.; Zhu, D.; Martinez-Fernandez, J.
Journal of Materials Science, **52** (2017) 10038-10046
Septiembre, 2017 | **DOI:** 10.1007/s10853-017-1199-y

Thermal conductivity of biomorphic SiC/Si, a silicon carbide + silicon containing two phase material, was evaluated using the laser steady-state heat flux method. These materials were processed via silicon melt infiltration of wood-derived carbon scaffolds. In this approach, heat flux was measured through the thickness when one side of the specimen was heated with a 10.6-A mu m CO2 laser. A thin mullite layer was applied to the heated surface to ensure absorption and minimize reflection losses, as well as to ensure a consistent emissivity to facilitate radiative loss corrections. The influence of the mullite layer was accounted for in the thermal conductivity calculations. The effect of microstructure and composition (inherited from the wood carbonaceous performs) on measured conductivity was evaluated. To establish a baseline for comparison, a dense, commercially available sintered SiC ceramic was also evaluated. It was observed that at a given temperature, thermal conductivity falls between that of single-crystal silicon and fine-grained polycrystalline SiC and can be rationalized in terms of the SiC volume fraction in biomorphic SiC/Si material.

Precision and accuracy of stress measurement with a portable X-ray machine using an area detector

Lee, SY; Ling, JJ; Wang, SH; Ramirez-Rico, J
Journal of Applied Crystallography, **50** (2017) 131-144
Febrero, 2017 | **DOI:** 10.1107/S1600576716018914

The use of portable X-ray stress analyzers, which utilize an area detector along with the newly adopted 'cos alpha' or full-ring fitting method, has recently attracted increasing interest. In laboratory conditions, these measurements are fast, convenient and precise because they employ a single-exposure technique that does not require sample rotation. In addition, the effects of grain size and orientation can be evaluated from the Debye ring recorded on the area detector prior to data analysis. The accuracy of the measured stress, however, has been questioned because in most cases just a single reflection is analyzed and the sample-to-detector distances are relatively short. This article presents a comprehensive analysis of the uncertainty associated with a state-of-the-art commercial portable X-ray device. Annealed ferrite reference powders were used to quantify the instrument precision, and the accuracy of the stress measurement was tested by in situ tensile loading on 1018 carbon steel and 6061 aluminium alloy bar samples. The results show that the precision and accuracy are sensitive to the instrument (or sample) tilt angle (0) as well as to the selected hkl reflection of the sample. The instrument, sample and data analysis methods all affect the overall uncertainty, and each contribution is described for this specific portable X-ray system. Finally, on the basis of the conclusions reached, desirable measurement/analysis protocols for accurate stress assessments are also presented.

Insolubilization and thermal stabilization of a long-chain polyester by noncatalyzed melt-polycondensation synthesis in air

Benitez, JJ; Heredia-Guerrero, JA; Cruz-Carrillo, MA; Barthel, MJ; Knicker, HE; Heredia, A
Journal of Applied Polymer Science, **134** (2017) art. 44350
Enero, 2017 | **DOI:** 10.1002/app.44350

Self-standing films of poly(-hydroxyl hexadecanoic acid) [poly(-OHC16)] have been prepared by noncatalyzed melt-polycondensation in air at 150, 175, and 200 degrees C. Poly(-OHC16)s obtained are characterized as polyesters by infrared spectroscopy (FT-IR) and solid state magic angle spinning C-13 nuclear magnetic resonance (C-13 MAS-NMR). Structurally, poly(-OHC16)s are quite crystalline as revealed by wide angle X-ray diffraction (WAXD). The presence of oxygen in the reaction atmosphere causes a mild oxidation in the form of peroxyester species, tentatively at the interphase between poly(-OHC16) crystallites, and the structure amorphization. The interfacial peroxyester phase ends up in the encapsulation of the polyester grains and provides a barrier towards the action of solvents. Thermal stabilization and insolubility resulting from the synthesis conditions used are interesting features to prepare solvent and heat resistant poly(-OHC16) coatings. Thus, a few microns thick poly(-OHC16) layer has been fabricated on aluminum foil and its resistivity towards a chloroform:methanol (1:1, v:v) mixture has been confirmed.

Failure mode and effect analysis of a large scale thin-film CIGS photovoltaic module

Delgado-Sanchez, JM; Sanchez-Cortezon, E; Lopez-Lopez, C; Aninat, R; Alba, MD
Engineering failure analysis, **76** (2017) 55-60
Junio, 2017 | **DOI:** 10.1016/j.engfailanal.2017.02.004

The efficiency of thin-film CIGS based cells at the laboratory scale is now getting closer to conventional Silicon technologies. As a consequence, the long-term stability of CIGS is now one of the main challenges left to address in order to assess its potential as an alternative for photovoltaic plants. This paper reports an overview of the critical risks for the commercial viability of the CIGS thin-film technology. The key causes of the potential failures of this technology are determined through the Failure Mode Analysis and Effects (FMEA) methodology. To validate the results obtained from the FMEA, aging tests and outdoor monitoring were also carried out. Based on the results obtained, we argue that the encapsulation material is the main cause of degradation in CIGS modules.

Features of electrical properties of BE-C(Fe) biocarbons carbonized in the presence of an Fe-containing catalyst

Popov, VV; Orlova, TS; Gutierrez-Pardo, A; Ramirez-Rico, J
Physics of the Solis State, **59** (2017) 703-709
Abril, 2017 | **DOI:** 10.1134/S1063783417040205

The effect of partial graphitization on electrical and galvanomagnetic properties of BE-C(Fe) biomorphic carbons produced by beech wood carbonization at temperatures of 850-1600A degrees C in the presence of an iron-containing catalyst is studied. The use of an Fe catalyst at

D cent (carb) ae<yen> 1000A degrees C leads to the formation of nanoscale graphite-phase inclusions; its total volume and nanocrystallite sizes increase with D cent (carb). The data on the carrier concentration and mobility are obtained. It was shown that partially graphitized BE-C(Fe) carbons with D cent (carb) ae<yen> 1000A degrees C in the conductivity type and magnetoresistance features relate to highly disordered metal systems whose conductivity can be described taking into account the contribution of quantum corrections, mainly the correction caused by the electron-electron interaction. It is shown that nonmonotonic dependences of the Hall constant R on the magnetic field are characteristic of BE-C(Fe) samples with 1000 ae<currency> D cent (carb) < 1600A degrees C, which is most probably caused by the contribution of various carrier groups, i.e., electrons and holes. In BE-C(Fe) samples with D cent (carb) = 1600A degrees C, the Hall coefficient corresponds to the metal state, which is associated with conducting medium homogenization resulting from the formation of a significant graphite phase volume.

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

EMN Soft Materials 2017
18 – 22 junio [Viena, Austria]

Organoclay nanocomposites for sustainable management of toxic waste compounds
E. Pavon; M. Escudey; F. Albornoz; A. Cota; F.J. Osuna; M.D. Alba
Conferencia Invitada

Materials and the Environment Symposium at the XXVI International Materials Research Congress
20 – 25 agosto [Cancún, México]

New method of synthesis of aluminium-pillared mica from synthetic Na-2-Mica
L. Ruiz; J. R. Chaparra; M. C. Pazos; M. D. Alba; A. Cota; F. J. Osuna; E. Pavón
Poster

The 7th International Conference on Clays in Natural and Engineered Barriers for Radioactive Waste Confinement
24 – 27 septiembre [Davos, Suiza]

Cesium immobilization by designed micaceous adsorbent under subcritical conditions
M.D. Alba; A. Cota; F.J. Osuna; E. Pavon; M.C. Pazos
Poster

Chemical processes of designed clays for anionic radioactive waste management
F.J. Osuna; A. Cota; E. Pavón; M.D. Alba
Poster

FORMACION / TRAINING

FORMACIÓN DE GRADUADOS / MASTER DEGREE THESIS

Título:	**Propuesta de evaluación de vitrificación de residuos radioactivos por bentonitas**
Autor:	Agustín Cota Reguero
Directora:	María Dolores Alba Carranza
Grado:	Trabajo Fin de Master
Centro:	Universidad de Sevilla
Año Académico:	2017-2018 (10 julio 2017)

Título:	**Desarrollo y caracterización de recubrimientos nanoporosos por magnetrón sputtering como blancos solidos de He para aplicaciones en física nuclear**
Autor:	David Feria Cervantes
Directores:	Joaquín Ramírez Rico, Vanda Cristina Fortio Godinho, M. Asunción Fernández Camacho
Grado:	Trabajo Fin de Grado
Centro:	Universidad de Sevilla
Año Académico:	2017-2018 (20 septiembre 2017)

Título:	**Funcionalización de sepiolita natural y adsorbentes sintéticos basados en silicatos de magnesio potencialmente aplicables en adsorción de derivados de petróleo**
Autor:	Christian Fabián Valera Olivera
Directora:	María Dolores Alba Carranza
Grado:	Trabajo Fin de Grado
Centro:	Universidad de Sevilla
Año Académico:	2017-2018 (29 noviembre 2017)

DOCENCIA / TEACHING

Student of Second Cycle of Chemistry Engineer-FLILLE13

Erasmus Mundus
Dra. M. Dolores Alba Carranza
Dra. Esperanza Pavón González
Lugar: National Graduate School of Engineering Chemistry of Lille (Lille, Francia)

Master Maestría en Quimica

Química Inorgánica
Dra. M. Dolores Alba Carranza
Dra. Esperanza Pavón González
Lugar: Universidad Pedagógica y Tecnológica de Colombia (UPTC, Tunja)

Programa de Doctorado en Ciencia y Tecnología de Nuevos Materiales

Introducción a la Resonancia Magnética Nuclear de Estado Sólido
Dra. M. Dolores Alba Carranza
Dra. Esperanza Pavón González
Lugar: Universidad de Sevilla

EQUIPAMIENTO CIENTÍFICO AVAILABLE EQUIPMENT

- Horno tubular hasta 1200º C
- Horno tubular en atmósfera controlada hasta 1700º C
- Horno de Cámara hasta 1650º C
- Horno de Cámara hasta 1200 ºC
- Estufa hasta 400º C
- Estufa hasta 200º C
- Caja de guantes
- Reactores hidrotermales
- Línea de vacío
- 3 microscopios AFM.
- Microscopio STM en aire.
- Calorímetro diferencial de barrido (DSC).
- Analizador mecánico dinámico (DMA).
- Máquina de ensayos mecánicos por tracción.
- Analizador de ángulo de contacto.
- Balanza Langmuir-Blodgett (LB).
- Spin coater.

MECANOQUÍMICA Y REACTIVIDAD DE MATERIALES

MECHANOCHEMISTRY AND REACTIVITY OF MATERIALS

PERSONAL / PERSONNEL

Investigadores Científicos

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

Científicos Titulares

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

Profesores Titulares

Dra. María Dolores Alcalá González

Profesor Vinculado "Ad Honorem"

Dr. José Manuel Criado Luque

Investigadores Contratados

Dr. José Manuel Córdoba Gallego
Dr. Antonio Perejón Pazo
Dr. Pedro E. Sánchez Jiménez

Profesor Contratado Doctor

Dra. María Regla Ayala Espinar

Becarios Predoctorales

Lda. Cristina García Garrido ()
Lda. Eva Gil González
Lda. Beatriz Sarrión Aceytuno

Personal Contratado

Dra. Mónica Benítez Guerrero
Ldo. José Manuel Obrero Pérez

PROYECTOS DE INVESTIGACIÓN / RESEARCH PROJECTS

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

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

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

RESUMEN / ABSTRACT

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

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

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

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

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

RESUMEN / ABSTRACT

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

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

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

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

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

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

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

Preparación por molienda reactiva de nanocomposites de interés tecnológico

Preparation of technically interesting nanocomposites by mechanochemistry

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

RESUMEN / ABSTRACT

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

la muestra a la vez que se somete a un campo eléctrico. Con este procedimiento se espera una reducción drástica de la temperatura requerida para la compactación total de la zirconia. El estudio de la cinética de sinterización bajo los efectos de un campo eléctrico se llevará a cabo mediante un equipo a desarrollar en el proyecto a partir de un dilatómetro que se modificará de modo que permita seguir la evolución de la dimensión de la muestra en función de la temperatura estando ésta sometida a un campo eléctrico.

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

Convenios y Contratos / Contracts and Agreements

Analizador térmico para estudios en condiciones realistas de funcionamiento de materiales

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

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

Lead-Free Polycrystalline Ferroelectric Nanowires with Enhanced Curie Temperature

Datta, Anuja; Sanchez-Jimenez, Pedro E.; Al Orabi, Rabih Al Rahal; Calahorra, Yonatan; Ou, Canlin; Sahonta, Suman-Lata; Fornari, Marco; Kar-Narayan, Sohini
Advanced Functional Materials, **27** (2017) 1701169
Agosto, 2017 | **DOI:** 10.1002/adfm.201701169

Ferroelectrics are important technological materials with wide-ranging applications in electronics, communication, health, and energy. While lead-based ferroelectrics have remained the predominant mainstay of industry for decades, environmentally friendly lead-free alternatives are limited due to relatively low Curie temperatures (T-C) and/or high cost in many cases. Efforts have been made to enhance T-C through strain engineering, often involving energy-intensive and expensive fabrication of thin epitaxial films on lattice-mismatched substrates. Here, a relatively simple and scalable sol-gel synthesis route to fabricate polycrystalline (Ba0.85Ca0.15)(Zr0.1Ti0.9)O-3 nanowires within porous templates is presented, with an observed enhancement of T-C up to similar to 300 degrees C as compared to similar to 90 degrees C in the bulk. By combining experiments and theoretical calculations, this effect is attributed to the volume reduction in the template-grown nanowires that modifies the balance between different structural instabilities. The results offer a cost-effective solution-based approach for strain-tuning in a promising lead-free ferroelectric system, thus widening their current applicability.

The Oxy-CaL process: A novel CO_2 capture system by integrating partial oxy-combustion with the Calcium-Looping process

Ortiz, C; Valverde, JM; Chacartegui, R; Benitez-Guerrero, M; Perejon, A; Romeo, LM
Applied Energy, **196** (2017) 1-17
Junio, 2017 | **DOI:** 10.1016/j.apenergy.2017.03.120

This paper proposes a novel CO_2 capture technology from the integration of partial oxy-combustion and the Calcium -Looping capture process based on the multicycle carbonation/calcination of limestone derived CaO. The concentration of CO_2 in the carbonator reactor is increased by means of partial oxycombustion, which enhances the multicycle CaO conversion according to thermogravimetric analysis results carried out in our work, thus improving the CO_2 capture efficiency. On the other hand, energy consumption for partial oxy-combustion is substantially reduced as compared to total oxy-combustion. All in all, process simulations indicate that the integration of both processes has potential advantages mainly regarding power plant flexibility whereas the overall energy penalty is not increased. Thus, the resulting energy consumption per kilogram of CO_2 avoided is kept smaller than 4 MI/kg CO_2, which remains below the typical values reported for total oxy-combustion and amine based CO_2 capture systems whereas CO_2 capture efficiency is enhanced in comparison with the Calcium - Looping process.

The Oxy-CaL process: A novel CO2 capture system by integrating partial oxy-combustion with the Calcium-Looping process

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This paper proposes a novel CO_2 capture technology from the integration of partial oxy-combustion and the Calcium -Looping capture process based on the multicycle carbonation/calcination of limestone derived CaO. The concentration of CO_2 in the carbonator reactor is increased by means of partial oxycombustion, which enhances the multicycle CaO conversion according to thermogravimetric analysis results carried out in our work, thus improving the CO_2 capture efficiency. On the other hand, energy consumption for partial oxy-combustion is substantially reduced as compared to total oxy-combustion. All in all, process simulations indicate that the integration of both processes has potential advantages mainly regarding power plant flexibility whereas the overall energy penalty is not increased. Thus, the resulting energy consumption per kilogram of CO_2 avoided is kept smaller than 4 MI/kg CO_2, which remains below the typical values reported for total oxy-combustion and amine based CO_2 capture systems whereas CO_2 capture efficiency is enhanced in comparison with the Calcium - Looping process.

Large-Scale Storage of Concentrated Solar Power from Industrial Waste

Perejon, Antonio; Valverde, Jose Manuel; Miranda-Pizarro, Juan; Sanchez-Jimenez, Pedro E.; Perez-Maqueda, Luis A.
ACS Sustainable Chemistry & Engineering, **5** (2017) 2265-2272
Marzo, 2017 | **DOI:** 10.1021/acssuschemeng.6b02576

Deep penetration of renewable energies into the grid relies on the development of large-scale energy storage technologies using cheap, abundant, and nontoxic materials. Concentrated solar power (CSP) is particularly suitable to massively store thermal energy for dispatchable electricity generation. This is currently accomplished in a few demonstration plants by using molten salts albeit in a not competitive way yet. Process simulation studies indicate that thermochemical energy storage of CSP by means of the calcium looping (CaL) technology would reduce the cost of storage and increase the flexibility of energy supply provided that widely available and cheap CaO precursors with high and stable multicycle activity are used. In this work, we investigate the behavior of calcium rich steel slag at CaL conditions that would expectedly maximize the efficiency of CSP energy storage and power production. When treated with acetic acid, this nontoxic widely abundant waste yields a CaO rich solid with stable conversion near 0.8 over successive carbonation/calcination cycles at these CaL conditions.

Defect chemistry and electrical properties of $BiFeO_3$

Schrade, M; Maso, N; Perejon, A; Perez-Maqueda, LA; West, AR
Journal of Materials Chemistry C, **5** (2017) 10077-10086
Octubre, 2017 | **DOI:** 10.1039/c7tc03345a

$BiFeO_3$ attracts considerable attention for its rich functional properties, including room temperature coexistence of magnetic order and ferroelectricity and more recently, the discovery of conduction pathways along ferroelectric domain walls. Here, insights into the defect chemistry and electrical properties of $BiFeO_3$ are obtained by in situ measurements of electrical conductivity, sigma, and Seebeck coefficient, a, of undoped, cation-stoichiometric $BiFeO_3$ and acceptor-doped Bi1-xCaxFeO_3-delta ceramics as a function of temperature and oxygen partial pressure pO(2). Bi1-xCaxFeO_3-delta exhibits p-type conduction; the dependencies of s and a on pO(2) show that Ca dopants are compensated mainly by oxygen vacancies. By contrast, undoped $BiFeO_3$ shows a simultaneous increase of s and a with increasing pO(2), indicating intrinsic behavior with electrons and holes as the main defect species in almost equal concentrations. The pO(2)-dependency of s and a cannot be described by a single point defect model but instead, is quantitatively described by a combination of intrinsic and acceptor-doped characteristics attributable to parallel conduction pathways through undoped grains and defect-containing domain walls; both contribute to the total charge transport in $BiFeO_3$. Based on this model, we discuss the charge transport mechanism and carrier mobilities of $BiFeO_3$ and show that several previous experimental findings can readily be explained within the proposed model.

Calcium-Looping performance of steel and blast furnace slags for thermochemical energy storage in concentrated solar power plants

Manuel Valverde, Jose; Miranda-Pizarro, Juan; Perejon, Antonio; Sanchez-Jimenez, Pedro E.; Perez-Maqueda, Luis A.
Journal of CO2 Utilization, **22** (2017) 143-154
Diciembre, 2017 | **DOI:** 10.1016/j.jcou.2017.09.021

The Calcium Looping (CaL) process, based on the carbonation/calcination of CaO, has been proposed as a feasible technology for Thermochemical Energy Storage (TCES) in Concentrated Solar Power (CSP) plants. The CaL process usually employs limestone as CaO precursor for its very low cost, non-toxicity, abundance and wide geographical distribution. However, the multicycle activity of limestone derived CaO under relevant CaL conditions for TCES in CSP plants can be severely limited by pore plugging. In this work, the alternative use of calcium-rich steel and blast furnace slags after treatment with acetic acid is investigated. A main observation is that the calcination temperature to regenerate the CaO is significantly reduced as compared to limestone. Furthermore, the multicycle activity of some of the slags tested at relevant CaL conditions for TCES remains high and stable if the treated samples are subjected to filtration. This process serves to remove silica grains, which helps decrease the porosity of the CaO resulting from calcination in the mesoporous range thus mitigating pore plugging.

Large-scale high-temperature solar energy storage using natural minerals

Benitez-Guerrero, Monica; Sarrion, Beatriz; Perejon, Antonio; Sanchez-Jimenez, Pedro E.; Perez-Maqueda, Luis A.; Manuel Valverde, Jose
Solar Energy Materials and Solar Cells, **168** (2017) 14-21
Agosto, 2017 | **DOI:** 10.1016/j.solmat.2017.04.013

The present work is focused on thermochemical energy storage (TCES) in Concentrated Solar Power (CSP) plants by means of the Calcium-Looping (CaL) process using cheap, abundant and non-toxic natural carbonate minerals. CaL conditions for CSP storage involve calcination of CaCO3 in the solar receiver at relatively low temperature whereas carbonation of CaO is carried out at high temperature and high CO2 concentration to use the heat of reaction for power production by means of a CO2 closed power cycle. Under these conditions, large CaO particles derived from limestone to be used in industrial processes are rapidly deactivated due to pore plugging, which limits the extent of the reaction. This is favored by the relatively small pores of the CaO skeleton generated by low temperature calcination, the large thickness of the CaCO3 layer built upon the CaO surface and the very fast carbonation kinetics. On the other hand, at CaL conditions for CSP storage does not limit carbonation of CaO derived from dolomite (dolime). Dolime is shown to exhibit a high multicycle conversion regardless of particle size, which is explained by the presence of inert MgO grains that allow the reacting gas to percolate inside the porous particles.

CO_2 capture performance of Ca-Mg acetates at realistic Calcium Looping conditions

Miranda-Pizarro, J; Perejon, A; Valverde, JM; Perez-Maqueda, LA; Sanchez-Jimenez, PE
Fuel, **196** (2017) 497-507
Mayo, 2017 | **DOI:** 10.1016/j.fuel.2017.01.119

The Calcium Looping (CaL) process, based on the cyclic carbonation/calcination of CaO, has emerged in the last years as a potentially low cost technique for CO_2 capture at reduced energy penalty. In the present work, natural limestone and dolomite have been pretreated with diluted acetic acid to obtain Ca and Ca-Mg mixed acetates, whose CO_2 capture performance has been tested at CaL conditions that necessarily imply sorbent regeneration under high CO_2 partial pressure. The CaL multicycle capture performance of these sorbents has been compared with that of CaO directly derived from limestone and dolomite calcination. Results show that acetic acid pretreatment of limestone does not lead to an improvement of its capture capacity, although it allows for a higher calcination efficiency to regenerate CaO at reduced temperatures (similar to 900 degrees C) as compared to natural limestone (>similar to 930 degrees C). On the other hand, if a recarbonation stage is introduced before calcination to reactivate the sorbent, a significantly higher residual capture capacity is obtained for the Ca -Mg mixed acetate derived from dolomite as compared to either natural dolomite or limestone. The main reason for this behavior is the enhancement of carbonation in the solid-state diffusion controlled phase. It is argued that the presence of inert MgO grains in the mixed acetate with reduced segregation notably promotes solid state diffusion of ions across the porous structure created after recarbonation.

Multicycle activity of natural $CaCO_3$ minerals for thermochemical energy storage in Concentrated Solar Power plants

Benitez-Guerrero, M; Valverde, JM; Sanchez-Jimenez, PE; Perejon, A; Perez-Maqueda, LA
Solar Energy, **153** (2017) 188-199
Septiembre, 2017 | **DOI:** 10.1016/j.solener.2017.05.068

Thermochemical energy storage in Concentrated Solar Power plants by means of the Calcium-Looping process is a promising novel technology that would allow for a higher share of renewables. A main benefit of this technology is the use of widely available, non-toxic and environmentally friendly calcium carbonate minerals as raw materials to store energy. Efficient integration of the Calcium-Looping process into Concentrated Solar Power plants involves the endothermic calcination of $CaCO_3$ in the solar receiver while the exothermic carbonation of CaO is carried out at high temperature under high CO_2 partial pressure. The heat released by this reaction is carried out by the excess CO_2 and employed for power generation by means of a closed CO_2 cycle. This work explores the multicycle Calcium-Looping performance of naturally occurring $CaCO_3$ minerals such as limestone, chalk and marble for thermochemical energy storage in Concentrated Solar Power plants. Despite their similar composition (almost pure $CaCO_3$), these minerals exhibit a significant difference in their Calcium-Looping multicycle activity, which may be attributed to differences in particle size and microstructure. Pore plugging at the Calcium-Looping conditions for thermochemical energy storage tested in our work is a main limiting mechanism on the multicycle CaO carbonation activity.

A novel, simple and rapid route to the synthesis of boron cabonitride nanosheets: combustive gaseous unfolding

Jalaly, Maisam; Jose Gotor, Francisco; Semnan, Masih; Jesus Sayagues, Maria
Scientific Reports, **7** (2017) art. 3453
Junio, 2017 | **DOI:** 10.1038/s41598-017-03794-7

The ternary compound boron carbonitride (BCN) was synthesized in the form of few-layer nanosheets through a mechanically induced self-sustaining reaction (MSR). Magnesium was used to reduce boron trioxide in the presence of melamine in a combustive manner. The process to form the nanostructured material was very rapid (less than 40 min). The prepared powder was investigated by various techniques such as X-ray diffraction (XRD), Fourier Transform infrared (FTIR), Micro-Raman spectroscopy, X-ray photoelectron spectroscopy (XPS), high-resolution transmission electron microscopy (HRTEM), and electron energy loss spectroscopy (EELS). The thermal stability and the optical behavior of the BCN nanosheets were also studied by thermal analysis and UV-vis spectroscopy, respectively. The formation mechanism of the nanosheet morphology was described in detail.

Preparation of ytterbium substituted BiFeO3 multiferroics by mechanical activation

Gil-Gonzalez, E; Perejon, A; Sanchez-Jimenez, PE; Hayward, MA; Perez-Maqueda, LA
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Febrero, 2017 | **DOI:** 10.1016/j.jeurceramsoc.2016.09.014

Samples in the system Bi1-xYbxFeO3 (0.02 <= x <= 0.07) have for the first time been prepared by mechanical activation followed by sintering. XRD and DSC measurements show that the solubility limit of ytterbium in the R3c Bi1-xYbxFeO3 system is reached at x similar to 0.03. Higher ytterbium contents lead to a two-phase mixture of a main R3c phase of approximate composition Bi0.97Yb0.03FeO3 and ytterbium enriched secondary phases that cannot be readily indexed or quantified due to their small amount. DSC and temperature-dependent XRD showed that while the magnetic ordering temperature, T-N, was unaffected by Yb substitution, the ferroelectric ordering, T-C, declined. Temperature-dependent XRD patterns show that all samples exhibit rhombohedral R3c to orthorrhombic Pnma phase transitions. Diffuse reflectance spectroscopy suggests the potential use of the samples in photocatalytic applications due to their low band gap energy. Impedance spectroscopy and magnetic measurements show that samples are electrically homogenous and highly insulating, exhibiting antiferromagnetic behaviour at room temperature.

Characterization of mechanosynthesized $Bi_{1-x}Sm_xFeO_3$ samples unencumbered by secondary phases or compositional inhomogeneity

Gil-Gonzalez, E; Perejon, A; Sanchez-Jimenez, PE; Hayward, MA; Criado, JM; Sayagues, MJ; Perez-Maqueda, LA
Journal of Alloys and Compounds, **711** (2017) 541–551
Julio, 2017 | **DOI:** 10.1016/j.jallcom.2017.03.289

High-quality samples, in terms of phase purity and dielectric properties, of composition $Bi_{1-x}Sm_xFeO_3$ (0.05 <= x <= 0.20) have for the first time been prepared by mechanosynthesis. Close inspection of the powder diffraction data, analysis via Rietveld refinement and TEM microscopy demonstrates that the $Bi_{1-x}Sm_xFeO_3$ samples contain only perovskite phases. Additionally, by a combination of Rietveld analysis, TEM, DSC, temperature-dependent XRD and permittivity data a tentative phase diagram has been proposed where the high temperature paraelectric phase Pnma has been confirmed for samarium substituted $BiFeO_3$. Regarding the physical properties, the samples resulted to be electrically homogenous and highly insulating at room temperature, suggesting that other sources of conductivity, such as mixed valence of Fe associated with possible oxygen non-stoichiometry, have been avoided during the samples synthesis. In spite of the high quality of the samples, the dielectric and magnetic behaviour of the $Bi_{1-x}SmxFeO_3$ samples change only modestly on Sm substitution, with neither a great change in the resistivity or remnant magnetisation of Sm substituted samples in comparison with $BiFeO_3$.

Microcalorimetry: A powerful tool for quantitative analysis of aging hardening response of Cu-Ni-Sn alloys

Donoso, E; Dianez, MJ; Perejon, A; Sanchez-Jimenez, PE; Perez-Maqueda, LA; Sayagues, MJ; Criado, JM
Journal of Alloys and Compounds, **694** (2017) 710-714
Enero, 2017 | **DOI:** 10.1016/j.jallcom.2016.10.060

The method for the deconvolution of overlapping DSC peaks here proposed has been used by the first time for the quantitative determination of the enthalpies associated to the phase transitions undergone during the aging of an alloy. They have been determined the enthalpies evolved along the first and the second overlapping DSC traces of Cu-10 wt%Ni-5.5 wt%Sn alloy, which are associated, respectively, to the spinodal decomposition of the alloy and the segregation of a DO22 (CuxNi1-x)(3)Sn tetragonal phase. The fraction of the DO22 phase (responsible of the aging hardening of this alloy) has been successfully determined from DSC as a function of the annealing treatment, while TEM and XRD failed for this purpose. It has been demonstrated that a threshold higher than 50% of crystallization of the DO22 phase is required for achieving a significant increase of the hardness as a function of the crystallization percentage. These results suggest that microcalorimetric measurement can be a powerful tool to establish quantitative relationships between the mechanical, electrical or functional properties of alloys and their structural changes undergone by aging.

Influence of milling parameters on the solid-gas synthesis of TiC_xN_{1-x} by mechanically induced self-sustaining reaction

Chicardi, E; Gotor, FJ; Alcala, MD; Cordoba, JM

Powder Technology, **319** (2017) 12-18

Septiembre, 2017 | **DOI:** 10.1016/j.powtec.2017.06.035

The synthesis of a titanium carbonitride solid solution (TiCxN1 − x) performed in a high-energy planetary mill by a solid-gas Mechanically induced Self-sustaining Reaction (MSR) was used to study the influence of a full set of experimental milling parameters on the ignition time (tig) as a measure of the mechanical dose rate provided by the mill. The highly exothermic Ti-C-N mixture was selected to ensure no competitiveness between MSR and diffusion-controlled routes under the milling conditions employed. The results showed that the dependence of tig on the spinning rate followed a potential function, with a potential factor higher than the value of 3 that would be obtained if a perfect collision model is assumed. The scalability of milling processes performed using planetary mills was confirmed. The results suggested that to define a milling experiment, it is necessary to provide not only the Ball-to-Powder mass Ratio (BPR) and spinning rate values, as is usually performed, but also the full set of milling parameters including the nature of the milling media (vial and balls), the number and size of balls, the mass of the powder charge, the pressure of the reactive gas and even the volume of the vial.

Effect of the impact energy on the chemical homogeneity of a (Ti,Ta,Nb)(C,N) solid solution obtained via a mechanically induced self-sustaining reaction

de La Obra, AG; Gotor, FJ; Chicardi, E

Journal of Alloys and Compounds, **708** (2017) 1008-1017

Junio, 2017 | **DOI:** 10.1016/j.jallcom.2017.03.109

A titanium-tantalum-niobium carbonitride solid solution, (Ti,Ta,Nb)(C,N), was synthesised in a planetary mill via a mechanochemical process that involves a mechanically induced self-sustaining reaction (MSR) from stoichiometric Ti/Ta/Nb/C mixtures that are milled under a nitrogen atmosphere. The influence of the spinning rate of the planetary mill, which determines the impact energy of the milling process, on the ignition time (t(ig)) of the MSR process as well as the chemical homogeneity of the final product was analysed. The results indicated that the dependence of tig on the spinning rate followed a potential function with a potential factor of 4.85, implying a remarkable reduction in the milling time required to induce the self-sustaining reaction at increasing spinning rates (i.e., from 4200 min at 200 rpm to 15 min at 800 rpm). However, the chemical and structural characterisation of the obtained products at ignition without any extra milling treatment indicated that a single solid solution phase was only obtained at the lowest spinning rates (i.e., less than 300 rpm). At increasing rates, the relative amount of the intended solid solution phase continuously decreased, and new undesirable secondary phases were formed. Despite the long milling times required for the milling experiments that were performed at the slowest spinning rates, iron contamination from the milling media was negligible due to the low intensity milling regime.

Mechanochemical Solvent-Free Synthesis of Quaternary Semiconductor Cu-Fe-Sn-S Nanocrystals

Balaz, Peter; Balaz, Matej; Sayagues, Maria J.; Skorvanek, Ivan; Zorkovska, Anna; Dutkova, Erika; Briancin, Jaroslav; Kovac, Jaroslav; Kovac, Jaroslav, Jr.; Shpotyuk, Yaroslav

Nanoscale Research Letters, **12** (2017) art. 256

Abril, 2017 | **DOI:** 10.1186/s11671-017-2029-5

In this study, we demonstrate a one-pot mechanochemical synthesis of the nanocomposite composed of stannite Cu_2FeSnS_4 and rhodostannite Cu2FeSn3S8 nanocrystals using a planetary ball mill and elemental precursors (Cu, Fe, Sn, S). By this approach, unique nanostructures with interesting properties can be obtained. Methods of XRD, Raman spectroscopy, UV-Vis, nitrogen adsorption, SEM, EDX, HRTEM, STEM, and SQUID magnetometry were applied. Quaternary tetragonal phases of stannite and rhodostannite with crystallite sizes 18-19 nm were obtained. The dominant Raman peaks corresponding to the tetragonal stannite structure corresponding to A-symmetry optical modes were identified in the spectra. The bandgap 1.25 eV calculated from UV-Vis absorption spectrum is very well-acceptable value for the application of the synthesized material. The SEM micrographs illustrate the clusters of particles in micron and submicron range. The formation of agglomerates is also illustrated on the TEM micrographs. Weak ferromagnetic properties of the synthesized nanocrystals were documented.

Effect of Thermal Pretreatment and Nanosilica Addition on Limestone Performance at Calcium-Looping Conditions for Thermochemical Energy Storage of Concentrated Solar Power

Valverde, Jose Manuel; Barea-Lopez, Manuel; Perejon, Antonio; Sanchez-Jimenez, Pedro E.; Perez-Maqueda, Luis A.

Energy & Fuels, **31** (2017) 4226-4236

Abril, 2017 | **DOI:** 10.1021/acs.energyfuels.6b03364

The share of renewable energies is growing rapidly, partly in response to the urgent need for mitigating CO_2 emissions from fossil fuel power plants. However, cheap and efficient large-scale energy storage technologies are not yet available to allow for a significant penetration of renewable energies into the grid. Recently, a potentially low-cost and efficient thermochemical energy storage (TCES) system has been proposed, based on the integration of the calcium-looping (CaL) process into concentrated solar power plants (CSPs). The CaL process relies on the multicycle carbonation/calcination of CaO, which can be derived from calcination of widely available, cheap, and nontoxic natural limestone ($CaCO_3$). This work explores the effect on the multicycle activity of limestone-derived CaO of thermal pretreatment under diverse atmospheres and the addition of nanosilica, which would be expected to hinder CaO grain sintering. Importantly, optimum CaL conditions for CSP energy storage differ radically from those used in the application of the CaL process for CO_2 capture. Thus, calcination should be ideally carried out under low CO_2 partial pressure at moderate temperature (below 750 degrees C), whereas CO_2 concentration and temperature should be high for carbonation in order to maximize thermoelectric efficiency. When limestone is subjected to carbonation/calcination cycles at these conditions, its performance is critically dependent on the type of pretreatment.

Our results indicate that the multicycle CaO activity is correlated with the size of the particles and the CaO pore size distribution. Thus, CaO activity is impaired as particle size is increased and/or CaO pore size is decreased. These observations suggest that pore plugging poses a main limitation to the multicycle performance of limestone-derived CaO at the optimum CaL conditions for TCES in CSPs, which is supported by scanning electron microscopy analysis. Strategies to enhance the performance of natural limestone at these conditions should be therefore oriented toward minimizing pore plugging rather than CaO grain sintering, which stands as the main limitation at CaL conditions for CO_2 capture.

Flash sintering of highly insulating nanostructured phase-pure BiFeO3

Perez-Maqueda, LA; Gil-Gonzalez, E; Perejon, A; Lebrun, JM; Sanchez-Jimenez, PE; Raj, R
Journal of the American Ceramic Society, **100** (2017) 3365-3369
Agosto, 2017 | **DOI:** 10.1111/jace.14990

We show that BiFeO3, that is electrically homogeneous, is a good insulator, and has a low dielectric constant (the properties desired in its applications), can be produced by flash sintering, which is nominally difficult to achieve by conventional and spark plasma sintering processes. The flash-sintered specimens had a uniform microstructure with a nanometric grain size of similar to 20 nm.

A Promising approach to the kinetics of crystallization processes: The sample controlled thermal analysis

Perejon, A; Sanchez-Jimenez, PE; Criado, JM; Perez-Maqueda, LA
Journal of the American Ceramic Society, **100** (2017) 1125-1133
Marzo, 2017 | **DOI:** 10.1111/jace.14604

Constant Rate Thermal Analysis (CRTA) method implies controlling the temperature in such a way that the reaction rate is maintained constant all over the process. This method allows determining simultaneously both the kinetic parameters and the kinetic model from a single experiment as the shape of the CRTA -T curves strongly depends on the kinetic model. CRTA method has been developed in the market only for thermogravimetric and thermodilatometric systems and, therefore, its use has been limited until now to the kinetic study of processes involving changes in mass or size of the samples, respectively. To overcome this obstacle, a method has been developed in this work for using the DSC signal for controlling the process rate in such a way that CRTA would be applied to the kinetic analysis of either phase transformations or crystallizations. The advantages of CRTA for performing the kinetics of crystallization processes have been here successfully demonstrated for the first time after selecting the crystallization of zirconia gel as test reaction.

Microstructure characterization of multifunctional As_4S_4/Fe_3O_4 nanocomposites prepared by high-energy mechanical milling

Shpotyuk, O; Bujnakova, Z; Sayagues, MJ; Balaz, P; Ingram, A; Shpotyuk, Y; Demchenko, P
Materials Characterization, **132** (2017) 303-311
Octubre, 2017 | **DOI:** 10.1016/j.matchar.2017.08.028

Multifunctional As_4S_4/Fe_3O_4 nanocomposites prepared by high-energy mechanical milling are probed by complementary methods of positron annihilation lifetime (PAL) spectroscopy, supported by microstructure characterization using X-ray powder diffraction (XRPD) with analysis applied to the first sharp diffraction peak (FSDP), morphology studies by transmission electron microscopy (TEM) with energy-dispersive spectroscopy (EDS) and high-resolution TEM (HRTEM).

These nanocomposites are shown to consist of Fe_3O_4 crystallites with particle sizes of 8-21 nm, and far separated beta-As_4S_4 crystallites surrounded by amorphous As-S phase. In respect to PAL data, the effect of milling is identified as possible conversion from Ps traps to positron-trapping sites depending on preferential chemistry of atomic surrounding. So, the interfacial triple junctions at the intersections of Fe_3O_4 crystallites are identified as principal positron traps in As_4S_4/Fe_3O_4 nanocomposites with competitive influence from free-volume defects of amorphous As-S phase.

Analytical investigation of Mudejar polychrome on the carpentry in the Casa de Pilatos palace in Seville using non-destructive XRF and complementary techniques

Garrote, MA; Robador, MD; Perez-Rodriguez, JL
Spectrochimica Acta Part A-Molecular and Biomolecular Spectroscopy, **173** (2017) 279–291
Enero, 2017 | **DOI:** 10.1016/j.saa.2016.09.027

The pigments, execution technique and repainting used on the polychrome wood ceilings and doors in the Casa de Pilatos (Seville, Spain) were studied using portable X-ray fluorescence equipment. Cross-sections of small samples were also analysed by optical microscopy, SEM with EDX analysis, micro-Raman and micro-infrared spectroscopy and X-ray diffraction. These carpentry works are magnificent examples of the Mudejar art made in Spain in the early 16th century. Portable X-ray fluorescence gave good information on the different components of the polychrome. The SEM-EDX study of the surfaces of small samples gave information on their components and also characterized the compounds that had been deposited or formed by environmental contamination or by the alteration of some pigments. The SEM-EDX study of cross-sections facilitated the characterization of all layers and pigments from the support to the most external layer. The following pigments were characterized: red (cinnabar/vermillion, lead oxide, iron oxides and orpiment/realgar), black (carbon black), white (white lead and titanium barium white), yellow-orange-red-brown (orpiment/realgar and iron oxides), green (chromium oxide), blue (indigo blue and ultramarineblue), and gilding (gold leaf on bole). False gold, bronze and brass were also found. The pigments were applied with the oil painting technique over a support layer that had been primed with animal glue. This support layer was gypsum in some cases and white lead in others. This study is essential to the polychrome conservation of the studied artwork, and it will help clarify uncertainties in the history and painting of Mudejar art.

Structure evolution in the $LaMn_{1-x}Fe_xO_{3+\delta}$ system by Rietveld analysis

Cordoba, J. M.; Ponce, M.; Sayagues, M. J.
Solid State Ionics, **303** (2017) 132-137
Mayo, 2017 | **DOI:** 10.1016/j.ssi.2017.02.020

The synthesis of $LaMn_{1-x}Fe_xO_{3+\delta}$ ($0 \le x \le 1$) solid solutions perovskite powder was carried out using high-energy milling from the constituent oxides, and further crystallization by high temperature treatment. The compositions of the crystalline phases as a function of x were determined by X-ray powder diffraction using a Rietveld refinement. The relationship between composition and structure was covered. This showed that $LaMn_{1-x}Fe_xO_{3+\delta}$ exists with the rhombohedral structure (R-3c, 167) only below x = 0.3 and with the orthorhombic structure (Pnma, 62) over x = 0.7. The rhombohedral phase coexists with the orthorhombic phase between $0.4 < x < 0.6$.

A new family of cermets: Chemically complex but microstructurally simple

de la Obra, AG; Aviles, MA; Torres, Y; Chicardi, E; Gotor, FJ

International Journal of Refractory Metals & Hard Materials, **63** (2017) 17-25

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Cermets based on Ti(C,N) have interesting properties, such as high wear resistance, high chemical stability and good mechanical strength at high temperature, but to become a viable alternative to cemented carbides, the fracture toughness and damage tolerance must be significantly improved. Complete solid-solution cermets (CSCs) have been proposed to further improve the mechanical properties of these materials. However, to develop this family of cermets with a high level of quality and reliability, using pre-fabricated complex carbonitrides is necessary instead of unalloyed mixtures as the raw ceramic material. A mechanochemical process called mechanically induced self-sustaining reaction (MSR) is suitable to obtain these complex carbonitrides with high stoichiometric control. On the other hand, high entropy alloys (HEAs), which can also be obtained by mechanochemical processes, are a good candidate to replace the current binder phase in cermets because they exhibit high strength and ductility at high temperature and good resistance to both wear and corrosion. In this work, a new family of CSCs based on (Ti,Ta,Nb)CxN1-x, with HEAs belonging to the Fe-Co-Ni-Cr-Mn-V system as the binder phase is developed by mechanochemical processes. With only two constituent phases, these cermets have a simple microstructure but a high compositional complexity because both the ceramic and binder phases are complex solid solutions with at least five components.

New findings on thermal degradation properties of fluoropolymers

Liu, SE; Zhou, WL; Yan, QL; Qi, XF; An, T; Perez-Maqueda, LA; Zhao, FQ

Journal of Thermal Analysis and Calorimetry, **128** (2017) 675-685

Mayo, 2017 | **DOI:** 10.1007/s10973-016-5963-z

In this paper, the thermal degradation properties of Viton A and Fluorel are investigated by both isoconversional and combined kinetic analysis methods using non-isothermal thermogravimetry technique. It has been found that the heating rate has little affect on the degradation residue of Fluorel and Viton A, where around 1.3% char was formed for Fluorel and 3.5% for Viton A. Different from the literature, the decomposition of Viton A should be considered as an overlapped dehydrofluorination and carbon chain scission process, with activation energy of 214 +/- 11 and 268 +/- 13 kJ mol(-1), respectively. The effect of dehydrofluorination on degradation of Fluorel is not so significant due to low content of H, and hence, it could be considered as a single-step mechanism with average activation energy of 264 +/- 14 kJ mol(-1). The thermal stability of Fluorel is much better than that of Viton A, and the predicted half-life is around 218 min for Fluorel and 49 min for Viton A at 420 A degrees C, which are consistent with experimental values. If using a single-step model as in the literature for Viton A, its half-life at 420 A degrees C would be underestimated for > 20%.

Thermal study of residues from greenhouse crops plant biomass

Morales, Laura; Garzón, Eduardo; Martínez-Blanes José María; Sánchez-Soto, Pedro José
Journal of Thermal Analysis and Calorimetry, **129** (2017) 1111-1120
Agosto, 2017 | **DOI:** 10.1007/s10973-017-6243-2

The principal aim of this work is to examine the effect of thermal treatments using a muffle furnace (static heating) and by simultaneous TG/DTA (dynamic heating) on selected greenhouse crops plant biomass investigated here as the first time. The effect of fractionation by sieving (<25 and <2.5 mm), preheating at 150 °C for 48 h and leaching with water on the thermal behavior has been studied. The observation of similar profiles of mass variation corresponding to several samples heated in air up to 1150 °C allows to conclude that particle size did not influence the thermal evolution, but the effect of heating cycle is evidenced. Thermal analysis in air of a representative sample showed the several mass variation steps and DTA exothermic effects produced by the complex thermal decomposition and pyrolysis of the organic matter. Elemental analysis (CHNS and O) of the starting samples and thermally treated revealed the effect of the temperature, with formation of ashes with lower C content from 44.37 to 0.70 mass% as a minimum after elimination of organic matter by heating. Leaching increased the thermal mass variation as an effect of elimination of water-soluble components. According to the present results, the size fractionation of the greenhouse crops biomass did not influence the results of elemental composition. The present study has provided results of interest concerning this biomass source of renewable energy originated by the remains of tomato (Solanum lycopersicum L.), being estimated the highest of all the biomass produced by the greenhouse crops agricultural industry in Almería (SE Spain).

Non-isothermal Characterization of the Precipitation Hardening of a Cu-11Ni-19Zn-1Sn Alloy

Donoso, E; Dianez, MJ; Criado, JM; Espinoza, R; Mosquera, E
Metallurgical and Materials Transactions A-Physical Metallurgy and Materials Science, **48A** (2017) 3090-3095
Junio, 2017 | **DOI:** 10.1007/s11661-017-4063-4

The precipitation hardening of a Cu-11Ni-19Zn-1Sn alloy has been studied by means of Differential Scanning Calorimetry (DSC), High-Resolution Transmission Electron Microscopy (HRTEM), and hardness measurements. The calorimetric curves, in the range of temperatures analyzed, show the presence of one exothermic reaction followed by an endothermic one. The exothermic DSC peak is due to the segregation of Cu2NiZn precipitates and it is associated to a noticeable improvement of the mechanical properties of the alloy. The endothermic effect is associated to the dissolution of the Cu2NiZn precipitates into the copper matrix for restoring the starting Cu-11Ni-19Zn-1Sn homogeneous solid solution. The reaction mechanisms of these processes have been proposed from the kinetic analysis of the exothermic and endothermic DSC signals. The results obtained point out that tin plays a decisive role on the precipitation hardening of the alloy, because age hardening is not observed in the case of a Cu-Ni-Zn ternary alloy of similar composition.

Chalcogenide Quaternary Cu_2FeSnS_4 Nanocrystals for Solar Cells: Explosive Character of Mechanochemical Synthesis and Environmental Challenge

Balaz, P.; Balaz, M.; Sayagues, M.J.; Eliyas, A.; Kostova, N.G.; Kanuchova, M.; Dutkova, E.; Zorkovska, A.
Crystal, **7** (2017) art. 367
Diciembre, 2017 | **DOI:** 10.3390/cryst7120367

In this study we demonstrate the synthesis of quaternary semiconductor nanocrystals of stannite Cu_2FeSnS_4/rhodostannite $Cu_2FeSn_3S_8$ (CFTS) via mechanochemical route using Cu, Fe, Sn and S elements as precursors in one-pot experiments. Methods of X-ray diffraction (XRD), nitrogen adsorption, high-resolution transmission electron microscopy (HRTEM), scanning transmission electron microscopy (STEM), energy-dispersive X-ray spectroscopy (EDX) and X-ray photoelectron spectroscopy (XPS) were applied to characterize properties of the unique nanostructures. Mechanochemical route of synthesis induced new phenomena like explosive character of reaction, where three stages could be identified and the formation of nanostructures 5-10 nm in size. By using XPS method, Cu(I), Fe(II), Sn(IV) and S(-II) species were identified on the surface of CFTS. The value of optical band gap 1.27 eV is optimal for semiconductors applicable as absorbers in solar cells. The significant photocatalytic activity of the CFTS nanocrystals was also evidenced. The obtained results confirm the excellent properties of the quaternary semiconductor nanocrystals synthesized from earth-abundant elements.

The epigraphic stela of Montoro (Cordoba): the earliest monumental script in Iberia?

Sanjuan, LG; Diaz-Guardamino, M; Wheatley, DW; Barra, JPV; Rodriguez, JAL; Rogerio-Candelera, MA; Erbez, AJ; Barker, D; Strutt, K; Ariza, MC
Antiquity, **91** (2017) 946-932
Agosto, 2017 | **DOI:** 10.15184/aqy.2017.86

A remarkable stela from Montoro, southern Spain, is unique in its morphology, epigraphic traits and landscape context. A programme of chemical characterisation, digital imaging, and geo-lithological and epigraphic analyses were conducted to determine its age and significance, and the results were integrated with data from archaeological investigations of the surrounding area. This multi-faceted approach allowed the stela to be interpreted within the context of early interactions between literate Mediterranean societies of the Late Bronze Age and Iron Age and non-literate Iberian societies. A key outcome of this research is a wider understanding of the complex patterns in the use and perception of early scripts.

Characterization of ashes from greenhouse crops plant biomass residues using X-ray fluorescence analysis and X-ray diffraction

Garzon, E; Morales, L; Martinez-Blanes, JM; Sanchez-Soto, PJ
X-ray spectrometry, **46** (2017) 569-578
Noviembre, 2017 | **DOI:** 10.1002/xrs.2801

A characterization of ashes obtained by thermal treatments on greenhouse crops plant biomass residues is presented. The chemical analysis, by X-ray fluorescence (wavelength-dispersive X-ray fluorescence), and phase analysis, by X-ray diffraction, of the resultant ashes are reported. Thermal treatments of selected samples of these residues increase the relative amounts of inorganic Mg, Si, P, and S in the ashes, being these amounts as high as increasing temperature. As an opposite effect, Na, Cl, and K contents decrease as increasing temperature by a volatilization process of the chlorides, as confirmed by X-ray diffraction. The crystalline phase analysis of the ashes demonstrates the formation of inorganic constituents of the biomass, including alkaline chlorides and calcium salts (calcite, anhydrite, and apatite). Progressive thermal treatments induce the formation of new silicate phases (akermanite and grossularite) and silica (-quartz and cristobalite). Furthermore, the particle size of the starting biomass samples does not influence the evolution of the crystalline phases by thermal treatments. In contrast, a previous leaching using water and subsequent heating at 1,000 degrees C produces the formation of periclase (MgO), lime (CaO), and the silicate gehlenite, without the presence of anhydrite. This study is interesting for future investigations on the residues as a profitable biomass source for energy production and sustainable large-scale management. Some potential applications of the resultant ashes can be proposed.

Synthesis, Characterisation, and Photocatalytic Behaviour of Mesoporous ZnS Nanoparticles Prepared Using By-Product Templating

Emrooz, HBM; Rahmani, AR; Gotor, FJ
Australian Journal of Chemistry, **70** (2017) 1099-1105
Mayo, 2017 | **DOI:** 10.1071/CH17192

High surface area mesoporous ZnS nanoparticles (MZN) were obtained with the aid of the by-product of the synthesising reaction. This by-product, namely NaNO3, can be considered as a soft template responsible for the formation of pores. Ethanol and water were chosen as the synthesis media. Ultrasonic waves were used as an accelerator for the synthesis of MZNs. Photocatalytic activities of the synthesised samples for the degradation of methylene blue (MB) were investigated under ultraviolet irradiation. Synthesised specimens were characterised using field emission scanning electron microscopy, transmission electron microscopy, powder X-ray diffraction, diffuse reflectance spectroscopy, N-2-physisorption, and FT-IR spectroscopy. Results indicated that the synthesis media has a pronounced effect on the surface properties of the final porous particles by several mechanisms. The specific surface area of the MZN samples synthesised in water and ethanol were determined to be 53 and 201m(2)g(-1), respectively. The difference in the specific surface area was attributed to the weak solvation of S2- ions (Na(2)S5H(2)O in ethanol) and also to the by-product of the synthesis reaction. The photocatalytic behaviour of the mesoporous ZnS nanoparticles synthesised in these two media were investigated and the results have been interpreted with the aid of effective surface area, pore volume, and bandgap energy of the specimens.

Simultaneous adsorption and photocatalytic behavior of hybrid mesoporous ZnS-SiO_2 nanocomposite

Emrooz, HBM; Gotor, FJ

Materials Research Express, **4** (2017) art. 085037

Agosto, 2017 | **DOI:** 10.1088/2053-1591/aa85cf

Mesoporous ZnS-SiO_2 nanocomposite was synthesized with a facile process. At first a large pore volume (1.86 cm(3).g(-1)), moderate pore size (about 12.8 nm) and moderate surface area (586 m(2).g(-1)) mesoporous SiO_2 was synthesized in an acidic PH using cationic surfactant. ZnS nanoparticles were infiltrated in the porosities of the synthesized SiO_2, with a room temperature post grafting method. The synthesized particles have been characterized with transmission electron microscopy (TEM), x-ray diffraction (XRD), x-ray photoelectron spectroscopy (XPS), Brunauer-Emmett-Teller (BET), fourier transformation infrared spectroscopy (FTIR) and diffuse reflectance spectroscopy (DRS). Results confirm the mesoporous characteristics of ZnS-SiO_2 nanocomposite with specific surface area as high as 248 m(2).g(-1), pore volume of 0.89 cm(3).g(-1) and average pore diameter of about 14.4 nm. Simultaneous adsorption-photocatalytic behavior of this hybrid mesoporous nanocomposite for degradation of methylene blue (MB) was investigated. The efficiency of this material was compared with that of mesoporous SiO_2 and also lone ZnS nanoparticles. Results showed that by using ultraviolet irradiation, ZnS-SiO_2 can degrade greater than 85% of MB only after 3 min. The case for lone ZnS is less than 5% after 30 min. Degradation mechanism of MB using ZnS-SiO_2 and ultraviolet radiation was explained with simultaneous adsorption and photocatalytic phenomena. Ultraviolet irradiation can degrade adsorbed MB of mesoporous SiO_2 which can prevent it from saturation.

Structural and Chemical Characteristics of Sisal Fiber and Its Components: Effect of Washing and Grinding

Benitez-Guerrero, M; Perez-Maqueda, LA; Artiaga, R; Sanchez-Jimenez, PE; Pascual-Cosp, J

Journal of Natural Fibers, **14** (2017) 26-39

Enero, 2017 | **DOI:** 10.1080/15440478.2015.1137529

This work covers the study of microstructural changes of natural sisal fibers induced by different conditioning pre-treatments: mechanical grinding, cryogenic grinding, and hot waterwashing. The aim of the work is to clarify the effects of the pre-treatments on crystallinity and infrared spectra of sisal. Scanning electron microscopy results allowed to identify morphological changes on the fiber surface. Deeper changes of chemical origin were studied by attenuated total reflectance/Fourier transform infrared spectroscopy (FTIR) and focused on the main components of cellular walls: cellulose, lignin, and xylan. The work was complemented with crystallinity index (I-c) data determined by two very different methods: the widely used for lignocellulosic fibers Segal equation based on X-ray diffraction measurements, and the other based on FTIR through the 1430/900 cm(-1) band intensity ratio, which is mostly used with cellulosic samples.

Processing and characterization of surrogate nuclear materials with controlled radial porosity

Torres, Y; Garcia-Ostos, C; Arevalo, C; Gotor, FJ; Pavon, JJ; Trueba, P; Rodriguez-Ortiz, JA

Journal of Nuclear Science and Technology, **54** (2017) 167-173

Febrero, 2017 | **DOI:** 10.1080/00223131.2016.1222918

Irradiated fuel pellets present radial gradient porosity. CeO_2 has been proven as a surrogate material to understand irradiated mixed oxide (MOX) due to its similar structural and mechanical properties. A novel compaction device was developed to produce CeO_2 cylindrical pellets with controlled radial porosity. Three blends of CeO_2 with different binder contents (0.5, 3 and 7.5 vol.% of ethylene-bis-stearamide, EBS) were prepared and used to obtain three different porosities for the core, intermediate and outer rings of pellets, respectively. Different compaction pressures were employed in each region to get the intended porosities. The whole pellet was subjected to a heating rate up to 500 degrees C to remove the EBS binder. Finally, a pressureless sintering step was performed at 1700 degrees C for 4 h. A microstructural characterization was performed in the three areas, including grain size and porosity. Mechanical properties like hardness, fracture toughness and tribo-mechanical response, as scratch resistance, were also determined. Pellets fabricated from this device have shown microstructural and mechanical properties with a good correlation to those of irradiated nuclear fuel.

Mechanochemically Synthesized $CuFeSe_2$ Nanoparticles and Their Properties

Dutkova, E; Skorvanek, I; Sayagues, MJ; Zorkovska, A; Kovac, J; Balaz, P

Acta Physica Polonica A, **131** (2017) 1156-1158

Abril, 2017 | **DOI:** 10.12693/APhysPolA.131.1156

The mechanochemical synthesis of nanocrystalline $CuFeSe_2$ particles prepared by high-energy milling in a planetary mill in an argon atmosphere from copper, iron, and selenium for 60 min is reported for the first time. The $CuFeSe_2$ nanoparticles crystallize in tetragonal structure with mean crystallite size of about 32 +/- 1 nm. High resolution transmission electron microscopy measurements confirmed the presence of agglomerates which are formed by small nanocrystalline domains (5-40 nm). The magnetic data revealed that paramagnetic $CuFeSe_2$ nanoparticles coexist with a small amount of ferromagnetic impurities at room temperature. The magnetic transition towards a weak ferromagnetic or ferrimagnetic behavior occurs in $CuFeSe_2$ at approximately 79 K. The band gap of the $CuFeSe_2$ particles is 0.95 eV which is wider than the band gap in bulk materials (0.16 eV), which could be in many aspects of application more beneficial.

Modellization of the process of natural aging of a polymer material applied for greenhouse covers

Garzón, E; Ortiz, I. Ma; Castillo, J; Rojano, A; Sánchez-Soto, P. J

Revista Latinoamericana de Metalurgia y Materiales, **60-82** (2017) 60-82

It is presented a procedure to determine the aging of greenhouse cover materials constituted by a polymer material. For this purpose, it is performed a continued monitoring of the photosynthetically active radiation (PAR) and the global radiation during three campaigns. It has been studied in this research samples taken at different zones of the cover, after breakage using mechanical traction. It is deduced a useful life of the polymer of two years, being the average transmitted PAR of 37.44%. However, the absorbed global radiation is determined a value of 36.29%. Furthermore, the elongation to the breakage is above of 500%. This high value is associated to an intense degradation of the polymer material. At the same time, the zone where the sample was taken seems to influence both the strength of mechanical traction and the elongation at the fluence point. The designed tests have been useful to develop a modellization procedure based on the minimum squared method for the adjustment of the data. The proposed model can be used for the design of greenhouse covers and thus allows predict the degree of deterioration of the polymer material at every moment. It is also interesting to keep watch of the need of removing it. The numerical simulations using the finite elements method can represent adequately the mechanical behavior of the cover material. It is been also deduced in this research that the behavior of the covers is at the plastic zone of material.

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

Instituto de Cerámica y Vidrio. 50 años de investigación en Ciencia y Tecnología (Reseña bibliográfica)
P.J. Sánchez-Soto
Revista LLULL, 40 (2017) 306-309

LIBROS Y OTRAS PUBLICACIONES / BOOKS AND OTHER PUBLICATIONS

Guadua angustifolia as a Structural Material for Greenhouse Design
Garzón, E.; Cano, M; Pérez-Villarejo, L.; Martínez, S.; Sánchez Soto, P.J.
"Project Management and Engineering Research", serie: Lecture Notes in Management and Industrial Engineering, eds. J.L. Ayuso, J.L. Yagüe, S. F. Capuz-Rizo; Springer International Publishing AG, págs. 201-213

Ecocemento sostenible con el medio ambiente como visión de futuro
Martínez, S.; Pérez-Villarejo, L.; Eliche-Quesada, D.; Bonet, E.; Sánchez-Soto, P.J.
Medio Ambiente y Sociedad. Cuadernos de didáctica, eds. M.M. Jordán y F. Pardo, Editorial Club Universitario, págs. 243-253

La estabilización de taludes de tierras de cultivo: aplicación de la bioingeniería como herramienta medioambiental
Martínez, S.; Pérez-Villarejo, L.; Eliche-Quesada, D.; Bonet, E.; Sánchez-Soto, P.J.
Medio Ambiente y Sociedad. Cuadernos de didáctica, eds. M.M. Jordán y F. Pardo, Editorial Club Universitario, págs. 233-242

Caracterización de la materia inorgánica de residuos vegetales de invernadero, con interés para la valorización de biomasa, sometidos a tratamiento térmico
Martínez-Blanes, J.Mª; Morales, L.; Garzón, E.; Sánchez-Soto, P.J.
Medio Ambiente y Sociedad. Cuadernos de didáctica, eds. M.M. Jordán y F. Pardo, Editorial Club Universitario, págs. 107-127

PATENTES / PATENTS

Composición para el tratamiento químico sistemático de la verruga plantar, Patente Española

Inventores: Juárez Jiménez, J.Mª; Romero Prieto, M.; Córdoba Fernández, A.; Rayo Rosado, R.; Montaño Jiménez, P.; Avilés Escaño, M.A.; Sánchez-Soto, P.J.
Tipo de Patente: Nacional
Número de Solicitud: ES 2614779 A1
Fecha Solicitud: 1 de junio de 2017
Entidad Titular: Universidad de Sevilla y Consejo Superior de Investigaciones Científicas

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

Inventores: Cano García, M.; Garzón Garzón, E.; Pulido Calvo, I.; Sánchez-Soto, P.J.
Tipo de Patente: Nacional
Número de Solicitud: ES 2593952 B2
Fecha Solicitud: 7 de septiembre de 2017
Entidad Titular: Universidad de Almeria y Consejo Superior de Investigaciones Científicas

Procedimiento de preparación de un mortero impermeabilizante que emplea filitas, producto obtenido y utilización

Inventores: Sánchez-Soto, P.J.; Gotor Martínez F.J.; Fernández Escobar, N.; Garzón Garzón E.
Tipo de Patente: Nacional
Número de Solicitud: ES 2582672 R1
Fecha Solicitud: 2 de enero de 2017
Entidad Titular: Universidad de Almeria y Consejo Superior de Investigaciones Científicas

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

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

Vitrification and Geopolymerization of Wastes for Immobilization or Recycling | VITROGEOWASTES
14 – 15 Septiembre [Elche, España]
Pedro José Sánchez Soto [Miembro del Comité Organizador)

COMUNICACIONES / COMMUNICATIONS

VI Congreso Internacional de Educación Artística y Visual
20 – 22 abril [Badajoz, España]

Educación artística para la formación de futuros profesionales: actividades y colaboraciones de la escuela "Della Robbia" de Artes y Artesanías de Andalucía de Gelves (Sevilla)
Lupión Alvarez J.J.; Durán-Domínguez, G.; Sánchez-Soto, P.J.
Poster

Educación en Arte y Patrimonio Cultural. El examen científico de microestratigrafías como herramienta formativa en Educación Artística y Formación de especialistas en Patrimomio Cultural
Kriznar A.; Durán-Domínguez G.; Sánchez-Soto P.J.
Poster

24th International Symposium on Metastable, Amorphous and Nanostructured Materials
18 – 23 junio [San Sebastian, España]

Chemical nature changes on sintered ball milled synthesized FeCoNiCrMn(Al) HEA phases
M.D. Alcalá; C. Real; I. Fombella; I. Trigo; J.M. Córdoba
Poster

First attempt for a new class of hard metals. WC-FeCoNiCrMn composites
I.L . Velo; F.J. Gotor; M.D. Alcalá; C. Real; J.M. Córdoba
Poster

Future and Prospect for Nanostructured High Entropy Alloys. Aerospace and Biomedical Applications
I.L. Velo; C. Real; M.D. Alcalá; M.A. Avilés; J.M. Córdoba
Poster

21st International Conference on Project Management and Engineering

12 – 24 julio [Cádiz, España]

Mortar with impermeabilizing properties using phyllites
Garzón Garzón E.; Fernández Escobar N.; Sánchez-Soto P.J.
Poster

Processing of ashes from treatment of greenhouse crops wastes for soil stabilization in linear works
Morales L.; Garzón Garzón E.; Sánchez-Soto P.J.
Poster

Application of Vetiveria Zizanioides to diminish the erosion in Guatemala
González Miranda F.M.; Garzón Garzón E.; Reca J.; Sánchez-Soto P.J.
Poster

4th International Conference on Titanium Powder Metallurgy & Additive Manufacturing

8 – 10 septiembre [Xi'an, R.P. China]

Effects of the thermal treatment of Titanium samples fabricated by selective laser melting
J. E. González; A. M. Beltrán; I. Montelaegre; J. Negrín; E. Peón; F. J. Gotor; P. Trueba; J. A. Rodríguez-Ortiz; A. Fernández; Y. Torres
Poster

6th International Conference on Semiconductor Photochemistry

11 - 14 septiembre [Oldenburg, Alemania]

Outstanding visible photocatalytic activity of a new mixed Bismuth Titanate material
M.C. Hidalgo; P. Zambrano; M.J. Sayagués; J.A. Navío
Poster

Vitrification and Geopolimerization of Wastes for Immobilization or Recycling | VITROGEOWASTES

14 - 15 septiembre [Elche, España]

Biomass bottom ash and aluminium industry slags-based polymers
Pérez-Villarejo L.; Bonet E.; Eliche-Quesada D.; Sánchez-Soto P.J.; Rincón J.M.; Castro-Galiano E.
Poster

Aluminium dust ashes as raw materials in ceramic industry
Pérez-Villarejo L.; Bonet-Martínez E.; Martínez-Martínez S.; Sánchez-Soto P.J.; Eliche-Quesada D.; Carrasco-Hurtado B.; Castro-Galiano E.
Poster

Effect of temperature on the clinkerization of low energy belite cements using by-products from the ceramic industry as raw material
Martínez-Martínez S.; Eliche-Quesada D.; Pérez-Villarejo L.; Bonet E.; Sánchez-Soto P.J.
Poster

Manufacturing new ceramic materials from clay and chamotte derived form the structural ceramic industry
Martínez-Martínez S.; Pérez-Villarejo L.; Eliche-Quesada D.; Sánchez-Soto P.J.
Poster

Mullite and mullite-based ceramic composites from kaolinite and sericite clays, wastes and by-products of mining
Sánchez-Soto P.J.; Eliche-Quesada D.; Martínez-Martínez S.; Garzón E.; Carrasco-Hurtado B.; Pérez-Villarejo L.; Rincón J. Ma.
Poster

Vitrification of mining wastes containing the magnesium hydrous phyllosilicate mineral vermiculite with derived lithium glass-ceramics
Callejas P.; Sánchez-Soto P.J.; Rincón J. Ma.; Jordán M.M.
Poster

Sintering parameters optimization during inertization bauxite tailing in ceramic products
Martínez-Martínez S.; Eliche-Quesada D.; Pérez-Villarejo L.; Bonet E.; Sánchez-Soto P.J.; Rincón López J. Ma.
Poster

Assessment of electric Steel black slags for the manufacture of ceramic materials
Bonet E.; Martínez S.; Pérez-Villarejo L.; Carrasco B.; Sánchez-Soto P.J.; Castro E.
Poster

21st International Congress on Project Management and Engineering
12 - 24 julio [Cádiz, España]

Application of Vetiveria Zizanoides to diminish the erosion in Guatemala
González Miranda F.M.; Garzón Garzón E.; Reca J.; Sánchez-Soto P.J.
Comunicación oral

10th International Conference on Porous Metals and Metallic Foams
14 - 17 septiembre [Nanjing, China]

Thermo-chemical treatment of porous Ti6Al4V obtained by selective laser melting
J. E. González; J. Negrín; A. M. Beltrán; I. Montelaegre; E. Peón; P. Sarría; S. Muñoz; J. A. Rodríguez-Ortiz; F. J. Gotor; Y. Torres
Poster

III Foro Iberoamericano de Divulgación y Cultura Científica

23 - 25 noviembre [Córdoba, España]

Acciones para la divulgación científica y tecnológica: Fomento de relaciones entre investigadores y formadores en el ámbito de la Ciencia y Tecnología de Materiales
Sánchez-Soto P.J.; Eliche-Quesada D.; Martínez-Martínez S.; Garzón Garzón E.; Pérez-Villarejo L.
Poster

CONGRESOS Y REUNIONES NACIONALES / NATIONAL CONGRESS AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

VI Congreso Nacional de Pulvimetalurgia y I Congreso Iberoamericano de Pulvimetalurgia

7 – 9 junio [Ciudad Real, España]

Diseño, fabricación y caracterización de laminados de tipo WC-Co/WC-Co
Y. Torres; L. M. González; F. J. Gotor; E. Chicardi; E. Fernández; R. Bermejo
Comunicación oral

Aleaciones de alta entropía de mezcla como fase cementante en cermets de solución sólida completa
A.G. de la Obra; E. Chicardi; C. Real; M. J. Sayagués; F. J. Gotor
Comunicación oral

Obtención de una aleación cúbica TiTaNb mediante síntesis por aleado mecánico de baja energía y sinterización por Spark Plasma Sintering
E. Chicardi; C. Gutiérrez-González; A. Borrell; C. Aguilar
Comunicación oral

Reunión de la Sociedad Española de Catálisis | SECAT'17

26 – 28 junio [Oviedo, España]

Actividad catalítica de nanopartículas de TiO2 facetado en la condensación de compuestos oxigenados presentes en fracciones acuosas derivadas de la biomasa
A.Fernández-Arroyo; M.A. Lara; M.E. Domine; M.J. Sayagués; J.A. Navío; M.C.Hidalgo
Comunicación oral

FORMACION / TRAINING

TESIS DOCTORALES/ DOCTOR DEGREE THESIS

Título: **Estudio de cerámicas avanzadas (PDCS Polymer Derived Ceramics) obtenidas a partir de precursores poliméricos**
Autor: Cristina García Garrido
Directores: Pedro Enrique Sánchez Jiménez, Luis Allan Pérez Maqueda
Calificación: Sobresaliente "Cum Laude" por Unanimidad
Centro: Universidad de Sevilla
Fecha: 3 de julio de 2017

Título: **Del campo a la ciudad: Producción y comercialización de recipientes anfóricos en el bajo Guadalquivir durante la II Edad del Hierro**
Autor: Violeta Moreno Megías
Directores: Francisco J. García Fernández, Pedro José Sánchez Soto
Calificación: Sobresaliente "Cum Laude" por Unanimidad
Centro: Universidad de Sevilla
Fecha: 28 de septiembre de 2017

Título: **Mecanosíntesis y caracterización de materiales multiferroicos nanoestructurados. Mechanosynthesis and characterization of nanostructured multiferroic materials**
Autor: Eva Gil González
Directores: Antonio Perejón Pazo, Luis Allan Pérez Maqueda
Calificación: Sobresaliente "Cum Laude" por Unanimidad
Centro: Universidad de Sevilla
Fecha: 6 de octubre de 2017

FORMACIÓN DE GRADUADOS / MASTER DEGREE THESIS

Título: **Desarrollo de cermets tipo TiB2-Ti(C,N)/ni para aplicaciones avanzadas**
Autor: José Francisco Correa Ballesteros
Directores: Francisco José Gotor Martínez, Yadir Torres Hernández
Grado: Trabajo Fin de Grado
Año Académico: 2016-2017 (12 junio 2017)

Título: **Obtención de carbones a partir de biomasa**
Autor: Cristina Naranjo Calderón
Directores: María Dolores Alcalá González, Concepción Real Pérez
Grado: Trabajo Fin de Grado
Año Académico: 2016-2017 (7 julio 2017)

Título: **Fabricación de carburos cementados usando como fase aglomerante aleaciones de alta entropía (WC-FeCoNiCrMn)**
Autor: Irene Limón Velo
Director: Francisco José Gotor Martínez, José Manuel Córdoba Gallego
Grado: Trabajo Fin de Grado
Año Académico: 2016-2017 (13 julio 2017)

Título: **Viabilidad de la síntesis mecanoquímica para obtener $La_{1-x}Sr_xGaO_{3-\delta}$ con estructura perovskita**
Autor: Yunqing Tang
Directores: José Manuel Córdoba Gallego, María Jesús Sayagués de Vega
Grado: Trabajo Fin de Grado
Año Académico: 2016-2017 (13 julio 2017)

Título: **Viabilidad de la síntesis de materiales basados en Mg_2X X= Si, Ti,Zr) mediante técnicas mecanoquímicas**
Autor: Julián José Cáceres Murillo
Directores: José Manuel Córdoba Gallego, Francisco José Gotor Martínez
Grado: Trabajo Fin de Grado
Año Académico: 2017-2018 (21 septiembre 2017)

Título: **Caracterización microestructural y composicional de aleación de alta entropía basada en Fe, Co, Ni**
Autor: Elísabet Fernández Mansilla
Directores: Concepción Real Pérez, María Dolores Alcalá González
Grado: Trabajo Fin de Grado
Año Académico: 2017-2018 (27 septiembre 2017)

Docencia / Teaching

Máster en Biotecnología Sanitaria

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

EQUIPAMIENTO CIENTÍFICO AVAILABLE EQUIPMENT

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

MATERIALES FUNCIONALES NANOESTRUCTURADOS
NANOSTRUCTURED FUNCTIONAL MATERIALS

PERSONAL / PERSONNEL

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Dr. Manuel Ocaña Jurado
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Dra. Ana Isabel Borrás Martos
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Becarios Predoctorales

Ldo. Miguel Anaya Martín
Ldo. Aurelio García Valenzuela
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Lda. Victoria Esteso Carrizo
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Ldo. Mariano Laguna Moreno
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Lda. M. Carmen Gutiérrez Lázaro
Ing. Ester López Fernández

Proyectos de Investigación / Research Projects

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

Código/Code:	H2020-ERC-STG/0259
Periodo/Period:	1-04-2017 / 31-03-2022
Organismo Financiador/Financial source:	European Commission STARTING GRANT
Importe total/Total amount:	1.499.739 €
Investigador responsable/Research head:	Gabriel S. Lozano Barbero

RESUMEN / ABSTRACT

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

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

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

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

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

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

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

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

RESUMEN / ABSTRACT

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

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

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

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

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

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

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

RESUMEN / ABSTRACT

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

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

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

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

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

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

RESUMEN / ABSTRACT

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

PlasmaPerovSol tiene como objetivo principal la fabricación de una celda solar de perovskita completa mediante un proceso integrado de vacío y plasma llevado a cabo bajo el concepto de un solo reactor. Los diferentes componentes de la CS se depositarán secuencialmente en un reactor de vacío evitando la exposición de los materiales e intercaras al aire o disolventes. La tecnología de deposición asistida por plasma desarrollada por el grupo receptor permite la fabricación de películas altamente conformales sobre una amplia variedad de templates.Esta aproximación se propone para fabricar multicapas conformales sobre materiales unidimensionales, con el que se mejorarán numerosos aspectos de las CS. Los procesos de vacío y plasma presentan como ventajas una alta reproducibilidad, pureza y control estequiométrico en la deposición. La síntesis propuesta es compatible con la producción a escala industrial y permite la fabricación de CS en sustratos procesables y flexibles. Al mismo tiempo, las bajas temperaturas utilizadas la hacen compatible con la tecnología actual de dispositivos microelectrónicos, y mediante el uso de máscaras permiten su integración en dispositivos preformados.

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

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

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

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

RESUMEN / ABSTRACT

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

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

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

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

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

The proposed solution will be environment-friendly, will contribute to the reduction of

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

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

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

RESUMEN / ABSTRACT

NANOFlow es un projecto multidisciplinar que persigue el desarrollo de nuevos dispositivos optofluídicos mediante la integración de materiales nanostructurados multifuncionales. El proyecto está sólidamente fundamentado en la experiencia de los componentes del grupo de investigación en campos como la síntesis de películas multifuncionales, procesos avanzados de modificación de superficies y en el desarrollo de dispositivos fotónicos multicapa. El objetivo principal de este proyecto de investigación es combinar e integrar los distintos métodos avanzados de síntesis y procesado disponibles en la fabricación de dispositivos optofluídicos singulares que sean capaces de responder a la presencia de líquidos mediante un cambio físico. La combinación de este tipo de procesos de integración junto con el desarrollo de nuevos métodos de de detección fotónica, el uso de microplasmas de gran área como fuentes de luz y de sustratos flexibles que incorporan elementos sensores definen un conjunto muy rico de posibilidades de desarrollo de aplicaciones prácticas que se explorarán en el proyecto. Además, en el proyecto también se llevarán a cabo estudios de simulación de crecimiento de películas delgadas que, en combinación con estudios de diagnosis, permitirán ajustar los procesos de crecimiento para conseguir la funcionalidades requeridas. De esta forma, el proyecto NANOFlow intenta cubrir toda la cadena tecnológica que va desde el desarrollo de nuevos materiales hasta la aplicación final

incluyendo estudios de caracterización, rutas sintéticas flexibles, búsqueda de procesos alternativos de bajo costo y alto rendimiento (por ejemplo métodos de fabricación empleando plasmas a presión atmosférica), integración de dispostivos y test de éstos en condiciones reales.

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

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

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

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

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

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Unidad altamente optimizada para un Sistema solar sostenible y mejorado
Highly optimized unit for a sustainable enhanced solar system HOUSESS

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

RESUMEN / ABSTRACT

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

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

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

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

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MINISTERIO DE ECONOMÍA Y COMPETITIVIDAD

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

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

RESUMEN / ABSTRACT

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

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

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

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

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

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

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

RESUMEN / ABSTRACT

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

This project aims at the development of a new generation of low dimensional responsive systems and sensors that integrate nanostructured layers with well-controlled electrical and optical properties which, prepared by innovative vacuum and plasma methods, present a tunable and high porosity and are able to actively interact with the environment. The basic principles of the oblique angle approach (OAD) during the physical vapor deposition (PVD) of evaporated thin films will be extended to the fabrication of similar layers by plasma and magnetron sputtering techniques. Combination of these techniques along with other innovative plasma technologies, including atmospheric pressure plasma deposition or plasma-evaporation polymerization will be employed to achieve a strict control over the nanostructure and properties of final films

and complex systems . Supported metal and oxide nanostructured thin films, stacked multilayers and hybrid and composite suported nanostructures will be prepared and thereafter characterized by advanced electron and proximity microscopies and other techniques. Process-control strategies will be implemented in order to understand the fundamental mechanisms governing the film structurations and to propose new synthetic routes scalable to industrial production so as to achieve tailored morphologies and properties for these porous thin film materials. Highly ordered and homogenous arrays of these nanostructures will be used as ambient temperature gas and liquid sensors, microfluidic responsive devices and intelligent labelling tags. For these applications the supported porous thin films will be suitably functionalized with metal nanoparticles, grafted molecular chains or layers of other polymeric materials. They will be also stacked in the form of vertically ordered photonic structures. Innovative device integration approaches including the water removal of evaporated sacrificial layers of NaCl and their integration in the form of microdevices will be carried out to fabricate advanced sensors, microreactors and responsive systems. Photonic, electrical and/or electrochemical principles of transduction will be implemented into the devices for detecting and/or fabricating i) oxygen and chlorine in solutions, ii) glucose and organic matter in water iii) gas and vapor sensors or iv) inteligent labels. Specific applications are foressen for the control of the outside environment (air and waters), industrial and greenhouse locations, agroindustrial processes such as fermentation and the tracking and trazability of different kinds of goods and foods.

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

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

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

RESUMEN / ABSTRACT

La presente propuesta de proyecto de investigación persigue en primera instancia abordar una serie de aspectos básicos no resueltos relacionados con los mecanismos de la descarga

barrera, las condiciones óptimas que deben cumplir los electrodos, la definición de un diseño óptimo de los mismos y el establecimiento de las mejores condiciones para la descarga.

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

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

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

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

RESUMEN / ABSTRACT

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

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

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

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

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

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

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

OTROS PROYECTOS / OTHER PROJECTS

Red de Terapia Cellular (TERCEL)

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

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

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

Desarrollo de nuevos pigmentos inorgánicos con aplicaciones cerámicas

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

Nanopartículas basadas en matrices inorgánicas de tierras raras para aplicaciones biotecnolóticas

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

Convenios y Contratos / Contracts and Agreements

Limpieza de sistemas electrónicos para el espacio y mediante etching por plasma a baja presión

Periodo/Period: 02-10-2017 / 16-10-2017
Organismo Financiador/Financial source: ALTER TECHNOLOGY TÜV NORD S.A.U.
Importe total/Total amount: 6.050 €
Investigador responsable/Research head: Angel Barranco Quero

Procesos de activación de cintas poliméricas metalizadas para condensadores

Periodo/Period: 01-11-2017 / 30-04-2018
Organismo Financiador/Financial source: EPCOS ELECTRONIC COMPONENTS, S.A.
Importe total/Total amount: 43.560 €
Investigador responsable/Research head: José Cotrino Bautista

SPIRE: Solar Power Integrated Renewable Energy System (FEDER INTERCONNECTA)

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

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

Periodo/Period: 01-09-2015 / 30-09-2018
Organismo Financiador/Financial source: Universidad Carlos III, Indra Sistemas S.A., Universidad Politécnica de Madrid, Repsol S.A.
Importe total (ICMS)/Total amount (ICMS): 220.000 €
Investigador responsable (ICMS)/Research head (ICMS): Agustín R. González-Elipe
Componentes/Research group: Francisco Yubero Valencia, Jorge Gil Rostra, Victor Rico Gavira, Juan Pedro Espinós Manzorro, Angel Barranco Quero, Ana Isabel Borrás Martos

Patentes / Patents

Sistema y procedimiento microfluídico de medición de propiedades Físico-químicas de un líquido para al menos dos estados de dilución del mismo

Inventores: Francisco Yubero Valencia, Agustín R. González-Elipe, Pedro Angel Salazar Carballo, Manuel Oliva Ramírez
Tipo de Patente: Nacional
Número de Solicitud: 17382039
Fecha Solicitud: 27 de enero de 2017
Entidad Titular: Consejo Superior de Investigaciones Científicas

Artículos Publicados en Revistas SCI / Papers In SCI Journals

Diverse Applications of Nanomedicine

Pelaz, Beatriz; Alexiou, Christoph; Alvarez -Puebla, Ramon A.; Alves, Frauke; Andrews, Anne M.; Ashraf, Sumaira; Balogh, Lajos P.; Ballerini, Laura; Bestetti, Alessandra; Brendel, Cornelia; Bosi, Susanna; Carril, Monica; Chan, Warren C. W.; Chen, Chunying
ACS Nano, **11** (2017) 2312-2381
Marzo, 2017 | **DOI:** 10.1021/acsnano.6b06040

The design and use of materials in the nanoscale size range for addressing medical and health-related issues continues to receive increasing interest. Research in nanomedicine spans a multitude of areas, including drug delivery, vaccine development, antibacterial, diagnosis and imaging tools, wearable devices, implants, high-throughput screening platforms, etc. using biological, nonbiological, biomimetic, or hybrid materials. Many of these developments are starting to be translated into viable clinical products. Here, we provide an overview of recent developments in nanomedicine and highlight the current challenges and upcoming opportunities for the field and translation to the clinic.

1-dimensional TiO2 nano-forests as photoanodes for efficient and stable perovskite solar cells fabrication

Salado, M; Oliva-Ramirez, M; Kazim, S; Gonzalez-Elipe, AR; Ahmad, S
Nano Energy, **35** (2017) 215-222
Mayo, 2017 | **DOI:** 10.1016/j.nanoen.2017.03.034

During the last years, perovskite solar cells have gained increasing interest among the photovoltaic community, in particularly after reaching performances at par with mature thin film based PV. This rapid evolution has been fostered by the compositional engineering of perovskite and new device architectures. In the present work, we report the fabrication of perovskite solar cells based on highly ordered 1-dimensional vertically oriented TiO2 nano-forests. These vertically oriented porous TiO2 photoanodes were deposited by physical vapor deposition in an oblique angle configuration, a method which is scalable to fabricate large area devices. Mixed (MA0.15FA0.85)Pb(I0.85Br0.15)3 or triple cation Cs0.05(MA0.15FA0.85) 0.95Pb(I0.85Br0.15)3 based perovskites were then infiltrated into these 1-dimensional nanostructures and power conversion efficiencies of 16.8% along with improved stability was obtained. The devices fabricated using 1D-TiO2 were found to be more stable compare to the classical 3-dimensional TiO2 photoanodes prepared by wet chemistry. These 1-D photoanodes will be of interest for scaling up the technology and in other opto-electrical devices as they can be easily fabricated utilizing industrially adapted methodologies.

Critical Role of Oxygen in Silver-Catalyzed Glaser-Hay Coupling on Ag(100) under Vacuum and in Solution on Ag Particles

Orozco, N; Kyriakou, G; Beaumont, SK; Sanz, JF; Holgado, JP; Taylor, MJ; Espinos, JP; Marquez, AM; Watson, DJ; Gonzalez-Elipe, AR; Lambert, RM

ACS Catalysis, **7** (2017) 3113-3120

Mayo, 2017 | **DOI:** 10.1021/acscatal.7b00431

The essential role of oxygen in enabling heterogeneously catalyzed Glaser–Hay coupling of phenylacetylene on Ag(100) was elucidated by STM, laboratory and synchrotron photoemission, and DFT calculations. In the absence of coadsorbed oxygen, phenylacetylene formed well-ordered dense overlayers which, with increasing temperature, desorbed without reaction. In striking contrast, even at 120 K, the presence of oxygen led to immediate and complete disruption of the organic layer due to abstraction of acetylenic hydrogen with formation of a disordered mixed layer containing immobile adsorbed phenylacetylide. At higher temperatures phenylacetylide underwent Glaser–Hay coupling to form highly ordered domains of diphenyldiacetylene that eventually desorbed without decomposition, leaving the bare metal surface. DFT calculations showed that, while acetylenic H abstraction was otherwise an endothermic process, oxygen adatoms triggered a reaction-initiating exothermic pathway leading to OH(a) + phenylacetylide, consistent with the experimental observations. Moreover, it was found that, with a solution of phenylacetylene in nonane and in the presence of O2, Ag particles catalyzed Glaser–Hay coupling with high selectivity. Rigorous exclusion of oxygen from the reactor strongly suppressed the catalytic reaction. Interestingly, too much oxygen lowers the selectivity toward diphenyldiacetylene. Thus, vacuum studies and theoretical calculations revealed the key role of oxygen in the reaction mechanism, subsequently borne out by catalytic studies with Ag particles that confirmed the presence of oxygen as a necessary and sufficient condition for the coupling reaction to occur. The direct relevance of model studies to a mechanistic understanding of coupling reactions under conditions of practical catalysis was reaffirmed.

Impact of moisture on efficiency-determining electronic processes in perovskite solar cells

Salado, Manuel; Contreras-Bernal, Lidia; Calio, Laura; Todinova, Anna; Lopez-Santos, Carmen; Ahmad, Shahzada; Borras, Ana; Idigoras, Jesus; Anta, Juan A.

Journal of Materials Chemistry A, **5** (2017) 10917-10927

Junio, 2017 | **DOI:** 10.1039/c7ta02264f

Moisture-induced degradation in perovskite solar cells was thoroughly investigated by structural (SEM, EDS, XRD and XPS) and device characterization (impedance and intensity modulated photocurrent spectroscopy) techniques. Both the influence of the perovskite composition and the nature of the hole selective material were analyzed. The degradation rate was found to be significantly slower for mixed perovskites and P3HT-based devices. However, for a fixed degradation degree (defined as a 50% drop from the initial photocurrent), all configurations show similar features in small-perturbation analysis. Thus, a new mid-frequency signal appears in the impedance response, which seems to be related to charge accumulation at the interfaces. In addition, faster recombination, with a more important surface contribution, and slower transport were clearly inferred from our results. Both features can be associated with the deterioration of the contacts and the formation of a higher number of grain boundaries.

Materials chemistry approaches to the control of the optical features of perovskite solar cells

Calvo, Mauricio E.

Journal of Materials Chemistry A, **5** (2017) 20561-20578

Octubre, 2017 | **DOI:** 10.1039/c7ta05666d

Perovskite solar cells have revolutionized the field of photovoltaics. Apart from their impressive photo-conversion efficiencies, the ease of their fabrication – principally when carried out by solution processing – has permitted addition of new functionalities to the photovoltaic cell. Some of these features are related to the optical properties. In this review, the different materials chemistry approaches that allow controlling the spectral absorption of ABX3 perovskite layers and the changes that they produce in the visual aspect of the solar cell will be covered. These modifications can be done either by playing with the composition of the precursors or by integrating different types of nanostructures. Spectral bandgap tuning, semitransparency, color and enhancement of light absorption are examples of how these modifications operate in the core of ABX3 perovskite solar cells. These optical features bring benefits in terms of photo-conversion efficiencies or else in the aesthetical integration of perovskite solar cells with architectonic elements for building integrated photovoltaics. Additionally, surface passivation approaches are covered to show its effect over the photo-conversion efficiency and stability of the cell.

Multicolored Emission and Lasing in DCM-Adamantane Plasma Nanocomposite Optical Films

Alcaire, M; Cerdan, L; Zamarro, FL; Aparicio, FJ; Gonzalez, JC; Ferrer, FJ; Borras, A; Espinos, JP; Barranco, A

ACS Applied Materials & Interfaces, **9** (2017) 8948-8959

Marzo, 2017 | **DOI:** 10.1021/acsami.7b01534

We present a low-temperature versatile protocol for the fabrication of plasma nanocomposite thin films to act as tunable emitters and optical gain media. The films are obtained by the remote plasma-assisted deposition of a 4-(dicyano-methylene)-2-methy1-6-(4-dimethylamino-styry1)-4Hpyran (DCM) laser dye alongside adamantane. The experimental parameters that determine the concentration of the dye in the films and their optical properties, including light absorption, the refractive index, and luminescence, are evaluated. Amplified spontaneous emission experiments in the DCM/adamantane nano composite waveguides show the improvement of the copolymerized nano composites' properties compared to films that were deposited with DCM as the sole precursor. Moreover, one-dimensional distributed feed-back laser emission is demonstrated and characterized in some of the nanocomposite films that are studied. These results open new paths for the optimization of the optical and lasing properties of plasma nanocomposite polymers, which can be straightforwardly integrated as active components in optoelectronic devices.

Energy-Sensitive Ion- and Cathode-Luminescent Radiation-Beam Monitors Based on Multilayer Thin-Film Designs

Gil-Rostra, Jorge; Ferrer, Francisco J.; Pedro Espinos, Juan; Gonzalez-Elipe, Agustin R.; Yubero, Francisco
ACS Applied Materials & Interfaces, **9** (2017) 16313-16320
Mayo, 2017 | **DOI:** 10.1021/acsami.7b01175

A multilayer luminescent design concept is presented to develop energy sensitive radiation-beam monitors on the basis of colorimetric analysis. Each luminescent layer within the stack consists of rare-earth-doped transparent oxides of optical quality and a characteristic luminescent emission under excitation with electron or ion beams. For a given type of particle beam (electron, protons, alpha particles, etc.), its penetration depth and therefore its energy loss at a particular buried layer within the multilayer stack depend on the energy of the initial beam. The intensity of the luminescent response of each layer is proportional to the energy deposited by the radiation beam within the layer, so characteristic color emission will be achieved if different phosphors are considered in the layers of the luminescent stack. Phosphor doping, emission efficiency, layer thickness, and multilayer structure design are key parameters relevant to achieving a broad colorimetric response. Two case examples are designed and fabricated to illustrate the capabilities of these new types of detector to evaluate the kinetic energy of either electron beams of a few kilo-electron volts or a particles of alpha few mega-electron volts.

Facile Synthesis of Hybrid Organic–Inorganic Perovskite Microcubes of Optical Quality Using Polar Antisolvents

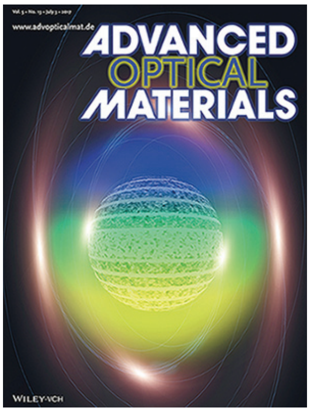
Li, Yuelong; Galisteo-Lopez, Juan F.; Calvo, Mauricio E.; Miguez, Hernan
ACS Applied Materials & Interfaces, **9** (2017) 35505-35510
Octubre, 2017 | **DOI:** 10.1021/acsami.7b08431

Herein, we demonstrate a synthetic approach producing highly crystalline methylammonium lead bromide perovskite (MAPbBr3) microcubes at room temperature by injecting a perovskite precursor solution into an environmentally friendly antisolvent (isopropyl alcohol). Confirmed by X-ray and electron diffraction, as well as electron microscopy, these MAPbBr3 microcubes are single crystals and have perfect cubic structure, with sizes varying between 1 and 15 µm depending on the synthesis conditions. The stoichiometry of the MAPbBr3 crystal is proven by energy-dispersive X-ray analysis. Finally, optical analysis carried out by means of laser scanning confocal microscopy evidences how the crystalline quality of the microcubes translates into a homogeneous photoluminescence throughout the cube volume.

Design and Realization of a Novel Optically Disordered Material: A Demonstration of a Mie Glass

Miranda-Munoz, Jose M.; Lozano, Gabriel; Miguez, Hernan
Advanced Optical Materials, **5** (2017) art 1700025
Mayo, 2017 | **DOI:** 10.1002/adom.201700025

Herein, a diffusive material presenting optical disorder is introduced, which represents an example of a Mie glass. Comprising spherical crystalline TiO2 nanoparticles randomly dispersed in a mesoporous TiO2 matrix, it is proved that the scattering of light in this inhomogeneous solid can be predicted in an unprecedented manner from single-particle considerations employing Mie theory. To that aim, a study of the dependence of the key parameters employed is performed to describe light propagation in random media, i.e., the scattering mean free path and the transport mean free path, as a function of the size and concentration of the spherical inclusions based on a comparison between experimental results and analytical calculations. It is also demonstrated that Mie glasses enable enhanced fluorescence intensity due to a combined absorptance enhancement of the excitation light combined with an improved outcoupling of the emitted light. The method offers the possibility to perform a deterministic design for the realization of a light diffuser with tailor-made scattering properties.



Photonic Tuning of the Emission Color of Nanophosphor Films Processed at High Temperature

Geng, Dongling; Lozano, Gabriel; Calvo, Mauricio E.; Nunez, Nuria O.; Becerro, Ana I.; Ocana, Manuel; Miguez, Hernan
Advanced Optical Materials, **5** (2017) art 170009
Julio, 2017 | **DOI:** 10.1002/adom.201700099

Photonics offers new possibilities to tailor the photoluminescence process in phosphor-converted light emitting diodes. Herein, it is demonstrated that the emission color of thin layers of rare-earth doped nanocrystals can be strongly modulated in tunable spectral ranges using optical resonators specifically designed to this end. GdVO4:Dy3+ nanoparticles of controlled size and shape are synthesized using a solvothermal method with which highly transparent nanophosphor thin films are prepared. This paper designs and fabricates optical multilayers, which are transparent in the UV and resonant at the frequencies where the Dy3+ ions emit, to prove that the color coordinates of this emitter can be tuned from green to blue or yellow with unprecedented precision. Key to the achievement herein reported is the careful analysis of the structural and optical properties of thin nanophosphor layers with the processing temperature in order to achieve efficient photoluminescence while preserving the transparency of the film. The results open a new path for fundamental and applied research in solid-state lighting in which photonic nanostructures allow controlling the emission properties of state-of-the-art materials without altering their structure or chemical composition.

Aperiodic Metal-Dielectric Multilayers as Highly Efficient Sunlight Reflectors

Alberto Jiménez-Solano; Miguel Anaya; Mauricio E. Calvo; Mercedes Alcon-Camas; Carlos Alcañiz; Elena Guillén; Noelia Martínez; Manuel Gallas; Thomas Preussner; Ramón Escobar-Galindo; Hernán Míguez
Advanced Optical Materials, **5** (2017) art 1600833
Marzo, 2017 | **DOI:** 10.1002/adom.201600833

The optimum reflection of the solar spectrum at well-defined incident directions as well as its durability in time are, both, fundamental requirements of the optics of thermosolar and photovoltaic energy conversion systems. The stringent high performance needed for these applications implies that, almost exclusively, second face mirrors based on silver are employed for this purpose. Herein, the possibility to develop solar mirrors using other metals, such as copper and aluminum, is theoretically and experimentally analyzed. It is found that reflectors based on these inexpensive metals are capable of reflecting the full solar spectrum with efficiencies comparable to that of silver-based reflectors. The designs herein proposed are based on aperiodic metal-dielectric multilayers whose optimized configuration is chosen employing a code based on a genetic algorithm that allows selecting the best one among 108 tested reflectors. The use of metals with wider spectral absorption bands is compensated by the use of multilayered designs in which metal absorption is almost suppressed, as the analysis of the electric field intensity distribution demonstrates. The feasibility of the proposed mirrors is demonstrated by their actual fabrication by large area deposition techniques amenable for mass production.

Fluorescent Humidity Sensors Based on Photonic Resonators

Szendrei, K; Jimenez-Solano, A; Lozano, G; Lotsch, BV; Miguez, H
Advanced Optical Materials, **5** (2017) art 1700663
Diciembre, 2017 | **DOI:** 10.1002/adom.201700663

Among the different approaches to humidity sensing available, those based on fluorescent signals are gathering a great deal of attention due to their fast response and versatility of detection and design. So far, all proposals have focused on the use of luminescent probes whose emission is either triggered or inhibited by the presence of water that reacts or alters their chemical environment, hence inducing the signal change. Here, a novel concept in fluorescent humidity sensing based on combining stimuli-responsive photonic resonators with molecular fluorescent probes is introduced. The resonator is assembled from humidity-swellable antimony phosphate nanosheets embedding a planar light-emitting probe, whose emission is dramatically modified by the changes that ambient humidity causes in its photonic environment. Guided by "in silico" optical design of the resonator architecture and subsequent experimental realization, two embodiments of fluorescent photonic humidity sensors featuring turn-on and turn-off detection schemes are presented. The interplay between the luminescent properties of an emitter and its photonic environment implies a fundamental advantage as the emitters are not chemically altered during the detection process. At the same time, it paves the way toward a new generation of photonic humidity sensors which can conveniently be interfaced with common fluorescence detection schemes.

Strong Quantum Confinement and Fast Photoemission Activation in CH3NH3PbI3 Perovskite Nanocrystals Grown within Periodically Mesostructured Films

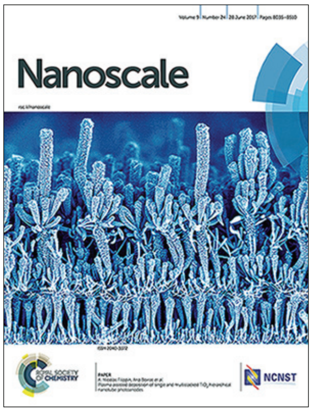
Miguel Anaya; Andrea Rubino; Teresa Cristina Rojas; Juan Francisco Galisteo-López; Mauricio Ernesto Calvo; Hernán Míguez
Advanced Optical Materials, **5** (2017) 1601087
Marzo, 2017 | **DOI:** 10.1002/adom.201601087

Efficient promotion of the Pt/CeO2/Al2O3 catalytic system was achieved by the addition of two different ceria promoters, Zr and Fe. From the exhaustive data analysis, the key features for enhanced catalytic performance and the roles of each doping metal are established. The combination of both doping agents manifests a synergistic effect reflected in noteworthy improvements in H2 reducibility. In addition, the catalyst's doping influences its chemisorptive properties, which is reflected in an increase of the easiness of carbonaceous species desorption, thus leading to superior catalyst resistance toward deactivation.

High vacuum synthesis and ambient stability of bottom-up graphene nanoribbons

Fairbrother, A.; Sanchez-Valencia, J.R.; Lauber, B.; Shorubalko, I.; Ruffieux, P.; Hintermann, T.; Fasel, R.
Nanoscale, **9** (2017) 2785-2792
Febrero, 2017 | **DOI:** 10.1039/C6NR08975E

Carbon-based nanomaterials such as graphene are at a crucial point in application development, and their promising potential, which has been demonstrated at the laboratory scale, must be translated to an industrial setting for commercialization. Graphene nanoribbons in particular overcome one limitation of graphene in some electronic applications because they exhibit a sizeable bandgap. However, synthesis of bottom-up graphene nanoribbons is most commonly performed under ultra-high vacuum conditions, which are costly and difficult to maintain in a manufacturing environment. Additionally, little is known about the stability of graphene nanoribbons under ambient conditions or during transfer to technologically relevant substrates and subsequent device processing. This work addresses some of these challenges, first by synthesizing bottom-up graphene nanoribbons under easily obtained high vacuum conditions and identifying water and oxygen as the residual gases responsible for interfering with proper coupling during the polymerization step. And second, by using Raman spectroscopy to probe the stability of nanoribbons during storage under ambient conditions, after transfer to arbitrary substrates, and after fabrication of field-effect transistor devices, which shows structurally intact nanoribbons even several months after synthesis. These findings demonstrate the potential of graphene nanoribbon technologies by addressing some limitations which might arise in their commercialization.



Plasma assisted deposition of single and multistacked TiO2 hierarchical nanotube photoanodes

Filippin, AN; Sanchez-Valencia, JR; Idigoras, J; Rojas, TC; Barranco, A; Anta, JA; Borras, A
Nanoscale, **9** (2017) 8133-8141
Julio, 2017 | **DOI:** 10.1039/c7nr00923b

We present herein an evolved methodology for the growth of nanocrystalline hierarchical nanotubes combining physical vapor deposition of organic nanowires (ONWs) and plasma enhanced chemical vacuum deposition of anatase TiO2 layers. The ONWs act as vacuum removable 1D and 3D templates, with the whole process occurring at temperatures ranging from RT to 250 degrees C. As a result, a high density of hierarchical nanotubes with tunable diameter, length and tailored wall microstructures are formed on a variety of processable substrates as metal and metal oxide films or nanoparticles including transparent conductive oxides. The reiteration of the process leads to the development of an unprecedented 3D nanoarchitecture formed by stacking the layers of hierarchical TiO2 nanotubes. As a proof of concept, we present the superior performance of the 3D nanoarchitecture as a photoanode within an excitonic solar cell with efficiencies as high as 4.69% for a nominal thickness of the anatase layer below 2.75 mu m. Mechanical stability and straightforward implementation in devices are demonstrated at the same time. The process is extendable to other functional oxides fabricated by plasma-assisted methods with readily available applications in energy harvesting and storage, catalysis and nanosensing.

High performance novel gadolinium doped ceria/yttria stabilized zirconia/nickel layered and hybrid thin film anodes for application in solid oxide fuel cells

Garcia-Garcia, FJ; Beltran, AM; Yubero, E; Gonzalez-Elipe, AR; Lambert, RM
Journal of Power Sources, **363** (2017) 251-259
Septiembre, 2017 | **DOI:** 10.1016/j.jpowsour.2017.07.085

Magnetron sputtering under oblique angle deposition was used to produce Ni-containing ultra thin film anodes comprising alternating layers of,gadolinium doped ceria (GDC) and yttria stabilized zirconia (YSZ) of either 200 nm or 1000 nm thickness. The evolution of film structure from initial deposition, through calcination and final reduction was examined by XRD, SEM, TEM and TOF-SIMS. After subsequent fuel cell usage, the porous columnar architecture of the two-component layered thin film anodes was maintained and their resistance to delamination from the underlying YSZ electrolyte was superior to that of corresponding single component Ni-YSZ and Ni-GDC thin films. Moreover, the fuel cell performance of the 200 nm layered anodes compared favorably with conventional commercially available thick anodes. The observed dependence of fuel cell performance on individual layer thicknesses prompted study of equivalent but more easily fabricated hybrid anodes consisting of simultaneously deposited Ni-GDC and Ni-YSZ, which procedure resulted in exceptionally intimate mixing and interaction of the components. The hybrids exhibited very unusual and favorable I-V characteristics, along with exceptionally high power densities at high currents. Their discovery is the principal contribution of the present work.

Improving the pollutant removal efficiency of packed-bed plasma reactors incorporating ferroelectric components

Gomez-Ramirez, Ana; Montoro-Damas, Antonio M.; Rodriguez, Miguel A.; Gonzalez-Elipe, Agustin R.; Cotrino, Jose

*Chemical Engineering Journal, **314*** (2017) 311-319

Abril, 2017 | **DOI:** 10.1016/j.cej.2016.11.065

In this work we have studied the plasma removal of air contaminants such as methane, chloroform, toluene and acetone in two parallel plate packed-bed dielectric barrier discharge (DBD) reactors of different sizes. Removal and energy efficiencies have been determined as a function of the residence time of the contaminated air within the reactor, the kind of packed-bed material (ferroelectrics or classical dielectric materials), the frequency and the incorporation of a ferroelectric plate onto the active electrode together with the inter-electrode ferroelectric pellets filling the gap. Results at low frequency with the small reactor and the ferroelectric plate showed an enhancement in energy efficiency (e.g., it was multiplied by a factor of six and three for toluene and chloroform, respectively) and in removal yield (e.g., it increased from 22% to 52% for chloroform and from 15% to 21% for methane). Such enhancements have been attributed to the higher energy of plasma electrons and a lower reactor capacitance found for this plate-modified configuration. A careful analysis of reaction efficiencies and electron energy distributions for the different investigated conditions and the simulation of the electric field at the necks between ferroelectric/dielectric pellets complete the present study. Overall, the obtained results prove the critical role of the barrier architecture and operating conditions for an enhanced performance of pollution removal processes using DBD systems.

Rare earth based nanostructured materials: synthesis, functionalization, properties and bioimaging and biosensing applications

Escudero, Alberto; Becerro, Ana I.; Carrillo-Carrion, Carolina; Nunez, Nuria O.; Zyuzin, Mikhail V.; Laguna, Mariano; Gonzalez-Mancebo, Daniel; Ocana, Manuel; Parak, Wolfgang J.

*Nanophotonics, **6*** (2017) 881-921

Septiembre, 2017 | **DOI:** 10.1515/nanoph-2017-0007

Rare earth based nanostructures constitute a type of functional materials widely used and studied in the recent literature. The purpose of this review is to provide a general and comprehensive overview of the current state of the art, with special focus on the commonly employed synthesis methods and functionalization strategies of rare earth based nanoparticles and on their different bioimaging and biosensing applications. The luminescent (including downconversion, upconversion and permanent luminescence) and magnetic properties of rare earth based nanoparticles, as well as their ability to absorb X-rays, will also be explained and connected with their luminescent, magnetic resonance and X-ray computed tomography bioimaging applications, respectively. This review is not only restricted to nanoparticles, and recent advances reported for in other nanostructures containing rare earths, such as metal organic frameworks and lanthanide complexes conjugated with biological structures, will also be commented on.

Electron injection and scaffold effects in perovskite solar cells

M. Anaya, W. Zhang, B. Clasen Hames, Y. Li, F. Fabregat-Santiago, M.E. Calvo, H.J. Snaith, H. Míguez, I. Mora-Seró
Journal of Materials Chemistry C, **5** (2017) 634-644
Enero, 2017 | **DOI:** 10.1039/C6TC04639H

In spite of the impressive efficiencies reported for perovskite solar cells (PSCs), key aspects of their working principles, such as electron injection at the contacts or the suitability of the utilization of a specific scaffold layer, are not yet fully understood. Increasingly complex scaffolds attained by the sequential deposition of TiO2 and SiO2 mesoporous layers onto transparent conducting substrates are used to perform a systematic characterization of both the injection process at the electron selective contact and the scaffold effect in PSCs. By forcing multiple electron injection processes at a controlled sequence of perovskite–TiO2 interfaces before extraction, interfacial injection effects are magnified and hence characterized in detail. An anomalous injection behavior is observed, the fingerprint of which is the presence of significant inductive loops in the impedance spectra with a magnitude that correlates with the number of interfaces in the scaffold. Analysis of the resistive and capacitive behavior of the impedance spectra indicates that the scaffolds could hinder ion migration, with positive consequences such as lowering the recombination rate and implications for the current–potential curve hysteresis. Our results suggest that an appropriate balance between these advantageous effects and the unavoidable charge transport resistive losses introduced by the scaffolds will help in the optimization of PSC performance.

Cholesterol biosensing with a polydopamine-modified nanostructured platinum electrode prepared by oblique angle physical vacuum deposition

Martin, M; Salazar, P; Alvarez, R; Palmero, A; Lopez-Santos, C; Gonzalez-Mora, JL; Gonzalez-Elipe, AR
Sensors and Actuators B-Chemical, **240** (2017) 37-45
Febrero, 2017 | **DOI:** 10.1016/j.snb.2016.08.092

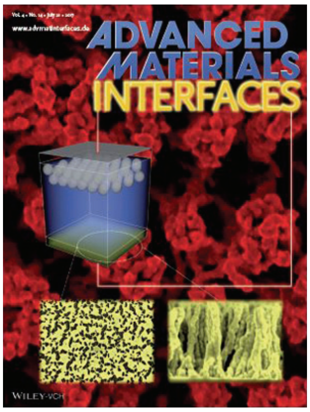
This paper reports a novel cholesterol biosensor based on nanostructured platinum (Pt) thin films prepared by Magnetron Sputtering (MS) in an oblique angle (OAD) configuration. Pt thin films were deposited onto a gold screen-printed electrode and characterized using Rutherford Back Scattering (RBS), Scanning Electron Microscopy (SEM), X-ray Diffraction (XRD), Cyclic Voltammetry (CV), X-ray Photo-electron Spectroscopy (XPS), Atomic Force Microscopy (AFM) and wetting analysis. Our results confirmed that the film is highly porous and formed by tilted nanocolumns, with an inclination of around 40 degrees and a total thickness of 280 nm. XRD and CV analysis confirmed the polycrystalline nature of the Pt thin film. Cholesterol oxidase (ChOx) was covalently immobilized using a bioinspired polymer, polydopamine (PDA), via Schiff base formation and Michael-type addition. After being immobilized, ChOx displayed apparent activation energy of 34.09 kJ mol(-1) and Michaelis constant (K-M) values of 34.09 kJ mol(-1) and 3.65 mM, respectively, confirming the high affinity between ChOx and cholesterol and the excellent ability of the PDA film for immobilizing biological material without degradation. Under optimized working conditions the developed biosensor presented a sensitivity of 14.3 mA M(-1)cm(-2) (R-2:0.999) with a linear range up to 0.5 mM and a limit of detection of 10.5 mu M

(S/N= 3). Furthermore, the biosensor exhibited a fast response (<8 s), good anti-interference properties and high stability after relatively long-term storage (2 months).

Non-enzymatic hydrogen peroxide detection at NiO nanoporous thin film-electrodes prepared by physical vapor deposition at oblique angles

Salazar, Pedro; Rico, Victor; Gonzalez-Elipe, Agustin R.
Electrochimica Acta, **235** (2017) 534-542
Mayo, 2017 | **DOI:** 10.1016/j.electacta.2017.03.087

In this work we report a non-enzymatic sensor for hydrogen peroxide (H2O2) detection based on nanostructured nickel thin films prepared by physical vapor deposition at oblique angles. Porous thin films deposited on ITO substrates were characterized by X-ray diffraction analysis, scanning electron microcopy (SEMs), X-ray photoelectron spectroscopy (XPS) and electrochemical techniques such as Cyclic Voltammetry (CV) and Constant Potential Amperometry (CPA). The microstructure of the thin films consisted of inclined and separated Ni nanocolumns forming a porous thin layer of about 500 nm thickness. Prior to their use, the films surface was electrochemically modified and the chemical state studied by CV and XPS analysis. These techniques also showed that Ni2+/Ni3+ species were involved in the electrochemical oxidation and detection of H2O2 in alkaline medium. Main analytical parameters such as sensitivity (807 mA M(-1)cm(-2)), limit of detection (3.22 mu M) and linear range (0.011-2.4 mM) were obtained under optimal operation conditions. Sensors depicted an outstanding selectivity and a high stability and they were successfully used to determine H2O2 concentration in commercial antiseptic solutions.



Low-Temperature Plasma Processing of Platinum Porphyrins for the Development of Metal Nanostructured Layers

Filippin, AN; Sanchez-Valencia, JR; Idigoras, J; Macias-Montero, M; Alcaire, M; Aparicio, FJ; Espinos, JP; Lopez-Santos, C; Frutos, F; Barranco, A; Anta, JA; Borras, A
Advanced Materials Interfaces, **4** (2017) 1601233
Julio, 2017 | **DOI:** 10.1002/admi.201601233

This article establishes the bases for a vacuum and plasma supported methodology for the fabrication at mild temperatures of nanostructured platinum in the form of porous layers and nanocolumns using platinum octaethylporphyrin as precursor. In addition, the application of these materials as tunable optical filters and nano-counterelectrodes is proved. On one hand, the transparency in the ultraviolet-visible-near infrared range can be adjusted precisely between 70% and 1% by tuning the deposition and processing conditions, obtaining a high spectral planarity. Deviations of the spectra from an ideal flat filter are below 4%, paving the way to the fabrication of neutral density filters. The transparency limit values yield a sheet resistivity of approximate to 1350 and 120 Omega square(-1), respectively. On the other hand, the catalytic properties of the nanostructures are further demonstrated by their implementation as counterelectrodes of excitonic solar cells surpassing the performance of commercial platinum as counterelectrode in a 20% of the overall cell efficiency due to simultaneous enhancement of short-circuit photocurrent and open-circuit photovoltage. One of the most interesting features of the developed methodology is its straightforward application to other metal porphyrins and phthalocyanines readily sublimable under mild vacuum and temperature conditions.

Local Disorder and Tunable Luminescence in Sr1–x/2Al2–xSixO4 (0.2 ≤ x ≤ 0.5) Transparent Ceramics

Fernandez-Carrion, AJ; Al Saghir, K; Veron, E; Becerro, AI; Porcher, F; Wisniewsld, W; Matzen, G; Fayon, F; Allix, M
Inorganic Chemistry, **56** (2017) 14446-14458
Diciembre, 2017 | **DOI:** 10.1021/acs.inorgchem.7b01881

Eu-doped Sr1–x/2Al2–xSixO4 (x = 0.2, 0.4, and 0.5) transparent ceramics have been synthesized by full and congruent crystallization from glasses prepared by aerodynamic levitation and laser-heating method. Structural refinements from synchrotron and neutron powder diffraction data show that the ceramics adopt a 1 × 1 × 2 superstructure compared to the SrAl2O4 hexagonal polymorph. While the observed superstructure reflections indicate a long-range ordering of the Sr vacancies in the structure, 29Si and 27Al solid-state NMR measurements associated with DFT computations reveal a significant degree of disorder in the fully polymerized tetrahedral network. This is evidenced through the presence of Si–O–Si bonds, as well as Si(OAl)4 units at remote distances of the Sr vacancies and Al(OAl)4 units in the close vicinity of Sr vacancies departing from local charge compensation in the network. The transparent ceramics can be doped by europium to induce light emission arising from the volume under UV excitation. Luminescence measurements then reveal the coexistence of Eu2+ and Eu3+ in the samples, thereby allowing tuning the emission color depending on the excitation wavelength and suggesting possible applications such as solid state lighting.

Comprehensive and Systematic Analysis of the Immunocompatibility of Polyelectrolyte Capsules

Zyuzin, MV; Diez, P; Goldsmith, M; Carregal-Romero, S; Teodosio, C; Rejman, J; Feliu, N; Escudero, A; Almendral, MJ; Linne, U; Peer, D; Fuentes, M; Parak, WJ
Bioconjugate Chemistry, **28** (2017) 556-564
Febrero, 2017 | **DOI:** 10.1021/acs.bioconjchem.6b00657

The immunocompability of polyelectrolyte capsules synthesized by layer-by-layer deposition has been investigated. Capsules of different architecture and composed of either non-degradable or biodegradable polymers, with either positively or negatively charged outer surface, and with micrometer size, have been used, and the capsule uptake by different cell lines has been studied and quantified. Immunocompatibility studies were performed with peripheral blood mononuclear cells (PBMCs). Data demonstrate that incubation with capsules, at concentrations relevant for practical applications, did not result in a reduced viability of cells, as it did not show an increased apoptosis. Presence of capsules also did not result in an increased expression of TNF-α, as detected with antibody staining, as well as at mRNA level. It also did not result in increased expression of IL-6, as detected at mRNA level. These results indicate that the polyelectrolyte capsules used in this study are immunocompatible.

Formation of Subsurface W5+ Species in Gasochromic Pt/WO3 Thin Films Exposed to Hydrogen

Castillero, Pedro; Rico-Gavira, Victor; Lopez-Santos, Carmen; Barranco, Angel; Perez-Dieste, Virginia; Escudero, Carlos; Espinos, Juan P.; Gonzalez-Elipe, Agustin R.

Journal of Physical Chemistry C, **121** (2017) 15719-15727

Julio, 2017 | **DOI:** 10.1021/acs.jpcc.7b03385

M/WO3 (M = Pt, Pd) systems formed by a porous WO3 thin film decorated by metal nanoparticles are known for their reversible coloring upon exposure to H2 at room temperature. In this work, this gasochromic behavior is investigated in situ by means of near-ambient photoemission (NAPP). Pt/WO3 systems formed by very small Pt nanoparticles (10 ± 1 nm average size) incorporated in the pores of nanocolumnar WO3 thin films prepared by magnetron sputtering at an oblique angle have been exposed to a small pressure of hydrogen at ambient temperature. The recorded UV–vis transmission spectra showed the reversible appearance of a very intense absorption band responsible for the blue coloration of these gasochromic films. In an equivalent experiment carried out in the NAPP spectrometer, W 4f, O 1s, Pt 4f, and valence band photoemission spectra have been recorded at various photon energies to follow the evolution of the reduced tungsten species and hydroxyl groups formed upon film exposure to hydrogen. The obtained results are compared with those of a conventional X-ray photoemission study after hydrogen exposure between 298 and 573 K. As investigated by NAPP, the gasochromic behavior at 298 K is accounted for by a reaction scheme in which hydrogen atoms resulting from the dissociation of H2 onto the Pt nanoparticles are spilt over to the WO3 substrate where they form surface OH–/H2O species and subsurface W5+ cations preferentially located in buried layers of the oxide network.

HoF3 and DyF3 Nanoparticles as Contrast Agents for High-Field Magnetic Resonance Imaging

Gonzalez-Mancebo, Daniel; Becerro, Ana I.; Rojas, T. Cristina; Garcia-Martin, Maria L.; de la Fuente, Jesus M.; Ocana, Manuel

Particle & particle systems characterization, **34** (2017) art. 1700116

Octubre, 2017 | **DOI:** 10.1002/ppsc.201700116

Clinical contrast agents (CAs) currently used in magnetic resonance imaging (MRI) at low fields are less effective at high magnetic fields. The development of new CAs is mandatory to improve diagnostic capabilities of the new generation of high field MRI scanners. The purpose of this study is to synthesize uniform, water dispersible LnF3 (Ln = Ho, Dy) nanoparticles (NPs) and to evaluate their relaxivity at high magnetic field (9.4 T) as a function of size and composition. Two different types of HoF3 NPs are obtained by homogeneous precipitation in ethylene glycol at 120 °C. The use of holmium acetate as holmium precursor leads to rhombus-like nanoparticles, while smaller, ellipsoid-like nanoparticles are obtained when nitrate is used as the holmium salt. To explain this behavior, the mechanism of formation of both kinds of particles is analyzed in detail. Likewise, rhombus-like DyF3 nanoparticles are prepared following the same method as for the rhombus-like HoF3 nanoparticles. We have found, to the best of knowledge, the highest transverse relaxivity values at 9.4 T described in the literature for this kind of CAs. Finally, the LnF3 NPs have shown negligible cytotoxicity for C6 rat glioma cells for concentrations up to 0.1 mg mL−1.

One-reactor plasma assisted fabrication of ZnO@TiO2 multishell nanotubes: assessing the impact of a full coverage on the photovoltaic performance

Filippin, Alejandro Nicolas; Macias-Montero, Manuel; Saghi, Zineb; Idigoras, Jesus; Burdet, Pierre; Sanchez-Valencia, Juan R.; Barranco, Angel; Migdley, Paul A.; Anta, Juan A.; Borras, Ana

Scientific Reports, **7** (2017) art. 9621

Agosto, 2017 | **DOI:** 10.1038/s41598-017-09601-7

This paper addresses the fabrication of vertically aligned ZnO@TiO2multishell nanotubes by a combined full vacuum-plasma approach at mild temperatures. The growth is carried out within the premises of a one-reactor approach, i.e. minimizing the number of vacuum chambers and sample transferences. In this way, the interface between ZnO and TiO2 is fully preserved from humidity thus increasing ZnO durability and stability. These nanostructures are studied by scanning electron microscopy (SEM), scanning transmission electron microscopy (STEM) and energy dispersive X-ray spectroscopy in STEM (EDX-STEM). High density one-dimensional arrays of these nanotubes formed on FTO substrates are applied as photoanode in a dye-sensitized solar cell (DSC). The evolution of the dye adsorption capacity and solar cells parameters are explored as a function of the crystallinity and thickness of the TiO2 shell. The results show the critical effect of a full coverage by TiO2 of ZnO core to explain the mixed results found in the literature.

Surface chemistry and germination improvement of Quinoa seeds subjected to plasma activation

Gomez-Ramierez, A.; Lopez-Santos, C.; Cantos, M.; Garcia, J. L.; Molina, R.; Cotrino, J.; Espinos, J. P.; Gonzalez-Elipe, A. R.

Scientific Reports, **7** (2017) art. 5924

Julio, 2017 | **DOI:** 10.1038/s41598-017-06164-5

Plasma treatment is recognized as a suitable technology to improve germination efficiency of numerous seeds. In this work Quinoa seeds have been subjected to air plasma treatments both at atmospheric and low pressure and improvements found in germination rate and percentage of success. Seed water uptake by exposure to water vapor, although slightly greater for plasma treated seeds, did not justify the observed germination improvement. To identify other possible factors contributing to germination, the chemical changes experienced by outer parts of the seed upon plasma exposure have been investigated by X-ray photoemission spectroscopy (XPS) and scanning electron microscopy (SEM-EDX). XPS revealed that the outer layers of the Quinoa plasma treated seeds were highly oxidized and appeared enriched in potassium ions and adsorbed nitrate species. Simultaneously, SEM-EDX showed that the enrichment in potassium and other mineral elements extended to the seed pericarp and closer zones. The disappearance from the surface of both potassium ions and nitrate species upon exposure of the plasma treated seeds to water vapor is proposed as a factor favoring germination. The use of XPS to study chemical changes at seed surfaces induced by plasma treatments is deemed very important to unravel the mechanisms contributing to germination improvement.

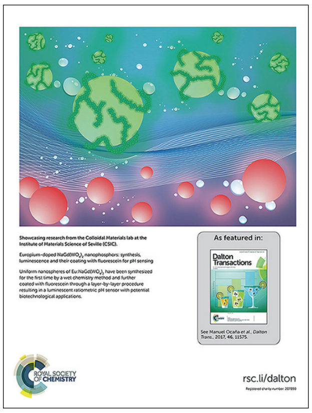
Crystal structure, NIR luminescence and X-ray computed tomography of Nd3+:Ba0.3Lu0.7F2.7 nanospheres

Gonzalez-Mancebo, D; Becerro, AI; Cantelar, E; Cusso, F; Briat, A; Boyer, D; Ocana, M

Dalton Transactions, **46** (2017) 6580-6587

Agosto, 2017 | **DOI:** 10.1039/c7dt00453b

Uniform, hydrophilic 50 nm diameter Nd3+-doped Ba0.3Lu0.7F2.7 nanospheres are synthesized at 120 degrees C using a singular one-pot method based on the use of ethylene glycol as solvent, in the absence of any additive. The composition and crystal structure of the undoped material are analyzed in detail using ICP and XRD, which reveals a BaF2 cubic crystal structure that is able to incorporate 70 mol% of Lu ions. This finding contrasts with the reported phase diagram of the system, where the maximum solubility is around 30 mol% Lu. XRD proves as well that the Ba0.3Lu0.7F2.7 structure is able to incorporate Nd3+ ions up to, at least 10 mol%, without altering the uniform particles morphology. The Nd-doped particles exhibit near-infrared luminescence when excited at 810 nm. The maximum emission intensity with the minimum concentration quenching effect is obtained at 1.5% Nd doping level. X-ray computed tomography experiments are carried out on powder samples of the latter composition. The sample significantly absorbs X-ray photons, thus demonstrating that the Nd3+-doped Ba0.3Lu0.7F2.7 nanospheres are good candidates as contrast agents in computed tomography.



Showcasing research from the Colloidal Materials lab at the Institute of Materials Science of Seville (CSIC).

Europium-doped $NaGd(WO_4)_2$ nanophosphors: synthesis, luminescence and their coating with fluorescein for pH sensing

Uniform nanospheres of $Eu:NaGd(WO_4)_2$ have been synthesized for the first time by a wet chemistry method and further coated with fluorescein through a layer-by-layer procedure resulting in a luminescent ratiometric pH sensor with potential biotechnological applications.

As featured in:

See Manuel Ocaña et al., *Dalton Trans.*, 2017, 46, 11575.

rsc.li/dalton

Registered charity number: 207890

Europium-doped NaGd(WO4)(2) nanophosphors: synthesis, luminescence and their coating with fluorescein for pH sensing

Laguna, M; Escudero, A; Nuñez, NO; Becerro, AI; Ocaña, M

Dalton Transactions, **46** (2017) 11575-11583

Septiembre, 2017 | **DOI:** 10.1039/c7dt01986f

Uniform Eu-doped NaGd(WO4)(2) nanophosphors with a spherical shape have been synthesized for the first time by using a wet chemistry method based on a homogeneous precipitation process at low temperature (120 degrees C) in ethylene glycol/water mixtures. The obtained nanoparticles crystallized into the tetragonal structure and presented polycrystalline character. The europium content in such phosphors has been optimized through the analysis of the luminescence dynamics (lifetime measurements). By coating the Eu3+-doped wolframate based nanoparticles with fluorescein through a layer-by-layer (LbL) approach, a wide range (4-10) ratiometric pH-sensitive sensor has been developed, which uses the pH insensitive emission of Eu3+ as a reference.

In Situ Determination of the Water Condensation Mechanisms on Superhydrophobic and Superhydrophilic Titanium Dioxide Nanotubes

Macias-Montero, Manuel; Lopez-Santos, Carmen; Nicolas Filippin, A.; Rico, Victor J.; Espinos, Juan P.; Fraxedas, Jordi; Perez-Dieste, Virginia; Escudero, Carlos; Gonzalez-Elipe, Agustin R.; Borras, Ana

Langmuir, **33** (2017) 6449-6456

Junio, 2017 | **DOI:** 10.1021/acs.langmuir.7b00156

One-dimensional (1D) nanostructured surfaces based on high-density arrays of nanowires and nanotubes of photoactive titanium dioxide (TiO2) present a tunable wetting behavior from superhydrophobic to superhydrophilic states. These situations are depicted in a reversible way by simply irradiating with ultraviolet light (superhydrophobic to superhydrophilic) and storage in dark. In this article, we combine in situ environmental scanning electron microscopy (ESEM) and near ambient pressure photoemission analysis (NAPP) to understand this transition. These experiments reveal complementary information at microscopic and atomic level reflecting the surface wettability and chemical state modifications experienced by these 1D surfaces upon irradiation. We pay special attention to the role of the water condensation mechanisms and try to elucidate the relationship between apparent water contact angles of sessile drops under ambient conditions at the macroscale with the formation of droplets by water condensation at low temperature and increasing humidity on the nanotubes surfaces. Thus, for the as-grown nanotubes, we reveal a metastable and superhydrophobic Cassie state for sessile drops that tunes toward water dropwise condensation at the microscale compatible with a partial hydrophobic Wenzel state. For the UV-irradiated surfaces, a filmwise wetting behavior is observed for both condensed water and sessile droplets. NAPP analyses show a hydroxyl accumulation on the as-grown nanotubes surfaces during the exposure to water condensation conditions, whereas the water filmwise condensation on a previously hydroxyl enriched surface is proved for the superhydrophilic counterpart.

Enhanced green fluorescent protein in optofluidic Fabry-Perot microcavity to detect laser induced temperature changes in a bacterial culture

Lahoz, F; Martin, IR; Walo, D; Freire, R; Gil-Rostra, J; Yubero, F; Gonzalez-Elipe, AR
Applied Physics Letters, **111** (2017) 111103
Septiembre, 2017 | **DOI:** 10.1063/1.4990870

Thermal therapy using laser sources can be used in combination with other cancer therapies to eliminate tumors. However, high precision temperature control is required to avoid damage in healthy surrounding tissues. Therefore, in order to detect laser induced temperature changes, we have used the fluorescence signal of the enhanced Green Fluorescent Protein (eGFP) overexpressed in an E. coli bacterial culture. For that purpose, the bacteria expressing eGFP are injected in a Fabry-Perot (FP) optofluidic planar microcavity. In order to locally heat the bacterial culture, external infrared or ultraviolet lasers were used. Shifts in the wavelengths of the resonant FP modes are used to determine the temperature increase as a function of the heating laser pump power. Laser induced local temperature increments up to 6-7 degrees C were measured. These results show a relatively easy way to measure laser induced local temperature changes using a FP microcavity and using eGFP as a molecular probe instead of external nanoparticles, which could damage/alter the cell. Therefore, we believe that this approach can be of interest for the study of thermal effects in laser induced thermal therapies.

Formation of nitrile species on Ag nanostructures supported on a-Al2O3: a new corrosion route for silver exposed to the atmosphere

Pelaez, RJ; Espinos, JP; Afonso, CN
Nanotechnology, **28** (2017) 175709
Abril, 2017 | **DOI:** 10.1088/1361-6528/aa65c0

The aging of supported Ag nanostructures upon storage in ambient conditions (air and room temperature) for 20 months has been studied. The samples are produced on glass substrates by pulsed laser deposition (PLD); first a 15 nm thick buffer layer of amorphous aluminum oxide (a-Al2O3) is deposited, followed by PLD of Ag. The amount of deposited Ag ranges from that leading to a discontinuous layer up to an almost-percolated layer with a thickness of <6 nm. Some regions of the as-grown silver layers are converted, by laser induced dewetting, into round isolated nanoparticles (NPs) with diameters of up to ~25 nm. The plasmonic, structural and chemical properties of both as-grown and laser exposed regions upon aging have been followed using extinction spectroscopy, scanning electron microscopy and x-ray photoelectron spectroscopy, respectively. The results show that the discontinuous as-grown regions are optically and chemically unstable and that the metal becomes oxidized faster, the smaller the amount of Ag. The corrosion leads to the formation of nitrile species due to the reaction between NO x species from the atmosphere adsorbed at the surface of Ag, and hydrocarbons adsorbed in defects at the surface of the a-Al2O3 layer during the deposition of the Ag nanostructures by PLD that migrate to the surface of the metal with time. The nitrile formation thus results in the main oxidation mechanism and inhibits almost completely the formation of sulphate/sulphide. Finally, the optical changes upon aging offer an easy-to-use tool for following the aging process. They are dominated by an enhanced absorption in the UV side of the spectrum and a blue-shift of the surface plasmon resonance that are, respectively, related to the formation of a dielectric overlayer on the Ag nanostructure and changes in the dimensions/features of the nanostructures, both due to the oxidation process.

Structural control in porous/compact multilayer systems grown by magnetron sputtering

Garcia-Valenzuela, A; Lopez-Santos, C; Alvarez, R; Rico, V; Cotrino, J; Gonzalez-Elipe, AR; Palmero, A

Nanotechnology, **28** (2017) 46

Noviembre, 2017 | **DOI:** 10.1088/1361-6528/aa8cf4

In this work we analyze a phenomenon that takes place when growing magnetron sputtered porous/compact multilayer systems by alternating the oblique angle and the classical configuration geometries. We show that the compact layers develop numerous fissures rooted in the porous structures of the film below, in a phenomenon that amplifies when increasing the number of stacked layers. We demonstrate that these fissures emerge during growth due to the high roughness of the porous layers and the coarsening of a discontinuous interfacial region. To minimize this phenomenon, we have grown thin interlayers between porous and compact films under the impingement of energetic plasma ions, responsible for smoothing out the interfaces and inhibiting the formation of structural fissures. This method has been tested in practical situations for compact TiO2/porous SiO2 multilayer systems, although it can be extrapolated to other materials and conditions.

Morphology control of uniform CaMoO4 microarchitectures and development of white light emitting phosphors by Ln doping (Ln = Dy3+, Eu3+)

Laguna, Mariano; Nuñez, Nuria O.; Becerro, Ana I.; Ocaña, Manuel
Crystengcomm, **19** (2017) 1590-1600
Marzo, 2017 | **DOI:** 10.1039/c6ce02611g

A very simple synthesis procedure based on precipitation reactions at moderate temperature (120 degrees C) from solutions containing calcium nitrate and sodium molybdate, using mixed solvents (polyols and water) has been developed, which produces uniform tetragonal CaMoO4 microarchitectures with different morphologies (peanuts, cocoons, spindles and spheres) composed of self-assembled entities. The morphology and crystal size of such assemblies could be tuned by a simple change of the nature of the components of the solvent mixture or their volumetric ratio in such a mixture. All particles presented similar excitation and emission spectra arising from a charge transfer process within the MoO4 2-groups. The emitted light presented a bluish-green color and its intensity was higher for the spindle-type particles. This synthesis procedure was also suitable for doping peanut-like CaMoO4 architectures with Eu3+ or Dy3+ cations up to a 1% molar ratio (Ln/Ln + Ca), without altering their morphology or crystalline structure. The so prepared phosphors emitted an intense red (Eu-doped) or greenish (Dy-doped) light when excited through the MoO42- group excitation band, indicating the presence of an energy transfer process from such groups to the Ln(3+) cations. Finally, a white light emitting phosphor with chromaticity coordinates x = 0.335 and y = 0.365 and a correlated color temperature of 5407 K was developed by codoping peanut-type CaMoO4 particles with suitable amounts of Dy3+ (0.35%) and Eu3+ (0.15%) cations, which could find applications in white light emitting diodes.

Effect of acid-treatment and colloidal-processing conditions on the room temperature mechanical and electrical properties of 3YTZP/MWNT ceramic nanocomposites

Poyato, R.; Morales-Rodríguez, A.; Gutiérrez-Mora, F.; Muñoz, A.; Gallardo-López, A.
Ceramics International, **43** (2017) 16560-16568
Diciembre, 2017 | **DOI:** 10.1016/j.ceramint.2017.09.043

Different colloidal powder processing routines have been used to prepare composites of 3 mol% Y2O3 -ZrO2 (tetragonal zirconia polycrystals, 3YTZP) with 2.5 vol% multiwall carbon nanotubes (MWNT) with the aim of achieving a homogeneous distribution of the MWNTs in the ceramic, eliminating agglomerates but also minimizing carbon nanotube (CNT) damage during processing. Modifications of the acid treatment applied to the nanotubes, including subjecting them to stirring or ultrasonic agitation, and use of acid or basic pH during composite powder mixing have been approached.
No MWNT damage during processing was detected by Raman spectroscopy. CNT bundles were found in all the composites forming different patterns depending on the processing route. Similar values of hardness were obtained for all the composites, while different anisotropy in fracture propagation was found when studying parallel and perpendicular directions to the sintering pressing axis on the cross sections of the composites due to the MWNT preferential

alignment. The CNT bundles were found to act as fracture short paths. A similar anisotropic behavior was observed for the electrical conductivity. These results have been correlated to the different microstructures obtained in the composites prepared with different processing routines.

Fabrication of black-gold coatings by glancing angle deposition with sputtering

Vitrey, A; Alvarez, R; Palmero, A; Gonzalez, MU; Garcia-Martin, JM
Beilstein Journal of Nanotechnology, **8** (2017) 434-439
Febrero, 2017 | **DOI:** 10.3762/bjnano.8.46

The fabrication of black-gold coatings using sputtering is reported here. Glancing angle deposition with a rotating substrate is needed to obtain vertical nanostructures. Enhanced light absorption is obtained in the samples prepared in the ballistic regime with high tilt angles. Under these conditions the diameter distribution of the nanostructures is centered at about 60 nm and the standard deviation is large enough to obtain black-metal behavior in the visible range.

In Vitro and in Vivo Study of Poly(Lactic-co-Glycolic) (PLGA) Membranes Treated with Oxygen Plasma and Coated with Nanostructured Hydroxyapatite Ultrathin Films for Guided Bone Regeneration Processes

Torres-Lagares, D; Castellanos-Cosano, L; Serrera-Figallo, MA; Garcia-Garcia, FJ; Lopez-Santos, C; Barranco, A; Elipe, ARG; Rivera-Jimenez, C; Gutierrez-Perez, JL
Polymers, **9** (2017) art. 410
Septiembre, 2017 | **DOI:** 10.3390/polym9090410

The novelty of this study is the addition of an ultrathin layer of nanostructured hydroxyapatite (HA) on oxygen plasmamodified poly(lactic-co-glycolic) (PLGA) membranes (PO2) in order to evaluate the efficiency of this novel material in bone regeneration. Methods: Two groups of regenerative membranes were prepared: PLGA (control) and PLGA/PO2/HA (experimental). These membranes were subjected to cell cultures and then used to cover bone defects prepared on the skulls of eight experimental rabbits. Results: Cell morphology and adhesion of the osteoblasts to the membranes showed that the osteoblasts bound to PLGA were smaller and with a lower number of adhered cells than the osteoblasts bound to the PLGA/PO2/HA membrane ($p < 0.05$). The PLGA/PO2/HA membrane had a higher percentage of viable cells bound than the control membrane ($p < 0.05$). Both micro-CT and histological evaluation confirmed that PLGA/PO2/HA membranes enhance bone regeneration. A statistically significant difference in the percentage of osteoid area in relation to the total area between both groups was found. Conclusions: The incorporation of nanometric layers of nanostructured HA into PLGA membranes modified with PO2 might be considered for the regeneration of bone defects. PLGA/PO2/HA membranes promote higher osteosynthetic activity, new bone formation, and mineralisation than the PLGA control group.

About the enhancement of chemical yield during the atmospheric plasma synthesis of ammonia in a ferroelectric packed bed reactor

Gomez-Ramirez, Ana; Montoro-Damas, Antonio M.; Cotrino, Jose; Lambert, Richard M.; Gonzalez-Elipe, Agustin R.
Plasma Processes and Polymers, **14** (2017) e1600081
Junio, 2017 | **DOI:** 10.1002/ppap.201600081

Plasma reactions offer an attractive alternative route for the synthesis of a variety of valuable chemical compounds. Here we investigate the parameters that determine the efficiency of ammonia synthesis in a ferroelectric packed bed dielectric barrier discharge (DBD) reactor. The effects of varying the operating frequency, the size of the ferroelectric pellets and the inter-electrode distance have been systematically studied. Under optimised conditions nitrogen conversions in excess of 7% were achieved, higher than those previously obtained using DBD reactors. These findings are discussed with respect to variations in the electrical characteristics of the reactor under operating conditions and in the light of emission spectra obtained as a function of reactant flow rates. These encouraging results signpost future developments that could very substantially improve the efficiency of ammonia synthesis by means of DBD technology.

Micron-scale wedge thin films prepared by plasma enhanced chemical vapor deposition

Lopez-Santos, M.C.; Alvarez, R.; Palmero, A.; Borras, A.; del Campo, R.C.; Holgado, M.; Gonzalez-Elipe, A.R.
Plasma Processes and Polymers, **14** (2017) e1700043
Diciembre, 2017 | **DOI:** 10.1002/ppap.201700043

Wedge-shaped materials are currently employed for optical analyses and sensing applications. In this paper, we present an easy to implement plasma enhanced chemical vapor deposition procedure to grow wedge-shaped thin films with controlled slope at the scale of few hundred microns. The method relies on the use of few tenths micron height obstacles to alter the laminar flow of precursor gas during deposition and is applied for the fabrication of wedge-shaped ZnO thin films. Local interference patterns, refractive index, and birefringence of the films have been measured with one micron resolution using a specially designed optical set-up. Their micro- and nano-structures have been characterized by means of scanning electron microscopy and theoretically reproduced by Monte Carlo calculations.

Regenerative Endodontic Procedures: A Perspective from Stem Cell Niche Biology

M. Marí-Beffa, J.J. Segura-Egea, A. Díaz-Cuenca
Journal of Endodontics, **43** (2017) 52-62
Enero, 2017 | **DOI:** 10.1016/j.joen.2016.09.011

Introduction
Endodontics uses cell therapy strategies to treat pulpal and periapical diseases. During these therapies, surgeons aim to reconstruct the natural microenvironments that regulate the activity of dental stem cells.
Methods
We searched for more than 400 articles in PubMed using key words from regenerative endodontics and dental stem cell biology. In 268 articles, we reviewed what factors may influence histologic results after preclinical dental treatments that use regenerative endodontic procedures after pulpectomy.
Results
Several factors, such as the origin of stem cells, the biomimicry of scaffolds used, and the size of lesions, are considered to influence the histologic appearance of the regenerated pulp-dentin complex after treatments. Information is accumulating on transcription factors that generate the pulp-dentin complex and survival/trophic factors that would benefit niche recovery and histologic results.
Conclusions
In this article, we discuss the noninterchangeability of stem cells, the influence of dentin-entrapped molecule release on pulp regeneration and survival of stem cells, and the need of positional markers to assess treatments histologically. The ex vivo amplification of appropriate dental stem cells, the search for scaffolds storing the molecular diversity entrapped in the dentin, and the use of positional transcription factors as histologic markers are necessary to improve future preclinical experiments.

Optical Gas Sensing of Ammonia and Amines Based on Protonated Porphyrin/TiO2 Composite Thin Films

Castillero, Pedro; Roales, Javier; Lopes-Costa, Tania; Sanchez-Valencia, Juan R.; Barranco, Angel; Gonzalez-Elipe, Agustin R.; Pedrosa, Jose M.
Sensor, **17** (2017) 24
Enero, 2017 | **DOI:** 10.3390/s17010024

Open porous and transparent microcolumnar structures of TiO2 prepared by physical vapour deposition in glancing angle configuration (GLAD-PVD) have been used as host matrices for two different fluorescent cationic porphyrins, 5-(N-methyl 4-pyridyl)-10,15,20-triphenyl porphine chloride (MMPyP) and meso-tetra (N-methyl 4-pyridyl) porphine tetrachloride (TMPyP). The porphyrins have been anchored by electrostatic interactions to the microcolumns by self-assembly through the dip-coating method. These porphyrin/TiO2 composites have been used as gas sensors for ammonia and amines through previous protonation of the porphyrin with HCl followed by subsequent exposure to the basic analyte. UV-vis absorption, emission, and time-resolved spectroscopies have been used to confirm the protonation-deprotonation of the two porphyrins and to follow their spectral changes in the presence of the analytes. The monocationic porphyrin has been found to be more sensible (up to 10 times) than its tetracationic counterpart. This result has been attributed to the different anchoring arrangements of the two porphyrins to the TiO2 surface and their different states of aggregation within the film. Finally, there was an observed decrease of the emission fluorescence intensity in consecutive cycles of exposure and recovery due to the formation of ammonium chloride inside the film.

Preparation and Optimization of Fluorescent Thin Films of Rosamine-SiO2/TiO2 Composites for NO2 Sensing

Guillen, MG; Gamez, F; Suarez, B; Queiros, C; Silva, AMG; Barranco, A; Sanchez-Valencia, JR; Pedrosa, JM; Lopes-Costa, T
Materials, **10** (2017) art. 124
Febrero, 2017 | **DOI:** 10.3390/ma10020124

The incorporation of a prototypical rosamine fluorescent dye from organic solutions into transparent and microstructured columnar TiO2 and SiO2 (MO2) thin films, prepared by evaporation at glancing angles (GAPVD), was evaluated. The aggregation of the adsorbed molecules, the infiltration efficiency and the adsorption kinetics were studied by means of UV-Vis absorption and fluorescence spectroscopies. Specifically, the infiltration equilibrium as well as the kinetic of adsorption of the emitting dye has been described by a Langmuir type adsorption isotherm and a pseudosecond order kinetic model, respectively. The anchoring mechanism of the rosamine to the MO2 matrix has been revealed by specular reflectance Fourier transform infrared spectroscopy and infiltration from aqueous solutions at different pH values. Finally, the sensing performance towards NO2 gas of optimized films has been assessed by following the changes of its fluorescence intensity revealing that the so-selected device exhibited improved sensing response compared to similar hybrid films reported in the literature.

Antibacterial response of titanium oxide coatings doped by nitrogen plasma immersion ion implantation

Esparza, J; Fuentes, GF; Bueno, R; Rodriguez, R; Garcia, JA; Vitas, AI; Rico, V; Gonzalez-Elipe, AR
Surface and Coatings Technology, **314** (2017) 67–71
Marzo, 2017 | **DOI:** 10.1016/j.surfcoat.2016.11.002

Plasma immersion ion implantation technology has been utilized to enhance the photocatalytic activity of the anatase phase of TiO2 thin films deposited by cathodic arc evaporation PVD. The main objective of this study is to shift the light absorbance of the titania in order to obtain antibacterial activity under visible light irradiation. TiO2 thin films, deposited on polished stainless steel AISI 304 and silicon wafers, were implanted with nitrogen ions (N+/N2+) at 20 kV energy and different temperatures between 250 and 350 °C. The antibacterial activity of nitrogen implanted titania coatings has been monitored for Escherichia coli under visible light irradiation. Additionally ultra violet/visible spectrophotometry tests have been carried out to measure the changes in the light absorbance of the doped films. Further characterization has been performed, including X-ray photoelectron spectroscopy, X-ray diffraction and glow discharge optical emission spectrometry. As a result of Nitrogen implantation, the light absorption peak shifted from ultra violet region (UV-A) to visible wavelength range, which led to an increase of the antibacterial efficacy under visible light irradiation.

A compact and portable optofluidic device for detection of liquid properties and label-free sensing

Lahoz, F; Martin, IR; Walo, D; Gil-Rostra, J; Yubero, F; Gonzalez-Elipe, AR

Journal of Physics D: Applied Physics, **50** (2017) 21

Junio, 2017 | **DOI:** 10.1088/1361-6463/aa6cdd

Optofluidic lasers have been widely investigated over the last few years mainly because they can be easily integrated in sensor devices. However, high power pulse lasers arc required as excitation sources, which, in practice, limit the portability of the system. Trying to overcome some of these limitations, in this paper we propose the combined use of a small CW laser with a Fabry-Perot optofluidic planar microcavity showing high sensitivity and versatility for detection of liquid properties and label-free sensing. Firstly, a fluorescein solution in ethanol is used to demonstrate the high performances of the FP microcavity as a temperature sensor both in the laser (high pump power above laser threshold) and in the fluorescence (low pump power) regimes. A shift in the wavelength of the resonant cavity modes is used to detect changes in the temperature and our results show that high sensitivities could be already obtained using cheap and portable CW diode lasers. In the second part of the paper, the demonstration of this portable device for label-free sensing is illustrated under low CW pumping. The wavelength positions of the optolluidic resonant modes are used to detect glucose concentrations in water solutions using a protein labelled with a fluorescent dye as the active medium.

Microemulsion-Mediated Synthesis and Properties of Uniform Ln:CaWO4 (Ln = Eu, Dy) Nanophosphors with Multicolor Luminescence for Optical and CT Imaging

Laguna, M; Nuñez, NO; Garcia, FJ; Corral, A; Parrado-Gallego, A; Balcerzyk, M; Becerro, AI; Ocaña, M

European Journal of Inorganic Chemistry, **44** (2017) 5158-5168

Diciembre, 2017 | **DOI:** 10.1002/ejic.201700650

A new room-temperature method has been developed that yields, for the first time in the literature, uniform and well-dispersed CaWO4 nanospindles. This method is based on the use of microemulsions consisting of aqueous solutions of Ca2+ and WO42- precursors, cyclohexane as the organic medium, Triton X-100 as the surfactant, and n-octanol as the cosurfactant. We show that the formation of uniform nanospindles requires a restrictive set of experimental conditions. These particles crystallize into the tetragonal CaWO4 phase and emit blue-green luminescence when excited by UV radiation. The reported method is also useful for doping the CaWO4 spindles with Eu3+ or Dy3+ cations, resulting in multicolor emissions (red for Eu3+; white for Dy3+). The luminescence is much stronger when excited through a WO42--Ln(3+) (Ln = Eu or Dy) energy-transfer band than through the f-f transition bands of the Ln(3+) cations. Interestingly, because of the white luminescence associated with the Dy:CaWO4 nanophosphor, it might be useful for LED technologies. Luminescence dynamics and energy-transfer efficiency have been analyzed to determine the optimum phosphors. Finally, the Eu-doped CaWO4 nanospindles also showed excellent X-ray attenuation efficacy, which confers double functionality to this material as both a luminescence bioprobe and as a contrasting agent for X-ray computed-tomography.

Optical properties and electronic transitions of zinc oxide, ferric oxide, cerium oxide, and samarium oxide in the ultraviolet and extreme ultraviolet

Pauly, N; Yubero, F; Espinos, JP; Tougaard, S
Applied Optics, **56** (2017) 6611-6621
Agosto, 2017 | **DOI:** 10.1364/AO.56.006611

Optical properties and electronic transitions of four oxides, namely zinc oxide, ferric oxide, cerium oxide, and samarium oxide, are determined in the ultraviolet and extreme ultraviolet by reflection electron energy loss spectroscopy using primary electron energies in the range 0.3 - 2.0 keV. This technique allows the evaluation of the optical response in these ultraviolet spectral regions of a thin layer of material, and the analysis is straightforward. It is performed within the dielectric response theory by means of the QUEELS-epsilon(k,omega)-REELS software developed by Tougaard and Yubero [Surf. Interface Anal. 36, 824 (2004)]. The method consists basically in the fitting of experimentally determined single-scattering electron energy loss cross sections with a parametric energy loss function of the corresponding material, to the one calculated within a dielectric response formalism. The obtained refractive index and extinction coefficients, as well as the identified electronic transitions are compared, when available, with previously published results.

Vapor and liquid optical monitoring with sculptured Bragg microcavities

Oliva-Ramirez, M; Gil-Rostra, J; Lopez-Santos, MC; Gonzalez-Elipe, AR; Yubero, F
Journal of Nanophotonics, **11** (2017) 046009
Octubre, 2017 | **DOI:** 10.1117/1.JNP.11.046009

Sculptured porous Bragg microcavities (BMs) formed by the successive stacking of columnar SiO2 and TiO2 thin films with a zig-zag columnar microstructure are prepared by glancing angle deposition. These BMs act as wavelength-dependent optical retarders. This optical behavior is attributed to a self-structuration of the stacked layers involving the lateral association of nanocolumns in the direction perpendicular to the main flux of particles during the multilayer film growth, as observed by focused ion beam scanning electron microscopy. The retardance of these optically active BMs can be modulated by dynamic infiltration of their open porosity with vapors, liquids, or solutions with different refractive indices. The tunable birefringence of these nanostructured photonic systems has been successfully simulated with a simple model that assumes that each layer within the BMs stack has uniaxial birefringence. The sculptured BMs have been incorporated as microfluidic chips for optical transduction for label-free vapor and liquid sensing. Several examples of the detection performance of these chips, working either in reflection or transmission configuration, for the optical monitoring of vapor and liquids of different refractive indices and aqueous solutions of glucose flowing through the microfluidic chips are described.

On the effect of wall slip on the determination of the yield stress of magnetorheological fluids

Caballero-Hernandez, J; Gomez-Ramirez, A; Duran, JDG; Gonzalez-Caballero, F; Zubarev, AY; Lopez-Lopez, MT
Applied Rheology, **27** (2017) 15001 (8 pages)
Marzo, 2017 | **DOI:** 10.3933/ApplRheol-27-15001

We study the effect of wall slip on the measured values of the yield stress of magnetorheological (MR) fluids. For this aim we used a rheometer provided with parallel-plate geometries of two types, distinguished by having smooth or rough surfaces. We found that wall slip led to the underestimation of the yield stress when measuring geometries with smooth surfaces were used, and that this underestimation was more pronounced for the static than for the dynamic yield stress. Furthermore, we analysed the effect that both irreversible particle aggregation due to colloidal interactions and reversible magnetic fieldinduced particle aggregation had on the underestimation provoked by wall slip. We found that the higher the degree of aggregation the stronger the underestimation of the yield stress. At low intensity of the applied magnetic field irreversible particle aggregation was dominant and, thus, the underestimation of the yield stress was almost negligible for well-dispersed MR fluids, whereas it was rather pronounced for MR fluids suffering from irreversible aggregation. As the magnetic field was increased the underestimation of the yield stress became significant even for the best dispersed MR fluid.

Effect of Nickel and Magnesium on the Electrochemical Behavior of AA 1050 Alloys in Nitric Acid Solution

Caballero-Hernandez, J; Gomez-Ramirez, A; Duran, JDG; Gonzalez-Caballero, F; Zubarev, AY; Lopez-Lopez, MT
Applied Rheology, **27** (2017) 15001 (8 pages)
Marzo, 2017 | **DOI:** 10.3933/ApplRheol-27-15001

We study the effect of wall slip on the measured values of the yield stress of magnetorheological (MR) fluids. For this aim we used a rheometer provided with parallel-plate geometries of two types, distinguished by having smooth or rough surfaces. We found that wall slip led to the underestimation of the yield stress when measuring geometries with smooth surfaces were used, and that this underestimation was more pronounced for the static than for the dynamic yield stress. Furthermore, we analysed the effect that both irreversible particle aggregation due to colloidal interactions and reversible magnetic fieldinduced particle aggregation had on the underestimation provoked by wall slip. We found that the higher the degree of aggregation the stronger the underestimation of the yield stress. At low intensity of the applied magnetic field irreversible particle aggregation was dominant and, thus, the underestimation of the yield stress was almost negligible for well-dispersed MR fluids, whereas it was rather pronounced for MR fluids suffering from irreversible aggregation. As the magnetic field was increased the underestimation of the yield stress became significant even for the best dispersed MR fluid.

Reliability of new poly (lactic-co-glycolic acid) membranes treated with oxygen plasma plus silicon dioxide layers for pre-prosthetic guided bone regeneration processes

Castillo-Dali, G; Castillo-Oyague, R; Batista-Cruzado, A; Lopez-Santos, C; Rodriguez-Gonzalez-Elipe, A; Saffar, JL; Lynch, CD; Gutierrez-Perez, JL; Torres-Lagares, D
Medicina Oral Patología Oral y Cirugía Oral, **22** (2017) E242-E250
Marzo, 2017 | **DOI:** 10.4317/medoral.21512

Background: The use of cold plasmas may improve the surface roughness of poly(lactic-co-glycolic) acid (PLGA) membranes, which may stimulate the adhesion of osteogenic mediators and cells, thus accelerating the biodegradation of the barriers. Moreover, the incorporation of metallic-oxide particles to the surface of these membranes may enhance their osteoinductive capacity. Therefore, the aim of this paper was to evaluate the reliability of a new PLGA membrane after being treated with oxygen plasma (PO2) plus silicon dioxide (SiO2) layers for guided bone regeneration (GBR) processes.
Material and Methods: Circumferential bone defects (diameter: 11 mm; depth: 3 mm) were created on the top of eight experimentation rabbits' skulls and were randomly covered with: (1) PLGA membranes (control), or (2) PLGA/ PO2/SiO2 barriers. The animals were euthanized two months afterwards. A micromorphologic study was then performed using ROI (region of interest) colour analysis. Percentage of new bone formation, length of mineralised bone, concentration of osteoclasts, and intensity of ostheosynthetic activity were assessed and compared with those of the original bone tissue. The Kruskal-Wallis test was applied for between-group com asignificance level of a=0.05 was considered.
Results: The PLGA/ PO2/SiO2 membranes achieved the significantly highest new bone formation, length of miner-alised bone, concentration of osteoclasts, and ostheosynthetic activity. The percentage of regenerated bone supplied by the new membranes was similar to that of the original bone tissue. Unlike what happened in the control group, PLGA/PO2/SiO2 membranes predominantly showed bone layers in advanced stages of formation. Conclusions: The addition of SiO2 layers to PLGA membranes pre-treated with PO2 improves their bone-regeneration potential. Although further research is necessary to corroborate these conclusions in humans, this could be a promising strategy to rebuild the bone architecture prior to rehabilitate edentulous areas.

Luminiscent Eu-doped $GdVO_4$ nanocrystals as optical markers for anti-Counterfeiting purposes

Moretti, E; Pizzol, G; Fantin, M; Enrichi, F; Scopece, P; Ocana, M; Polizzi, S
Chemical Papers, **71** (2017) 149-159
Enero, 2017 | **DOI:** 10.1007/s11696-016-0081-8

Luminescent Eu: $GdVO_4$ nanoparticles, with an average size of 60 nm, were deposited first on monocrystalline silicon wafers, then on four different natural stone materials, by a spray-coating technique and a silica layer was subsequently deposited by atmospheric pressure plasma jet to protect the luminescent layer and improve its adhesion to the substrate. The luminescent films were characterized by photoluminescence excitation and emission, while the surface morphology was examined by FEGSEM microscopy and spectroscopic ellipsometry to determine the coating thickness. The optical appearance of the coatings was also evaluated by colorimetric measurements and the efficacy of the fixing action of the silica layer was estimated by PL measurements performed before and after a Scotch TM tape peeling test. The proposed methodology, easily applied on the surface of stone supports, has led to the realization of a luminescent film displaying good mechanical properties, transparent and undetectable in the presence of visible light, but easily activated by UV light source, indicating that the Eu: GdVO4 nanophosphors could be used as luminescent nanotags for a reliable anti-counterfeiting technology.

High surface area biopolymeric-ceramic scaffolds for hard tissue engineering

Romero-Sanchez, LB; Borrego-Gonzalez, S; Diaz-Cuenca, A
Biomedical Physics & Engineering Express, **3** (2017) art UNSP 035012
Junio, 2017 | **DOI:** 10.1088/2057-1976/aa7001

The development of scaffolds mimicking native bone tissue composition and structure is a challenge in bone tissue engineering. 3D scaffolds with both an interconnected macropore structure and nanotextured surfaces are required. However, 3D scaffolds processed by microfabrication usually lack of nanotextured surface, while nanotextured materials generated by bottom-up nanofabrication are difficult to process conforming scaffolds having well interconnected microsized cavities. In this work, the processing of reticulated (macropore interconnected) structures using nanostructured precursors has been performed to improve the mechanical properties of the scaffolds. The application of a fibrillar collagen coating, using less than 1 wt% collagen per scaffold, has allow a significant increase of the compressive strength while preserving a high surface area and nanopore accessibility. Besides, the fibrillar nanostructured collagen coating promotes hydroxyapatite mineralization. Two different collagen-coating procedures are applied showing interesting differences in terms of mechanical performance.

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

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

Research Workshop Fundamental Processes in Perovskite and Thin Film Solar Cells

2 – 3 octubre [Sevilla, España]
Hernán Míguez García [Miembro del Comité Organizador)

COMUNICACIONES / COMMUNICATIONS

SPIE Photonics West 2017

27 enero [San Francisco, California, Estados Unidos]

Deterministic control of the emission from light sources in 1D nanoporous crystals
Juan F. Galisteo-López; Hernán Míguez; Alberto Jiménez-Solano
Conferencia Invitada

Optical liquid monitoring with sculptured Bragg microcavities incorporated to microfluidic chips
F. Yubero; J. Gil-Rostra; A.R. González-Elipe; M.C. López-Santos; M. Oliva-Ramírez
Comunicación oral

The International Conference on Perovskite Thin Film Photovoltaics | ABXPV17
1 - 2 marzo [Valencia, España]

Optical design of perovskite based tandem solar cells
M. Anaya; G. Lozano; M. Calvo; H. Míguez
Comunicación oral

44th International Conference on Metallurgical Coatings and Thin Films
24 - 28 abril [San Diego, California, Estados Unidos]

How Can the Icephobicity of an Engineered Surface be Screened by Means of Simple Laboratory Testing and Characterization?
G.F. de la Fuente; L.A. Angurel; C. López-Santos; V. Rico; A. Borrás; A.R. Gonzaléz-Elipe; J. Mora; P. García; A. Agüero
Comunicación oral

PIERS. Progress In Electromagnetics Research Symposium
22 – 25 mayo [San Petersburgo, Rusia]

Theoretical Prediction of Levitation Due to Casmir Force in Dielectric Plane-Parallel Systems.
Victoria Esteso; Sol Carretero-Palacios; Hernán Míguez
Conferencia invitada

EMRS Spring Meeting
21 - 26 mayo [Estrasburgo, Francia]

Unraveling the wetting angle of one dimensional photoactive surfaces under UV irradiation
Manuel Macias-Montero; Carmen Lopez-Santos; A. Nicolas Filippin; Victor J. Rico; Juan P. Espinós; Agustin R. Gonzalez-Elipe; Ana Borras
Comunicación oral

Fundamentals and Applications of Highly Porous Coatings Grown by Vacuum and Plasma-Assisted Techniques at Oblique Angles
A. Palmero; R. Alvarez; V. Rico; A. Garcia-Valenzuela; A. R. Gonzalez-Elipe
Comunicación oral

3rd International Workshop on Functionalized Surfaces for Sensor Applications | SURFOCAP 2017

30 - 31 mayo [Besançon, Francia]

Fundamentals and Applications of Highly Porous Coatings Grown by Vacuum and Plasma-Assisted Techniques at Oblique Angles
A. Palmero
Conferencia Plenaria

11th European Symposium on Electrochemical Engineering

4 - 8 junio [Praga, República Checa]

In situ monitoring of phenomenon of Electrochemical Promotion of Catalysis by Syncrotron analysis
A. de Lucas-Consuegra; J. Gonzalez-Cobos; J. P. Espinós; V. Rico; J. R. Sanchez-Valencia; A. R. Gonzalez-Elipe; V. Peres-Dieste; C. Escudero
Comunicación oral

21st International Colloquium on Plasma Processes

26 - 30 junio [Niza, Francia]

Plasma assisted oblique angle deposition of transparent and conducting anisotropic ITO thin films
A. Barranco; J. Parra-Barranco; J.R. Sanchez-Valencia; F.J. Aparicio; F. Garcia-Garcia; F.J. Ferrer; V. Rico; C. Lopez-Santos; A. Borras; A.R. Gonzalez-Elipe
Conferencia Invitada

From one to three dimensional nanostructures by vacuum and plasma assisted methods
Ana Borras
Conferencia Invitada

5th Magnetron Ion Processing & Arc Technologies European Conference

26 - 30 junio [Niza, Francia]

Development of 1D and 3D supported nanostructures by plasma assisted vacuum deposition: from synthesis to applications
A. Borras
Conferencia Plenaria

7th International Colloids Conference

18 - 21 junio [Barcelona, España]

One-pot synthesis and PAA-functionalization of uniform Ln:LaVO4 (Ln: Eu, Nd) nanophosphors with high colloidal stability and biocompatibility
N.O. Nuñez; P. Zambrano; J. García-Sevillano; E. Cantelar; S. Rivera-Fernández; J.M. de la Fuente; M. Ocaña
Poster

iPlasmaNano-VIII, 2017

2 - 6 julio [Orleans, Francia]

Plasma Assisted OAD of In-plane Anysotropic ITO Thin Films
J. Parra-Barranco; J. R. Sanchez-Valencia; F. J. Aparicio; F. Garcia-Garcia; V. Rico; C. Lopez-Santos; A. Borras; F.J. Ferrer; A. R. Gonzalez-Elipe; A. Barranco
Conferencia Invitada

International Conference on Phenomena in Ionized Gases | ICPIG 2017

9 - 14 julio [Estoril, Portugal]

On the influence of ferroelectric materials in a packed-bed DBD reactor
A. Gómez-Ramírez; R. Álvarez; F. J. García-García; A. Palmero; A. R. González-Elípe; J. Cotrino
Comunicación oral

Characterization of a ferro-electric packed bed plasma reactor
A.M. Montoro-Damas; A. Gómez-Ramírez; V. Rico; A.R. González-Elípe; J. Cotrino
Poster

Isotope labelling: A new technique to analyse reaction mechanisms in plasma-gas processes
A. Gómez-Ramírez; A.M. Montoro-Damas; A. R. González-Elípe; J. Cotrino
Poster

Complex Nanophotonics Science Camp

25 – 28 julio [Windsor, Reino Unido]

Boosting emission through optical disorder: the Mie Glass. Design and characterization of a novel optically random medium.
José M. Miranda-Muñoz; Gabriel Lozano; Hernán Míguez.
Póster

Euromat 2017

17 – 22 septiembre [Tesalonica, Grecia]

Large área ITO nano-structuring via self-organization by femtosecond-laser irradiation
Daniel Puerto; Maria del Carmen Lopez-Santos; Camilo Florian; Jan Siegel; Jorge Gil-Rostra; Agustin R. Gonzalez-Elipe; Javier Solis
Comunicación oral

Research Workshop Fundamental Processes in Perovskite and Thin Film Solar Cells

2 - 3 octubre [Sevilla, España]

Relevance of Optical Design for Perovskite Solar Cell Devices: from Structural Color to Absorption Enhancement
Hernán Míguez
Conferencia Invitada

Vacuum sublimation of porous one-dimensional organometal halide perovskites
Juan Ramón Sánchez-Valencia; Jesús Idígoras; Ana Borrás; Francisco Javier Aparicio; Juan Antonio Anta; Angel Barranco
Conferencia Invitada

Controlled disorder for the design and fabrication of bifacial dye-sensitized solar cells with enhanced performance.
José M. Miranda-Muñoz; Sol Carretero-Palacios; Alberto Jiménez-Solano; Yuelong Li; Gabriel Lozano; Hernán Míguez
Comunicación oral

Iberian Vacuum Conference, RIVA-X

4 - 6 octubre [Bilbao, España]

Energy-Sensitive Ion- and Cathode-Luminescent Radiation-Beam Monitors Based on Multilayer Thin-Film Design
J. Gil-Rostra; F.J. Ferrer; J.P. Espinós; A.R. González-Elipe; F. Yubero
Poster

Black gold obtained by sputtering glancing angle deposition
M.U. González; A. Vitrey; R. Alvarez; A. Palmero; J.M. García-Martín
Comunicación oral

7th Spanish Workshop in Nanolithography

23 - 25 octubre [Madrid, España]

Nanostructured coatings made by oblique deposition with sputtering
M. U. González; R. Alvarez; A. Palmero; J. M. García-Martín
Comunicación oral

4th International Workshop on Plasma Science & Entrepreneurship 2017

30 noviembre – 1 diciembre [Orleans, Francia]

Plasma assisted fabrication of multilayered PLGA membranes for guided bone regeneration in oral surgery
C. López-Santos; A. Terriza; F.J. García-García; D. Torres-Lagares; A.R. González-Elipe; Angel Barranco
Conferencia Invitada

CONGRESOS Y REUNIONES NACIONALES / NATIONAL CONGRESS AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

16th World Nano Conference

5 - 6 junio [Milán, Italia]

One-pot synthesis of hydrophilic Nd3+:Ba0.3Lu0.7F2.7 nanospheres: Crystal structure, X-ray computed tomography and NIR luminescence
D. Gonzalez-Mancebo; A.I. Becerro; E. Cantelar; F. Cussó; D. Boyer; M. Ocaña
Comunicación oral

XXXVI Reunión Bienal de la Sociedad Española de Química 2017

25 - 29 junio [Barcelona, España]

Solution Processed Optical Materials for Optoelectronic Devices
Hernán Míguez
Conferencia Invitada

2nd World Congress and Expo on Materials Science & Nanoscience

25 - 27 septiembre [Valencia, España]

Spectroscopic characterization of BaGdF5:Er/Yb nanoparticles
E. Cantelar; F. Cusso; D. Gonzalez-Mancebo; A.I. Becerro; M. Ocaña
Conferencia Invitada

IX Jornadas CPAN – Encuentros de Física Nuclear

23 – 25 octubre [Santander, España]

Monitor de energía de haz de partículas ionizantes basado en emisión de color
F.J. Ferrer; J. Gil Rostra; J.P. Espinos; A.R. Gonzalez Elipe; F. Yubero
Comunicación oral

II Workshop Química Sostenible

3 noviembre [Valencia, España]

Nanomateriales ópticos para dispositivos optoelectrónicos
Hernán Míguez
Conferencia Invitada

FORMACION / TRAINING

TESIS DOCTORALES/ DOCTOR DEGREE THESIS

Título: **Diseño de la absorción y emisión ópticas de nanomateriales en entornos fotónicos controlados**
Autor: Alberto Jiménez Solano
Directores: Juan Francisco Galisteo López, Hernán Míguez García
Calificación: Sobresaliente “Cum Laude” por Unanimidad
Centro: Universidad de Sevilla
Fecha: 23 de junio de 2017

Título: **Biomateriales nanoporosos ordenados y su funcionalidad como matriz biomimética administradora de iones terapéuticos para aplicación de regeneración tisular**
Autor: Lilian Beatriz Romero Sánchez
Directora: María Aránzazu Díaz Cuenca
Calificación: Sobresaliente “Cum Laude” por Unanimidad
Centro: Universidad de Sevilla
Fecha: 20 de septiembre de 2017

Título: **Nanopartículas uniformes luminiscentes basadas en tierras raras para aplicaciones biomédicas**
Autor: Sonia Rodríguez Liviano
Directora: Manuel Ocaña Jurado

Calificación: Apto “Cum Laude”
Centro: Universidad de Sevilla
Fecha: 13 de septiembre de 2017

Formación de Graduados / Master Degree Thesis

Título: **Síntesis, Caracterización y Funcionalización de Nanopartículas de DyVO4 y HoVO4**
Autor: Alberto Pérula Jiménez
Directores: Nuria O. Núñez Alvarez
Grado: Trabajo Fin de Grado
Año Académico: 2016-2017 (27 septiembre 2017)

Título: **Dispositivos sensores basados en nanohilos orgánicos monocristalinos**
Autor: Angel Campos Lendinez
Directores: Juan Ramón Sánchez Valencia, Ana Isabel Borras Martos
Grado: Trabajo Fin de Máster
Año Académico: 2017-2018 (29 junio 2017)

Título: **Fósforos basados en M:ZnGa2=4 (M=Cr^{3+}, Mn^{2+}) para dispositivos de iluminación**
Autor: Marine Fleury
Directores: Ana Isabel Becerro Nieto, Manuel Ocaña Jurado
Grado: Trabajo Fin de Máster
Año Académico: 2017-2018 (30 junio 2017)

Título: **Fabricación y caracterización de superficies repelentes al hielo mediante tratamiento láser de metales**
Autor: Martin Villagrá Lobo
Directores: Agustín R. González-Elipe, Victor Joaquín Rico Gavira
Grado: Trabajo Fin de Máster
Año Académico: 2017-2018 (30 junio 2017)

Título: **Técnicas de deposición usando descargas de barrera dieléctrica**
Autor: Paula Navascués Garvín
Directores: José Cotrino Bautista, Ana María Gómez Ramírez
Grado: Trabajo Fin de Grado
Año Académico: 2017-2018 (13 julio 2017)

Título: **Síntesis y funcionalización de nanopartículas uniformes luminiscentes basadas en $NaLa(WO_4)_2$ y $NaLa(MoO_4)_2$**
Autor: María del Mar Cogollo de Cádiz
Directores: Nuria Ofelia Núñez Alvarez, Manuel Ocaña Jurado
Grado: Trabajo Fin de Máster
Año Académico: 2017-2018 (22 septiembre 2017)

Título: **Nanopartículas basadas en fluoruros de tierras raras con aplicaciones biotecnológicas**
Autor: Andrés Olivencia Ibáñez
Directores: Ana Isabel Becerro Nieto, Manuel Ocaña Jurado
Grado: Trabajo Fin de Máster
Año Académico: 2017-2018 (13 diciembre 2017)

DOCENCIA / TEACHING

Máster "Láser, Plasma y Tecnología de Superficies"

Tecnología de lámina delgada
Ana Isabel Borrás Martos, Agustín R. González-Elipe, Victor Joaquín Rico Gavira, María del Carmen

Nanotecnología de superficies y sus aplicaciones
Angel Barranco Quero, Francisco Javier Aparicio Rebollo, Juan Ramón Sánchez Valencia, Victor López Flores

Nuevos materiales para dispositivos (electrónicos, fotónicos, aprovechamiento energético y magnéticos)
Victor López Flores

Funcionalización de superficies para aplicaciones mecánicas, protectoras y de bioactividad controladas
María Carmen López Santos, Francisco Yubero Valencia

Nuevos materiales para dispositivos
Victor López Flores, Ana Isabel Borrás Martos

Materiales e Ingeniería de Superficies
Ana Isabel Becerro Nieto

Lugar: Universidad de Córdoba, Universidad Politécnica de Madrid, Instituto de Ciencia de Materiales de Madrid

5th Intensive School on Conservation Science: Ceramic Materials

Layers' microsctructure: influence and utility control for specific applications
Agustín R. González-Elipe

Lugar: Toledo, España

Máster Oficial y Doctorado en Biotecnología Avanzada

Nanotecnología
María Aránzazu Díaz Cuenca
Lugar: Universidad Internacional de Andalucía. Universidad de Málaga

Doble Máster en Profesorado de Enseñanza Secundaria Obligatoria y Bachillerato, Formación Profesional y Enseñanza de Idiomas y Física Avanzada

Modelos de Sistemas Físicos
Gabriel Lozano Barbero
Lugar: Universidad de Córdoba

Premios y Reconocimientos / Prizes and Acknowledgements

Hernán Míguez García. **Premio de la Real Sociedad Española de Fisica (RSEF) y la Fundación BBVA,** en la modalidad de Física, Innovación y Tecnología

Gabriel Lozano Barbero. **Premio de la Real Sociedad Española de Fisica (RSEF) y la Fundación BBVA,** en la categoría de Investigador Novel en Física Experimental

Miguel Anaya Martín. **Primer Accesit cicCartuja Ebro para jóvenes investigadores**

Estancias y Visitas de Personal del ICMS en otros Centros

Personnel of the ICMS in Other Laboratories

Australia Macquarie University		
Sidney, Australia	**Hérnan Míguez García**	2 meses
Instituto Tecnológico de Milán		
Milán, Italia	**Andrea Rubino**	2 meses
Laboratorio Kastler Brossel		
París, Francia	**Victoria Esteso Carrizo**	3 meses

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

University of Utah		
Utah, EE.UU.	**Prof. David Lynn Patrick**	3 meses
Collegue of William & Mary		
Virginia, EE.UU.	**Aaron Bayles**	9 meses

EQUIPAMIENTO CIENTÍFICO AVAILABLE EQUIPMENT

- Sistema de Magnetron Sputttering para la preparación de láminas delgadas
- Potenciostato y sistema electroquímico
- Sistema de medida de ángulos de contacto (advancing and receding)
- Equipo de crecimiento en vacío de nanofibras y otros materiales nanoestructurados
- Microscopio de efecto túnel (STM)
- Sistema de desorción térmica programada
- Espectrómetro de Fotoemisión (XPS)
- Analizador de potencial Z, tamaño de partícula y pesos moleculares (Malvern, ZS90)
- Bomba híbrida de vacío resistente a químicos VACUUBRAND RC-6
- Software y sondas para monitorización para equipo de liofilización CHRIST EPSILON 2-4 LSC
- Liofilizador de altas prestaciones Epsilon 2-4 (CHRIST)
- Estufa de desecación de 90 litros (RAYPA)
- Cámara Incubadora Opaq + Orbital Maxi (OVAN)
- pH & Ion-metro GLP 22+ (CRISON A)
- Malvern, Modelo Zetasizer Nano: para determinar mediante “Dynamic light scattering” la distribución de tamaños de partícula de sistemas coloidales en suspensión (disolvente acuoso u orgánico) en los rangos 1-3000 nanómetros.
  Este equipo también es adecuado para la evaluación del potencial “Z” de sistemas coloidales en suspensión (disolvente acuoso u orgánico) mediante medidas de Movilidad Electroforética.
- Colorímetro Dr. Lange: para la medida de parámetros de color (x, y, Y/L*a*b*, etc.) de superficies y polvos
- Ellipsómetro espectroscópico Woolan VB-400 con rango de frecuencias entre 300 y 1700 nm. Medida de índices de refracción y coeficiente de extención de capas delgadas y superficies.
- Espectrómetro visible-UV CARY-100. Medidas de coeficiente de absorción con luz normal y polarizada.

- Fluorímetro espectroscópico (HORYBA Jobin Yvon Fluorolog) con accesorio para la determinación de tiempos de vida. Microscopio de fluorescencia (HORYBA Jobin Yvon sigle photon controller: FluoroHub).
- Medidor de ángulos de contacto líquidos. Medidas ángulos de avance y retroceso, así como de energías de adhesión de líquidos sobre superficies (Dataphysics Contact Angle System SCA 20).
- Medida de cuatro puntos de conductividad eléctrica en superficies y láminas delgadas (Fuente de corriente Keithley 617 y voltímetro Keithley 2400).
- Medidas eléctricas en capas delgadas en función de la temperatura y la atmósfera
- Microscopio de Fuerzas atómicas (AFM) para la caracterización de superficies (Cervantes de Nanotec).
- Microscopio de efecto túnel (STM) con posibilidad de trabajar desde nitrógeno líquido hasta 600 ºC (VT-STM de Omicrom).
- Técnicas de caracterización de plasmas: sonda de Langmuir (Plasma Consult single and double sound), espectroscopía de emisión óptica (Avantes 200-900 nm resolución 1 nm) y espectrometría de masas (Hyden=
- Espectrómetro FT-IR con celda DRIFT (Pelkin elmer Spectrum One)
- Sistema de medida de porosidades en capas delgadas.
- Sistema de desorción térmica programada dotado con espectroscopía Auger (VG-8047).
- Espectrómetro de XPS (espectrómetro VSW) con sistema REELS de alta resolución (Kimball Physics EGPS-1022B) y fuente de átomos incorporada (Oxford Scientific Osprey plasma Source).

DISEÑO DE NANOMATERIALES Y MICROESTRUCTURAS

TAILORED NANOMATERIALS AND MICROSTRUCTURE

Personal / Personnel

Profesores de Investigación

Dra. Asunción Fernández Camacho

Catedráticos

Dr. Luis M. Esquivias Fedriani
Dr. Diego Gómez García

Investigadores Científicos

Dr. Juan Carlos Sánchez López

Científicos Titulares

Dra. Rosalía Poyato Galán
Dra. T. Cristina Rojas Ruiz

Investigadores Contratados

Dra. Gisela M. Arzac Di Tomaso
Dr. Victor Manuel Morales Florez
Dra. Vanda C. Fortio Godinho

Becarios Predoctorales

Lda. Mariana Paladini San Martin

Personal Contratado

Ing. Tec. M. Rocío García Gil
Lda. Jennifer López Viejobueno
Ldo. Dirk Hufschmidt
D. Miguel Nieto Redondo

Proyectos de Investigación / Research Projects

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

Cerámicas nanoestructuradas a base de carburo de boro y nitruro de titanio para aplicaciones estructurales Boron carbide and titanium nitride-based nanostructured ceramics for structural applications

Código/Code:	MAT2015-71411-R
Periodo/Period:	01-01-2016 / 31-12-2019
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	157.300 €
Investigador responsable/Research head:	Diego Gómez García / Arturo Domínguez Rodríguez
Componentes/Research group:	Francisco L. Cumbreras Hernández, Felipe Gutíerrez Mora, Ana Morales Rodríguez

RESUMEN / ABSTRACT

El proyecto tiene como misión la fabricación de forma controlada científicamente de nanocerámicos de carburo de boro y de nitruro de titanio mediante la técnica de chispa de plasma. Se estudiarán las propiedades mecánicas de ambos cerámicos a temperatura ambiente (dureza y tenacidad), así como su plasticidad a alta temperatura (resistencia a la fluencia, deformación a velocidad constante).

Se pretende estudiar la influencia de la microestructura en la respuesta mecánica, así como dilucidar los mecanismos que controlan la plasticidad (particularmente la interacción de dislocaciones con maclas). Los resultados se modelarán analíticamente o mediante simulación a escala mesoscópica (vía modelos de campos de fases).

Boron carbide and titanium nitride are among the most promising ceramic materials nowadays. In the first case, this is due to the outstandng mechanical properties (it is the third hardest material in nature) and its high resistance to chemical attack. In the case of Titanium nitride, its remarkable optical properties and electrical conductivity makes this a potential material for electronic devices. In both cases, sintering is a challenging issue due to the low diffusitivity. In this project, sintering of these materials by spark plasma sintering will be studied and the conditions for nanostructuration will be determined. Preliminary results show that average grain sizes as low as 100 nm can be achieved. In a second stage, plasticity will be studied. A previous model developed by the authors show that twinning is a key ingredient as a driving force of plasticity of boron carbide. The case of titanium nitride is mostly exciting because the stacking fault energy is the lowest ever known and it can make twinnin very favoured. The comparison between these two systems can be a clue about the basic mechanism for hardening in these ceramic materials.

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Desarrollo de catalizadores soportados sobre estructuras porosas para aplicaciones de generación y combustión catalítica de hidrógeno en el contexto de energías renovables Development of supported catalysts on porous structrures for hydrogen generation and catalytic combustión applications in the framework of renewable energies

Código/Code:	CTQ2015-65918-R
Periodo/Period:	01-01-2016 / 31-12-2018
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	177.870 €
Investigador responsable/Research head:	Asunción Fernández Camacho
Componentes/Research group:	Mª Carmen Jiménez de Haro, Vanda Godinho, Gisela Arzac, Dirk Hufschmidt, Rocio García

RESUMEN / ABSTRACT

El agotamiento de combustibles fósiles a corto y medio plazo y los cambios climáticos producidos por el efecto invernadero son algunas de las principales consecuencias del uso extendido de estos combustibles. En este escenario el hidrógeno como vector de transporte y almacenamiento de energía es un candidato muy atractivo en el contexto de un mayor uso de las energías renovables y limpias. En consecuencia se plantean actualmente retos importantes para el desarrollo de tecnologías adecuadas, tanto en la producción de hidrógeno libre de CO2, como en su transporte y almacenamiento seguro, y en su combustión eficiente para producir calor ó electricidad en una pila de combustible. Sobre la base de los proyectos previos del grupo en el estudio de hidruros complejos para almacenamiento de hidrógeno y en el desarrollo de catalizadores y procesos integrados de generación y uso del hidrógeno en aplicaciones portátiles; se abordarán en este proyecto nuevas investigaciones para desarrollar catalizadores novedosos soportados sobre estructuras porosas: membranas y espumas de materiales poliméricos, metálicos y cerámicos de alto interés actual. Los catalizadores se desarrollarán y estudiarán en reacciones seleccionadas de generación y combustión de acuerdo a las siguientes líneas de actuación:

1) Desarrollo de materiales novedosos con alto valor añadido del conjunto soporte-catalizador. Por un lado los soportes porosos basados en membranas de PTFE, espumas metálicas de Ni y espumas cerámicas de SiC. El objetivo es desarrollar los nuevos catalizadores sobre soportes de interés como membranas separadoras, electrolitos, electrodos ó combustores de hidrógeno. Los nuevos catalizadores persiguen la reducción del uso de metales nobles (i.e. bimetálicos Pt-Cu, Ni-Fe) y el desarrollo de nuevos materiales metal-metaloide (carburos, boruros, etc.). Se usarán métodos químicos de impregnación, y muy especialmente la tecnología de deposición de películas delgadas, pulverizacón catódica, que hemos aplicado recientemente con éxito a la fabricación de catalizadores de Co. La metodología abre un campo de investigación de

gran interés al permitirnos el control de la microestructura y/o la composición (i.e. Co, Co-B, Co-C) de los catalizadores a demanda.

2) La caracterización microestructural y química de los nuevos materiales y catalizadores desarrollados en el proyecto. Se trata típicamente de materiales con una microestructura y nanoestructura controlada en donde las modernas técnicas nanoscopicas van a jugar un papel fundamental en la fabricación a medida de estos.

3) Estudio de actividad en tres ensayos catalíticos: i) la generación hidrolítica de hidrógeno, ii) la descomposición fotocatalítica del agua y iii) la combustión catalítica del hidrógeno. Todas ellas reacciones de alto interés en el contexto del uso del hidrógeno como vector de transporte y almacenamiento de energías renovables.

--Sobre la base de los resultados obtenidos en estas líneas de actuación, el proyecto se ha diseñado para alcanzar un conocimiento fundamental y un diseño racional en la nanoescala de catalizadores soportados en sustratos porosos. Las relaciones composición-estructura-propiedades se investigarán usando los ensayos catalíticos y fotocatalíticos acoplados a la microscopía electrónica de alta resolución analítica y otras técnicas espectroscópicas.

The depletion of fossil fuels (in a short and long term) and the global warming derived from greenhouse effect are consequences of the extensive use of these fuels. In this context, hydrogen appears as an attractive, clean and abundant energy carrier in the context of a wider use of clean and removable energies. For the implementation of the “hydrogen economy” many technological challenges regarding hydrogen production (free from CO2), transport, storage (in a safe manner) and combustion (to produce heat or electricity) should be met first. New research will be conducted in this project on the basis of our previous results regarding the study of complex hydrides for hydrogen storage and the development of catalysts and processes for hydrogen generation and use in portable applications. In particular, new catalysts will be developed on porous structures such as polymeric, metallic and ceramic membranes and/or foams with high actual interest. Catalysts will we developed and studied for hydrogen generation and combustion reactions according to the following research lines:

1) Development of new materials (catalysts and supports) with a high added value of the complete system catalyst + support. Porous Ni and SiC foams together with PTFE membranes will be selected as supports for the studies. The main objective is to design new catalysts on technologically interesting supports such as separating membranes, electrolytes, electrodes and/or hydrogen combustors. These new catalysts will be developed following the objective of reducing the amount of noble metals by combining or replacing with another non-noble metals (e.g. Pt-Cu and Ni-Fe) and/or with metalloids (e.g carbides, borides, etc). Wet impregnation methods will be used and special emphasis will be put on the use of the PVD methodology (magnetron sputtering) recently employed in our laboratory for the fabrication of Co thin films with very good results. The latter methodology opens a highly interesting research field because permits to tune microstructure and composition (i.e. Co, Co-B, Co-C) on demand.

2) Characterization of the prepared materials from a microstructural and chemical point of view. Modern nanoscopies will play a key role in the characterization, comprehension and further improvement of these highly nanostructured catalysts.

3) Catalytic studies on the prepared materials will be carried out in three catalytic tests: i) the hydrogen generation through hydrolysis reactions, ii) the photocatalytic water splitting, and iii) the catalytic hydrogen combustion. These reactions are of high interest in the context of the hydrogen economy.

--The interaction of these three research lines as proposed in this project will permit to achieve basic knowledge on the rational design of nanocatalysts supported on porous materials. Structure-composition-activity relationships will be established through catalytic and photo-catalytic studies in combination with characterization techniques based on high resolution analytical TEM and additional spectroscopic techniques.

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Procesado y caracterización microestructural, mecánica y eléctrica de compuestos cerámica-grafeno Processing and microstructural, mechanical and electrical characterization of ceramic-graphene composites

Código/Code:	MAT2015-67889-P
Periodo/Period:	01-01-2016 / 31-12-2018
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	89.177 €
Investigador responsable/Research head:	Angela Gallardo López (UEI). Rosalía Poyato Galán
Componentes/Research group:	Antonio Muñoz Bernabé, Felipe Gutiérrez Mora, Ana Morales Rodríguez

RESUMEN / ABSTRACT

En la actualidad se plantean interesantes expectativas sobre los compuestos cerámica/grafeno, propuestos para aplicaciones en catálisis, almacenamiento y conversión de energía, protección del medio ambiente y biotecnología. Pero aún se requieren importantes esfuerzos para dar respuesta a cuestiones abiertas. Hay que incidir en aspectos como la resistencia a la cizalla de las intercaras cerámica-grafeno -esencial para asegurar la transferencia de carga efectiva sobre las láminas de grafeno-, la distribución homogénea del grafeno en la matriz cerámica y la relación de tamaños entre matriz y refuerzo, para maximizar el aumento de tenacidad y de conductividad eléctrica, así como también explorar sus propiedades mecánicas a alta temperatura.

En este proyecto se plantea un estudio sistemático de compuestos de matrices cerámicas con grafeno, desde la fabricación hasta la caracterización microestructural, mecánica y eléctrica, con el objetivo de mejorar la comprensión de los mecanismos que controlan estas propiedades al incorporar nanoestructuras de grafeno a una matriz cerámica. Se procesarán compuestos de dos matrices cerámicas diferentes, de alúmina y de circona tetragonal dopada con óxido

de itrio (3YTZP), con grafeno mediante técnicas coloidales, prestando especial atención a la dispersión del grafeno en la matriz cerámica, aspecto no exento de dificultades y que es clave para conseguir la mejora de las propiedades. La sinterización se realizará en un horno de descarga de plasma (SPS, spark plasma sintering) de última generación, optimizando las condiciones para conseguir compuestos densos y de tamaño de grano nanométrico. Para el análisis microestructural se utilizarán técnicas como la difracción de rayos X, la espectroscopía Raman, y la microscopía electrónica de barrido y transmisión. Con ellas se evaluarán las fases cristalográficas presentes, el tamaño de grano, la distribución de las nanoestructuras de grafeno, etc.

Desde el punto de vista del diseño de materiales avanzados, es fundamental investigar la relación entre microestructura y propiedades mecánicas y eléctricas. Las propiedades mecánicas a temperatura ambiente (dureza, tenacidad a la fractura y resistencia a flexión) se abordarán mediante indentación y ensayos de flexión, a escalas macro y microscópica. A alta temperatura, se estudiará la deformación plástica de los compuestos cerámica-grafeno mediante ensayos de fluencia en atmósferas controladas. También se estudiará el comportamiento tribológico de los compuestos y se evaluará su conductividad eléctrica, una de las propiedades más interesantes ya que se modifica de forma notoria como resultado de la incorporación del grafeno a estos sistemas cerámicos. La respuesta eléctrica se analizará en un amplio rango de temperaturas, bien mediante espectroscopía de impedancia compleja, bien mediante medidas de conductividad en corriente continua en el caso de los compuestos menos resistivos.

Nowadays, interesting prospects are proposed for ceramic-graphene composites, in application fields such as catalysis, energy storage and conversion, environment protection and biotechnology. A great effort is still required to answer open questions. Issues such as shear resistance of the ceramic-graphene interface essential to obtain an effective load transfer to the graphene sheets-, distribution of graphene in the ceramic matrix -to maximize the reinforcement mechanisms and electrical conductivity- and the high temperature mechanical properties in these composites need special attention.

A systematic study of ceramic matrix graphene composites, including processing and microstructural, mechanical and electrical characterization is proposed in this project, with the aim of improving the comprehension of mechanisms controlling these properties when adding graphene nanostructures to a ceramic matrix.

Both alumina and yttria tetragonal zirconia (3YTZP) graphene composites will be processed by means of colloidal techniques. Special attention will be devoted to the dispersion of graphene in the ceramic matrix which is not a straightforward aspect, but is key to improve mechanical and functional properties. Sintering will be carried out by spark plasma sintering, SPS. Conditions will be optimized in order to obtain fully dense composites with nanometric grain size. Microstructural analysis will be performed by X ray diffraction, Raman spectroscopy, scanning and transmission electronmicroscopy (SEM and TEM). The present crystallographic phases, grain size and distribution of graphene nanostructures will be evaluated.

In order to design advanced materials, it is necessary to study the relationship between microstructure and mechanical or electrical properties. Room temperature mechanical properties (hardness, fracture toughness and flexural resistance) will be characterized by indentation and bending tests at macro and microscopic scales. At high temperature, the plastic behavior of these ceramic-graphene composites will be assessed by creep tests under controlled atmosphere. Tribological behavior of the composites will also be studied to evaluate their resistance to wear. The electrical response will be assessed in a wide range of temperatures by means of complex impedance spectroscopy or by direct current conductivity measurements in the composites with lower resistivity. This is a most interesting property since it can be strongly increased when incorporating graphene to these ceramic systems.

Recubrimientos para aplicaciones en energía y alta temperatura High temperatura energy application coatings

Código/Code: MAT2015-65539-P
Periodo/Period: 01-01-2016 / 31-12-2018
Organismo Financiador/Financial source: Ministerio de Economía y Competitividad
Importe total/Total amount: 83.006 €
Investigador responsable/Research head: Juan Carlos Sánchez López
Componentes/Research group: Teresa Cristina Rojas Ruiz, Maria Belinda Sigüenza Carballo, Iñigo Braceras Izaguirre, Marta Brizuela Parra

RESUMEN / ABSTRACT

La protección de las superficies frente a la temperatura, los fenómenos de oxidación o el desgaste ha logrado un progreso substancial mediante el desarrollo de nuevos materiales y recubrimientos con propiedades mejoradas tales como dureza extrema, baja fricción y tasas de desgaste, elevada resistencia ante la temperatura y la oxidación. Estas mejoras suponen un enorme ahorro de energía y reducción de costes debido a la vida media de los componentes mecánicos sin necesidad de sustitución, así como, a una reducción del impacto medioambiental. Este campo de investigación tiene una profunda repercusión en una gran variedad de sectores industriales (energía, herramientas de mecanizado, automoción, aeronáutico, metalurgia, etc.). El reto para la mayoría de estos procesos de funcionalización superficial residen en un control estricto de la micro y nanoestructura de la superficie y de las intercaras que hagan posible la aparición de nuevas propiedades y aplicaciones que la nanotecnología ofrece.

En este proyecto, se prepararán recubrimientos nanoestructurados para la protección de componentes sometidos a altas temperaturas y ambientes agresivos buscando un comportamiento mejorado. Este objetivo será abordado para tres diferentes aplicaciones que contribuirían a procesos energéticos más eficientes, energías renovables y soluciones para disminuir el impacto medioambiental. Basándonos en el sistema Cr-Al-N, se depositarán diferentes recubrimientos mediante la técnica de pulverización catódica reactiva cambiando la composición química (contenido en metal, incorporación de dopantes tales como Y o Si); microestructura; distribución de fases; arquitectura (multicapa/nanocomposite) o estructuras más complejas (tándem, multicapa en gradiente) sobre los sustratos apropiados dependiendo de la aplicación prevista: a) resistencia a la oxidación a alta temperatura (hasta 1000ºC) para herramientas; b) absorbedores solares selectivos estables térmicamente a medias (300-500ºC) y alta temperatura (>600ºC); resistencia a la corrosión para componentes en turbinas de vapor supercríticos (650ºC/100% vapor).

La investigación sobre los mecanismos de oxidación, transformaciones de fases, modificaciones estructurales, etc. serán objeto de un estudio detallado sobre los sustratos definidos para lograr un conocimiento fundamental sobre los procesos de degradación y los efectos protectores. El establecimiento de correlaciones entre las propiedades iniciales y el comportamiento funcional permitirá una mejor comprensión de los mecanismos de protección y por ende, una optimización de tales sistemas en forma de recubrimientos nanoestructurados para las aplicaciones previstas.

The protection of surfaces from thermal, wear and oxidation phenomena has reached a substantial progress by developing new materials and coatings with improved properties as extreme hardness, low friction and wear rates, increased thermal and oxidation resistance. These improvements suppose a huge energy-saving and cost reduction due to the increased life-time of mechanical components without needs of replacement as well as a reduction in the environmental impact. This field of research has a deep impact in a large variety of industrial sectors (energy, machining tools, automotive, aeronautic, metallurgy, etc.). The challenge for most of these surface functionalization procedures is to get a strict control of the micro and nanostructure of the surface and interfaces that make possible the advent of new properties and applications that nanotechnology concept offers.

In this project, tailored nanostructured coatings for protection of components submitted to high temperature and aggressive environments are prepared seeking for an improved performance. This goal will be explored for three different applications that would contribute to an energy efficiency, renewable energies and solutions to decrease environmental impact. Based on the Cr-Al-N system, different coatings will be prepared by reactive magnetron sputtering technology changing chemical composition (metal content, incorporation of dopants like Y or Si); microstructure; phase distribution; architecture (multilayer/ nanocomposite) or more complex structures (tandem, multilayer gradient) on appropriated substrates depending on the foreseen application: a) oxidation resistance at high temperature (up to 1000ºC) for tool components; b) thermal stable solar selective absorber coating for mid (300-500ºC) and high temperature (>600ºC); c) corrosion resistant coating for supercritical turbine components (650ºC and 100% steam atmosphere).

The investigation of the oxidation mechanisms, phase transformations, structural modifications, etc. will be object of a careful study directly over the defined substrates for these applications to get fundamental knowledge on the degradation phenomena and protective effects. The establishment of the relationships between the initial properties and observed functional performance will enable the better understanding of the protection mechanisms and the optimization of such nanostructured coating systems for the selected application.

c) Studies of coupling a hydrogen generator system with a low cost fuel cell. Typically a continuous reactor for the hydrolysis of NaBH4 with Co-B catalyst for providing H2 at constant flow rate conditions to directly feed a PEM fuel cell of 60 W.

d) Fundamental studies for the development of catalysts and supports for the controlled combustion of hydrogen. It's a new line in the research group based on wet chemical preparation of noble metal nanoparticle catalysts on commercial porous ceramic supports (i.e. SiC). The line also includes the design of a reactor for laboratory-scale study of heat production by controlled combustion of hydrogen.

e) Development of sputtering technology ("magnetron sputtering") for the preparation of catalysts and nano-structures on various substrates for use in the processes developed in the previous sections. The group has extensive experience in this technology to be applied in novel ways in this project leading to a great versatility regarding nanostructure, composition and addition of additives to improve catalytic activity, durability and selectivity of catalysts.

f) Microstructural and chemical characterization of new materials and catalysts developed in the project. We are dealing typically with materials of controlled nanostructure where modern nanoscopic techniques will play a key role in the custom manufacturing of these materials.

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Aplicación de técnicas avanzadas de microscopía electrónica para la caracterización de recubrimientos nano-estructurados para aplicaciones en energías limpias Application of advanced electron microscopy techniques to the characterization of nanostructured coatings for clean energy applications

Código/Code:	TAHUB-050. Programa Talent HUB
Periodo/Period:	01-03-2015 / 28-02-2017
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	153.025 €
Investigador responsable/Research head:	Ana María Beltrán Custodio

RESUMEN / ABSTRACT

Este proyecto se centra en la generación y almacenamiento de hidrógeno con el objetivo de producir hidrógeno para energías limpias. Esto sucede durante una reacción exotérmica en la que es necesaria la presencia de un catalizador para que se lleve a cabo en condiciones de seguridad. Los catalizados basados en metales nobles son buenos candidatos para este objetivo (cobalto, cobre…). Aquí, los sistemas completos catalizador-soporte son estudiados. Estos sistemas son crecidos mediante técnicas de pulverización catódica ("magnetron sputtering"). La estructura y la composición son estudiadas a escala nanométrica mediante técnicas avanzadas de microscopía electrónica de transmisión-barrido (STEM), como la microscopía electrónica de alta resolución (HRTEM), imágenes adquiridas en modo campo oscuro con detector de alto ángulo (HAADF), energía dispersiva de rayos X, espectroscopia de pérdida de energía de electrones (EELS), para análisis químico. Además, el uso de la técnica de caracterización tridimensional, tomografía electrónica, aporta un completo conocimiento del sistema analizado. La combinación de técnicas de análisis estructural y de composición, en modo TEM y STEM, nos permite obtener una completa nano-caracterización del sistema. Estos análisis STEM son una herramienta esencial para determinar la relación entre la microestructura, las condiciones de crecimiento y el comportamiento final y las propiedades del sistema, que nos ayudará a mejorarlos y, por tanto, contribuir a la producción de energía limpia.

Este proyecto tiene cuatro objetivos estratégicos.

1. Nano-materiales para aplicaciones en energía limpia. Materiales para la producción, uso y almacenamiento de hidrógeno.
2. Desarrollo de la técnica de magnetron sputtering para la fabricación de nano-estructuras (capas delgadas, recubrimientos y micro-estructuras multicapas).
3. Potenciación de las facilidades LANE (Laboratorio de microscopía del centro ICMSE-CSIC).
4. Uso de técnicas avanzadas de caracterización estructural y de análisis para el estudio a nano-escala de nuevos nano-materiales.

This project is focus on the hydrogen generation and storage with the aim of producing hydrogen for clean and sustainable energies. It happens due to an exothermic reaction where a catalyst is required to do so safety. Catalysts based on noble metals are good candidates for this purpose such as, cobalt, cupper… Here, the complete catalysts systems and different supports are studied. They have been grown by magnetron sputtering technology. The structure and composition are studied, up to nano-scale, by advanced scanning-transmission electron microscopy techniques, (S)TEM, such as high-resolution (HRTEM), high-angle annular dark field (HAADF), energy dispersive X-Ray (EDX), electron energy loss spectroscopy (EELS), for chemical analysis. Furthermore, the use of the three-dimensional characterization technique electron-tomography provides a full understanding of the analysed material. The combination of structural and compositional analytical microscope techniques, in both STEM and TEM mode, allows a full nano-characterization of the systems. The (S)TEM analyses are the essential tool to determine the relationship among the microstructure, the growth conditions and the final behaviour and properties of the systems which will help to improve them and, therefore, to contribute to the production of clean energy.

This project has four main strategic objectives:

1. Nano-materials for sustainable energy applications. Materials for the production, use and storage of Hydrogen.
2. Development of sputtering technology for the fabrication of nanostructures (thin films, coatings and controlled microstructure multilayers).
3. Development of the potential capabilities of the Laboratory for Nanoscopies and Spectroscopies (LANE).
4. Use of advanced structural and analytical techniques for the nano-analysis of new nanomaterials.

OTROS PROYECTOS / OTHER PROJECTS

Adquisición e instalación de un microscopio electrónico de transmisión

Código/Code:	CSIC15-CE-3298
Periodo/Period:	01-01-2016 / 31-12-2017
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	420.180 €
Investigador responsable/Research head:	Asunción Fernández Camacho

Convenios y Contratos / Contracts and Agreements

Research on tribological systems. Functional coatings and their characterization

Periodo/Period: 23-10-2014 / 31-12-2018
Organismo Financiador/Financial source: Robert Bosch Gmbh
Importe total/Total amount: 24.490 €
Investigador responsable/Research head: Juan Carlos Sánchez López
Componentes/Research group: Santiago Domínguez Meister

Caracterización microstructural y química de materiales por microscopía electrónica

Periodo/Period: 14-02-2014 / 13-02-2017
Organismo Financiador/Financial source: ABENGOA RESEARCH S.L.
Importe total/Total amount: 15.125 €
Investigador responsable/Research head: Asunción Fernández Camacho
Componentes/Research group: Vanda Fortio Godinho, M. Carmen Jiménez de Haro, Inmaculada Rosa Cejudo

Caracterización microstructural y química de materiales en células CIGSe

Periodo/Period: 13-05-2014 / 12-05-2017
Organismo Financiador/Financial source: ABENGOA Solar New Technologies, S.A.
Importe total/Total amount: 55.660 €
Investigador responsable/Research head: Asunción Fernández Camacho
Componentes/Research group: Vanda Fortio Godinho, M. Carmen Jiménez de Haro, Inmaculada Rosa Cejudo, Olga Montes Amorín

Caracterización microstructural y química de materiales para avisadores sonoros

Periodo/Period: 19-01-2010 / 30-06-2017
Organismo Financiador/Financial source: CLARTON HORN
Importe total/Total amount: 34.549 €
Investigador responsable/Research head: Asunción Fernández Camacho
Componentes/Research group: Vanda Fortio Godinho, M. Carmen Jiménez de Haro, Inmaculada Rosa Cejudo

Caracterización por TEM y ensayos tribológicos (desgaste y rayado) del Sistema Nb-C

Periodo/Period: 28-11-2017 / 28-12-2017
Organismo Financiador/Financial source: IQS-CETS Fundació Privada
Importe total/Total amount: 1.500 €
Investigador responsable/Research head: Juan Carlos Sánchez López
Componentes/Research group: Juan Carlos Sánchez López, T. Cristina Rojas

Desarrollo de capas PVD

Periodo/Period:	8-05-2017 / 7-05-2019
Organismo Financiador/Financial source:	Flubetech, S.L.
Importe total/Total amount:	7.200 €
Investigador responsable/Research head:	Juan Carlos Sánchez López
Componentes/Research group:	Juan Carlos Sánchez López, T. Cristina Rojas

PATENTES / PATENTS

Blanco sólido de gases nobles para reacciones nucleares

Inventores: Jaime Caballero Hernández, Begoña Fernández Martínez, Asunción Fernández Camacho, Francsico Javier Ferrer Fernández, Vanda Cristina Fortio Godinho, Joaquín José Gómez Camacho
Tipo de Patente: Nacional
Solicitud número: 201630711
Fecha Solicitud: 31 mayo 2016
Entidad Titular: Consejo Superior de Investigaciones Científicas y Universidad de Sevilla

COOPERACIÓN INTERNACIONAL Y OTROS INTERNATIONAL COOPERATION AND OTHERS

Nano-Phenomena and Functionality of Modern Carbon-Based Tribo-Coatings

Periodo/Period:	1-06-2015 / 30-06-2018
Código/Code:	CARBITRIB
Entidad Financiadora/Financial source:	Leverhulme Trust (UK)
Investigador responsable/Research head:	Feodor Borodich
Participantes/Participants:	Juan Carlos Sánchez López

Universidad Nacional de Ciencia y Tecnología de Rusia (National University of Science and Technology «MISiS») y la Agencia Estatal Consejo Superior de Investigaciones Científicas (CSIC) a través del Instituto de Ciencia de Materiales de Sevilla (ICMS)

Periodo/ Period:	02-12-2016 / 01-02-2019
Código/Code:	C-ES-2016-101
Referencia VATC:	20171119
Investigador responsable/Research head:	Juan Carlos Sánchez López

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

Pt-impregnated catalysts on powdery SiC and other commercial supports for the combustion of hydrogen under oxidant conditions

Arzac, G. M.; Montes, O.; Fernandez, A.
Applied Catalysis B: Environmental, **201** (2017) 391-399
Enero, 2017 | **DOI:** 10.1016/j.apcatb.2016.08.042

We report the study of the catalytic hydrogen combustion over Pt-impregnated powdery silicon carbide (SiC) using H2PtCl6 as precursor. The reaction was conducted in excess of oxygen. beta-SiC was selected for the study because of its thermal conductivity, mechanical properties, chemical inertness and surface area. The obtained Pt particles over SiC were medium size (average particle diameter of 5 nm for 0.5 wt% Pt). The activity of the Pt-impregnated catalyst over SiC was compared to those obtained in oxidized form over TiO2 and Al2O3 commercial supports (Pt particles very small in size, average particle diameter of 1 nm for 0.5 wt% Pt in both cases). The case of a SiO2 support was also discussed. Those Pt/SiC particles were the most active because of their higher contribution of surface Pt, indicating that partially oxidized surfaces have better activity than those totally oxidized in these conditions. SiC was modified with an acid treatment and thus bigger (average particle diameter of 7 nm for 0.5 wt% Pt) and more active Pt particles were obtained. Durability of the SiC and TiO2 supported catalysts was tested upon 5 cycles and both have shown to be durable and even more active than initially. Exposure to the oxidative reaction mixture activates the catalysts and the effect is more pronounced for the completely oxidized particles. This is due to the surface oxygen chemisorption which activates catalystsi surface.

The role of cobalt hydroxide in deactivation of thin film Co-based catalysts for sodium borohydride hydrolysis

Paladini, M; Arzac, GM; Godinho, V; Hufschmidt, D; de Haro, MCJ; Beltran, AM; Fernandez, A
Applied Catalysis B: Environmental, **210** (2017) 342-352
Agosto, 2017 | **DOI:** 10.1016/j.apcatb.2017.04.005

Deactivation of a Co catalyst prepared as thin film by magnetron sputtering was studied for the sodium borohydride (SB) hydrolysis reaction under different conditions. Under high SB concentration in single run experiments, the formation of a B-O passivating layer was observed after 1.5 and 24 h use. This layer was not responsible for the catalyst deactivation. Instead, a peeling-off mechanism produced the loss of cobalt. This peeling-off mechanism was further studied in cycling experiments (14 cycles) under low SB concentrations. Ex-situ study of catalyst surface after use and solid reaction products (precipitates) was performed by X-Ray photoelectron spectroscopy (XPS), transmission electron microscopy (TEM) and scanning transmission electron microscopy (STEM). The presence of cobalt hydroxide and oxyhydroxide was detected as major components on the catalyst surface after use and as precipitates in the supernatant solutions after washing. Cobalt borate, cobalt carbonate and oxycarbonate were also formed but in lesser amounts. These oxidized cobalt species were formed and further detached from the catalyst at the end of the reaction and/or during catalyst washing by decomposition of the unstable in-situ formed cobalt boride. Leaching of cobalt soluble species was negligible. Thin film mechanical detachment was also found but in a smaller extent. To study the influence of catalyst composition on deactivation processes, cycling experiments were performed with Co-B and Co-C catalysts, also prepared as thin films. We found that the deactivation mechanism proposed by us for the pure Co catalyst also occurred for a different pure Co (prepared at higher pressure) and the Co-B and Co-C samples in our experimental conditions.

Towards Extending Solar Cell Lifetimes: Addition of a Fluorous Cation to Triple Cation-Based Perovskite Films

Salado, M; Fernandez, MA; Holgado, JP; Kazim, S; Nazeeruddin, MK; Dyson, PJ; Ahmad, S
Chemsuschem, **10** (2017) 3846-3853
Octubre, 2017 | **DOI:** 10.1002/cssc.201700797

Organohalide perovskites have emerged as highly promising replacements for thin-film solar cells. However, their poor stability under ambient conditions remains problematic, hindering commercial exploitation. The addition of a fluorous-functionalized imidazolium cation during the preparation of a highly stable cesium-based mixed perovskite material Cs-0.05(MA(0.15)FA(0.85))(0.95)Pb(I0.85Br0.15)(3) (MA= methylammonium; FA= formamidinium) has been shown to influence its stability. The resulting materials, which vary according to the amount of the fluorous-functionalized imidazolium cation present during fabrication, display a prolonged tolerance to atmospheric humidity (> 100 days) along with power conversion efficiencies exceeding 16%. This work provides a general route that can be implemented in a variety of perovskites and highlights a promising way to increase perovskite solar cell stability.

Solid lubricant behavior of MoS2 and WSe2-based nanocomposite coatings

Dominguez-Meister, S; Rojas, TC; Brizuela, M; Sanchez-Lopez, JC
Science and Tecnology of Advances Materials, **18** (2017) 1
Marzo, 2017 | **DOI:** 10.1080/14686996.2016.1275784

Tribological coatings made of MoS2 and WSe2 phases and their corresponding combinations with tungsten carbide (WC) were prepared by non-reactive magnetron sputtering of individual targets of similar composition. A comparative tribological analysis of these multiphase coatings was done in both ambient air (30-40% relative humidity, RH) and dry nitrogen (RH<7%) environments using the same tribometer and testing conditions. A nanostructural study using advanced transmission electron microscopy of the initial coatings and examination of the counterfaces after the friction test using different analytical tools helped to elucidate what governs the tribological behavior for each type of environment. This allowed conclusions to be made about the influence of the coating microstructure and composition on the tribological response. The best performance obtained with a WSe x film (specific wear rate of 2 x 10(-8) mm(3) N(-1)m(-1) and a friction coefficient of 0.03-0.05) was compared with that of the well-established MoS2 lubricant material.

Determination of the thickness of the embedding phase in 0D nanocomposites

Martinez-Martinez, D; Sanchez-Lopez, JC
Applied Surface Science, **421** (2017) 179-184
Noviembre, 2017 | **DOI:** 10.1016/j.apsusc.2016.12.081

0D nanocomposites formed by small nanoparticles embedded in a second phase are very interesting systems which may show properties that are beyond those observed in the original constituents alone. One of the main parameters to understand the behavior of such nanocomposites is the determination of the separation between two adjacent nanoparticles, in other words, the thickness of the embedding phase. However, its experimental measurement is extremely complicated. Therefore, its evaluation is performed by an indirect approach using geometrical models. The ones typically used represent the nanoparticles by cubes or spheres.
In this paper the used geometrical models are revised, and additional geometrical models based in other parallelohedra (hexagonal prism, rhombic and elongated dodecahedron and truncated octahedron) are presented. Additionally, a hybrid model that shows a transition between the spherical and tessellated models is proposed. Finally, the different approaches are tested on a set of titanium carbide/amorphous carbon (TiC/a-C) nanocomposite films to estimate the thickness of the a-C phase and explain the observed hardness properties.

Ceramics of Ta-doping stabilized orthorhombic ZrO2 densified by spark plasma sintering and the effect of post-annealing in air

Sponchia, G; Moshtaghioun, BM; Benedetti, A; Riello, P; Gomez-Garcia, D; Dominguez-Rodriguez, A; Ortiz, AL
Scripta Materialia, **130** (2017) 128-132
Marzo, 2017 | **DOI:** 10.1016/j.scriptamat.2016.11.021

16 mol% Ta-doped ZrO2 powders were synthesized and densified by spark-plasma sintering (SPS) in vacuum, followed by post-SPS annealing in air, thus obtaining two ultrafine-grained ceramics consisting of Ta-doping stabilized orthorhombic ZrO2. The as-SPSed ceramic is black because it is actually a suboxide essentially with reduced cations and abundant oxygen vacancies, whereas the post-annealed ceramic is white because it is an oxide without vacancies and with only partially reduced cations. Both ceramics are relatively hard and brittle, but the as-SPSed ceramic was slightly more so, attributable to crystallographic and microstructural differences. Implications of interest for the ceramics community are discussed.

Carbon nanofibers replacing graphene oxide in ceramic composites as a reinforcing-phase: Is it feasible?

Cano-Crespo, Rafael; Malmal Moshtaghioun, Bibi; Gomez-Garcia, Diego; Dominguez-Rodriguez, Arturo; Moreno, Rodrigo
Journal of the European Ceramic Society, **37** (2017) 3791-3796
Septiembre, 2017 | **DOI:** 10.1016/j.jeurceramsoc.2017.03.027

In recent years, the interest of graphene and graphene-oxide has increased extraordinarily due to the outstanding properties concurring in this material. In ceramic science, the possibility of combining excellent electrical conductivities together with an enhancement of mechanical properties has motivated the research in fabrication of graphene oxide-reinforced ceramic composites despite the intrinsic difficulties for sintering. In this work a comparison is made between graphene oxide-reinforced alumina composites and carbon nanofiber-reinforced alumina ones. It will be concluded that the improvement of mechanical properties is scarce, if any. Since carbon nanofibers have also a good electrical conductivity their importance for future applications as a replacement of more sophisticated but expensive graphene-based ceramic composites will be stressed.

Dense graphene nanoplatelet/yttria tetragonal zirconia composites: Processing, hardness and electrical conductivity

Gallardo-Lopez, A; Marquez-Abril, I; Morales-Rodriguez, A; Munoz, A; Poyato, R
Ceramics International, **43** (2017) 11743-11752
Octubre, 2017 | **DOI:** 10.1016/j.ceramint.2017.06.007

Yttria tetragonal zirconia ceramic composites with 1, 2.5, 5 and 10 vol% nominal contents of graphene nanoplatelets (GNPs) were fabricated and characterized. First, the GNP dispersion in isopropanol was optimized to de-agglomerate the GNPs without damaging their structure. Then, submicrometric fully dense composites were obtained via spark plasma sintering (SPS) at 1250 degrees C with a 5 min holding time. The processing routine produced a nearly homogeneous GNP dispersion in the ceramic matrix, and the GNPs preferential orientation was perpendicular to the sintering compression axis. A ceramic grain refinement due to the GNPs was also detected. The Vickers hardness measured on the plane perpendicular to the sintering compression axis (basal plane) was lower than on the cross sections. This anisotropy increased with the increasing GNP content, while the average hardness decreased. The electrical conductivity was also highly anisotropic, up to seven times higher for the basal planes. The electrical percolation threshold for these composites was estimated to be between 2.2 and 4.4 vol% of the GNP measured content.

High-temperature creep of carbon nanofiber-reinforced and graphene oxide-reinforced alumina composites sintered by spark plasma sintering

Cano-Crespo, Rafael; Malmal Moshtaghioun, Bibi; Gomez-Garcia, Diego; Dominguez-Rodriguez, Arturo; Moreno, Rodrigo
Ceramics International, **43** (2017) 7136-7141
Junio, 2017 | **DOI:** 10.1016/j.ceramint.2017.02.146

Alumina (Al2O3) ceramic composites reinforced with either graphene oxide (GO) or carbon nanofibers (CNFs) were prepared using Spark Plasma Sintering. The effects of GO and CNFs on the microstructure and in consequence on their mechanical properties were investigated. The microstructure of the sintered materials have been characterized quantitatively prior to and after the creep experiments in order to discover the deformation mechanism. Graphene-oxide reinforced alumina composites were found to be more creep resistant than carbon nanofibers-reinforced alumina ones or monolithic alumina with the same grain size distribution. In all the cases, grain boundary sliding was identified as the deformation mechanism.

Spark plasma sintering of fine-grained alumina ceramics reinforced with alumina whiskers

Tamura, Y; Moshtaghioun, BM; Gomez-Garcia, D; Rodriguez, AD

Ceramics International, **43** (2017) 658-663

Enero, 2017 | **DOI:** 10.1016/j.ceramint.2016.09.210

Densification of alumina whisker-reinforced alumina ceramics by spark plasma sintering (SPS) has been investigated with the aim of obtaining a fine-grained microstructure and also studying the effect of whisker addition on the room-temperature mechanical properties. It was found that whisker addition retards slightly the sinterability of alumina by whisker hindering of particle rearrangement. Besides, the internal stress on the alumina matrix particles reduced due to the presence of a whisker network structure of strong rigid boundaries. Nevertheless near fully-dense and fine-grained alumina ceramics with alumina whisker content between 3 wt% and 10 wt% could be obtained under appropriate SPS conditions. The hardness of alumina ceramics with 3 wt% was comparable to that of pure alumina ceramics (similar to 26 GPa) whereas its fracture toughness (5.6 MPa m(1/2)) was higher (4.2 MPa m(1/2)). Crack bridging by well-dispersed whiskers and whiskers pull-out were identified as the main toughening mechanisms.

Effect of acid-treatment and colloidal-processing conditions on the room temperature mechanical and electrical properties of 3YTZP/MWNT ceramic nanocomposites

Poyato, R.; Morales-Rodríguez, A.; Gutiérrez-Mora, F.; Muñoz, A.; Gallardo-López, A.

Ceramics International, **43** (2017) 16560-16568

Diciembre, 2017 | **DOI:** 10.1016/j.ceramint.2017.09.043

Different colloidal powder processing routines have been used to prepare composites of 3 mol% Y2O3 -ZrO2 (tetragonal zirconia polycrystals, 3YTZP) with 2.5 vol% multiwall carbon nanotubes (MWNT) with the aim of achieving a homogeneous distribution of the MWNTs in the ceramic, eliminating agglomerates but also minimizing carbon nanotube (CNT) damage during processing. Modifications of the acid treatment applied to the nanotubes, including subjecting them to stirring or ultrasonic agitation, and use of acid or basic pH during composite powder mixing have been approached.

No MWNT damage during processing was detected by Raman spectroscopy. CNT bundles were found in all the composites forming different patterns depending on the processing route. Similar values of hardness were obtained for all the composites, while different anisotropy in fracture propagation was found when studying parallel and perpendicular directions to the sintering pressing axis on the cross sections of the composites due to the MWNT preferential alignment. The CNT bundles were found to act as fracture short paths. A similar anisotropic behavior was observed for the electrical conductivity. These results have been correlated to the different microstructures obtained in the composites prepared with different processing routines.

Incorporation of Calcium Containing Mesoporous (MCM-41-Type) Particles in Electrospun PCL Fibers by Using Benign Solvents

Liverani, L.;Boccardi, E.; Beltrán, A.M.; Boccaccini, A.R.
Polymers, **9** (2017) 487
Octubre, 2017 | **DOI:** 10.3390/polym9100487

The electrospinning technique is a versatile method for the production of fibrous scaffolds able to resemble the morphology of the native extra cellular matrix. In the present paper, electrospinning is used to fabricate novel SiO2particles (type MCM-41) containing poly(epsilon-caprolactone) (PCL) fibers. The main aims of the present work are both the optimization of the particle synthesis and the fabrication of composite fibers, obtained using benign solvents, suitable as drug delivery systems and scaffolds for soft tissue engineering applications. The optimized synthesis and characterization of calcium-containing MCM-41 particles are reported. Homogeneous bead-free composite electrospun mats were obtained by using acetic acid and formic acid as solvents; neat PCL electrospun mats were used as control. Initially, an optimization of the electrospinning environmental parameters, like relative humidity, was performed. The obtained composite nanofibers were characterized from the morphological, chemical and mechanical points of view, the acellular bioactivity of the composite nanofibers was also investigated. Positive results were obtained in terms of mesoporous particle incorporation in the fibers and no significant differences in terms of average fiber diameter were detected between the neat and composite electrospun fibers. Even if the Ca-containing MCM-41 particles are bioactive, this property is not preserved in the composite fibers. In fact, during the bioactivity assessment, the particles were released confirming the potential application of the composite fibers as a drug delivery system. Preliminary in vitro tests with bone marrow stromal cells were performed to investigate cell adhesion on the fabricated composite mats, the positive obtained results confirmed the suitability of the composite fibers as scaffolds for soft tissue engineering.

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

EERA JP FC&H2 Workshop 2017 Prioritization Workshop

3 - 4 abril [Roskilde, Dinamarca]

Highlights contributions of CSIC to SP6 (and SP7). SP6: Hydrogen production and handling. SP7: Hydrogen storage
A. Fernández
Poster

2nd CARBTRIB Workshop on "Nano-Phenomena and Functionality of Modern Carbon-based Tribo-Coatings"

19 - 21 abril [Sevilla, España]

Surface texturing for improved tribological performance: the snake skin case
J.C. Sánchez López
Comunicación oral

12th Pacific Rim Conference on Ceramic and Glass Technology (PACRIM 12), including Glass & Optical Materials Division Meeting (GOMD 2017)

21 - 26 mayo [Hawai, Estados Unidos]

Innovative Engineering Applications for Reticulated Cellular Ceramics
U. F. Vogt; B. Fumey; G. Plesch; A. Fernandez; A. Bonk; P. Dimopoulos; A. Steinfeld
Conferencia Invitada

EMRS Spring Meeting

21 - 26 mayo [Estrasburgo, Francia]

High-temperature oxidation of multi-layered CrAlYN coatings: implications of the presence of Y and type of steel
T.C. Rojas;S. Domínguez-Meister;M. Brizuela;J.C. Sánchez-López
Comunicación oral

IX Iberian Conference on Tribology

12 - 13 junio [Guimaraes, Portugal]

Comparative tribological behaviour of MoS2 and WSe2-based nanocomposite coatings
J.C. Sánchez-López; S. Domínguez-Meister; T.C. Rojas; M. Brizuela
Comunicación oral

Microstructure, tribological and antibacterial properties of silver-containing carbon coatings
S. Domínguez-Meister; T.C. Rojas; J.C. Sánchez-López
Poster

15th Conference & Exhibition of the European Ceramic Society

9 – 13 julio [Budapest, Hungria]

Effect of graphene content on the tribological behaviour of 3YTZP reinforced with graphene nanoplatelets
F. Gutiérrez-Mora; Á. Gallardo-López; A. Morales-Rodríguez; R. Poyato
Comunicación oral

Graphene nanoplatelet/3 mol% yttria doped zirconia composites with high electrical conductivity
R. Poyato; J. Osuna; A. Morales-Rodríguez; Á. Gallardo-López
Comunicación oral

Microstructure and properties of presureless sintered graphene nanoplatelet/3YTZP composites
C. López-Pernía; R. Poyato; A. Morales-Rodríguez; Á. Gallardo-López
Poster

Graphene-ceramic composites with 3YTZP matrix
Á. Gallardo-López; C. Muñoz-Ferreiro; A. Morales-Rodríguez; R. Poyato
Poster

Creep behaviour of alumina reinforced composites sintered by Spark Plasma Sintering
R. Cano-Crespo; B. Malmal-Moshtaghioun; D. Gómez-García; A. Domínguez-Rodríguez; R. Moreno
Poster

European Advanced Materials Congress 2017
22 – 24 agosto [Estocolmo, Suecia]

Enhancing processing, microstructure and properties of graphene/3YTZP ceramic composites
A. Gallardo-López; C. Muñoz-Ferreiro; A. Morales-Rodríguez; F. Gutiérrez-Mora; R. Poyato
Comunicación oral

Influence of graphene nanoplatelets orientation in a ceramic matrix on its scratch behaviour
F. Gutiérrez-Mora; A. Gallardo-López; C. Muñoz-Ferreiro; A. Morales-Rodríguez; A. Muñoz; J.C. Sánchez-López; R. Poyato
Poster

4th International Conference on Titanium Powder Metallurgy & Additive Manufacturing | PMTi 2017
8 – 10 septiembre [Xian Shi, China]

Effects of the thermal treatment of Titanium samples fabricated by selective laser melting
J.E. González; A.M. Beltrán; I. Montelaegre; J. Negrín; E. Peón; F.J. Gotor; P. Trueba; J.A. Rodríguez-Ortiz; A. Fernández; Y. Torres
Poster

10th International Conference on Porous Metals and Metallic Foams
14 – 17 septiembre [Nanjing, China]

Thermo-chemical treatment of porous Ti6Al4V obtained by selective laser melting
J. E. González; J. Negrín; A. M. Beltrán; I. Montelaegre; E. Peón; P. Sarría; S. Muñoz; J. A. Rodríguez-Ortiz; F. J. Gotor; Y. Torres
Poster

2017 E-MRS Fall Meeting
18 – 20 septiembre [Varsovia, Polonia]

Sodium borohydride solutions for liquid-state chemical hydrogen storage
G. M. Arzac; V. Godinho; D. Hufschmidt; M. Paladini; M. C. Jiménez de Haro; A. M. Beltrán; A. Fernández
Comunicación oral

Influence of Al and Y content in the oxidation resistance of CrAlYN protective coatings for high temperature applications
T.C. Rojas, S. Domínguez-Meister, M. Brizuela, J.C. Sánchez-López
Poster

Iberian Vacuum Conference, RIVA-X
4 - 6 octubre [Bilbao, España]

Benefits of the multilayer design in the oxidation resistance of cathodic arc evaporated nitride coatings in the energy industry
S. Mato; G. Alcalá; I. Ciarsolo; J. Barriga; F.J. Pérez; J.C. Sánchez-López
Comunicación oral

Energy-Sensitive Ion- and Cathode-Luminescent Radiation-Beam Monitors Based on Multilayer Thin-Film Design
J. Gil-Rostra; F.J. Ferrer; J.P. Espinós; A.R. González-Elipe; F. Yubero
Poster

On the reactive element effect of yttrium in the oxidation behavior of multilayered CrAlYN coatings
T.C. Rojas; S. Domínguez-Meister; M. Brizuela; J.C. Sánchez-López
Poster

Comparative tribological behaviour of MoS2 and WSe2-based nanocomposite coatings
J.C. Sánchez-López; S. Domínguez-Meister; T.C. Rojas; M. Brizuela
Poster

Oxidation resistance of CrAlYN protective hard coatings for high temperature applications: Influence of Al and Y content
T.C. Rojas; S. Domínguez-Meister; M. Brizuela; J.C. Sánchez-López
Poster

FORMACION / TRAINING

FORMACIÓN DE GRADUADOS / MASTER DEGREE THESIS

Título:	**Pt-Cu catalytic coatings for hydrogen combustion: nanostructure and dealloying effects**
Autor:	Francesco Giarratano
Directoras:	Gisela M. Arzac Di Tomaso, M. Asunción Fernández Camacho, Vanda Cristina Fortio Godinho
Grado:	Trabajo Fin de Grado. Universidad de Bolonia
Año Académico:	2017-2018 (21 de marzo 2017)

Título:	**Desarrollo de recubrimientos nanoestructurados protectores por la técnica de magnetrón sputtering**
Autor:	Marina Calero De Ory
Directoras:	Juan Carlos Sánchez López, Teresa Cristina Rojas Ruiz
Grado:	Trabajo Fin de Grado
Año Académico:	2017-2018 (20 de septiembre 2017)

Título:	**Desarrollo y caracterización de recubrimientos nanoporosos por magnetrón sputtering como blancos sólidos de He para aplicacines en física nuclear**
Autor:	David Feria Cervantes
Directoras:	Joaquín Ramírez Rico, Vanda Cristina Fortio Godinho, M. Asunción Fernández Camacho
Grado:	Trabajo Fin de Grado
Año Académico:	2017-2018 (20 de septiembre 2017)

Título:	**Recubrimientos protectores preparados por tecnología de plasma para el sector de la energía y alta temperatura**
Autor:	Alvaro Caro Martínez
Directoras:	Juan Carlos Sánchez López, Teresa Cristina Rojas Ruiz
Grado:	Trabajo Fin de Master
Año Académico:	2017-2018 (14 de diciembre 2017)

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

IQS – Instituto Químico de Sarria		
Barcelona, España	**Neus Sala Bascompte**	2 semanas

DOCENCIA / TEACHING

Máster “Láser, Plasma y Tecnología de Superficies”

Técnicas de caracterización de superficies y láminas delgadas
Cristina Rojas Ruiz

Funcionalización de superficies para aplicaciones mecánicas, protectoras y de bioactividad controladas
Juan Carlos Sánchez López

Interacción de partículas y radiación con la materia
Asunción Fernández Camacho

Lugar: Universidad de Córdoba, Universidad Politécnica de Madrid, Instituto de Ciencia de Materiales de Madrid

EQUIPAMIENTO CIENTÍFICO AVAILABLE EQUIPMENT

Laboratorio de síntesis y catálisis
Material básico de laboratorio químico: PHmetro, agitadores, calefactores, estufa de secado a vacío, centrífuga.
Reactores y material de vidrio convencional para síntesis de nanopartículas y catalizadores por via química.
Rotavapor, sistemas de filtrado.
Cámara seca MBRAUN.
Dos campanas extractoras.
Reactores catalíticos de lecho fijo para catálisis heterogenea solido-gas.
Reactores catalíticos para catálisis heterogenea sólido-líquido.
Sistemas controladores de flujo másico (gases y líquidos evaporados), bombas peristálticas para líquidos.
Tres cromatógrafos de gases, columnas y detectors FID y TCD.
Buretas automáticas para medición de gases.

Cámaras de deposición de recubrimientos y películas delgadas y tratamientos con plasma.
Sistema de tratamiento con plasmas Diener.
Tres cámaras de deposición por la técnica de pulverización catódica (magnetrón sputtering). Con una dotación total de 7 cabezas magnetrón, 2 fuentes DC, 2 fuentes RF y 1 fuente pulsada, portamuestras girables, calentables y “biased”.
Una cámara de deposición por la técnica de pulverización catódica con fuente HIPIMS.

Equipamiento para microscopía electrónica
Sistema de preparación de muestras en película delgada TXP de Leica.
Pulidora, trípode y microscopio óptico.
TEM de 300kV Tecnai F30 dotado de modo STEM, detector HAADF, analizador EDX Oxford Max80 y filtro de energías GIF Quantum.

Propiedades mecánicas de superficies y recubrimientos
Tribómetro CSM (movimiento lineal y rotativo) para evaluación de coeficientes de fricción y desgaste.
Tribómetro de alta temperatura (hasta 800ºC) –Microtest
Calotest para medida de espesores y evaluación del desgaste.
Equipo de rayado (Scratch-test) hasta 200N (Tribotechnic).
Perfilómetro-rugosímetro (Mahr) de tipo táctil y resolución vertical nanométrica.

Propiedades eléctricas de materiales
Sistema de medida de espectroscopía de impedancia compleja, formado por un impedancímetro Agilent modelo 4294A, un horno Hobersal ST115020, y una celda de medida hermética para la realización de medidas en atmósfera.

UNIDAD EXTERNA DE INVESTIGACIÓN: FÍSICA DE MATERIALES

EXTERNAL UNIT: PHYSICS OF MATERIALS

PERSONAL / PERSONNEL

Catedrático

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

Profesor Titular

Dr. Javier S. Blázquez Gámez
Dra. Josefa María Borrego Moro
Dra. María Dolores Estrada de Oya
Dra. M. del Carmen Gallardo Cruz
Dra. Angela Gallardo López
Dr. Felipe Gutiérrez Mora
Dr. José María Martín Olalla
Dra. Ana Morales Rodríguez
Dr. Francisco Javier Romero Landa

Investigador Contratado

Dr. Jhon J. Ipus Bados

Becario Predoctoral

Ldo. Luis M. Moreno Ramírez
Lda. Cristina López Pernía
Ldo. Alejandro F. Manchón Gordón

Proyectos de Investigación / Research Projects

Modelado y Control de la Histéresis en Materiales Magnetocalóricos para Refrigeración y Conversión de Energía

Código/Code: MAT2016-77265-R
Periodo/Period: 30-12-2016 / 29-12-2019
Organismo Financiador/Financial source: Ministerio de Economía y Competitividad
Investigador responsable/Research head: Victorino Franco García / Javier Sebastián Blázquez Gámez
Componentes/Research group: Josefa María Borrego Moro, Alejandro Conde Amiano, Clara Francisca Conde Amiano, Jhon J. Ipus Bados

Materiales Magnéticos y Eficiencia Energética: Caracterización y Modelado Magnetic Materials and Energy Efficiency: Characterization and Models

Código/Code: MAT2013-45165-P
Periodo/Period: 01-01-2014 / 31-12-2017
Organismo Financiador/Financial source: Ministerio de Ciencia e Innovación
Importe total/Total amount: 118.530,61 €
Investigador responsable/Research head: Alejandro Conde Amiano / Victorino Franco García
Componentes/Research group: Clara F. Conde Amiano, Josefa María Borrego Moro, Javier S. Blázquez Gámez , Jhon J. Ipus Bados, Laszlo F. Kish

RESUMEN / ABSTRACT

Se estudiarán materiales magnéticos con aplicacabilidad en sistemas para un uso eficiente de la energía, centrando nuestra atención, fundamentalmente, en dos aspectos interrelacionados: la refrigeración magnética y los materiales compuestos para la recuperación de energía. En lo referente al efecto magnetocalórico, se estudiarán materiales con una transición de fase de primer orden, en los que el control de la microestructura, la composición y campos externos como la presión o el campo eléctrico pueden alterar la respuesta termomagnética. Se abordará la problemática actual de dos familias de materiales magnetocalóricos con alto interés tecnológico: los compuestos del tipo LaFeSi y las aleaciones Heusler. Los primeros, especialmente los compuestos hidrogenados, son altamente prometedores para aplicaciones industria-

les, pero sufren una migración del hidrógeno que da lugar a una disminución del pico de respuesta magnetocalórica que se desdobla en dos picos menores. El control de la microestructura y la modificación de la composición debe dar lugar a una mejora en la estabilidad de las propiedades. Las aleaciones Heusler, a pesar de contar con una elevada respuesta magnetocalórica, presentan dificultades para su aplicación tecnológica, debido a la significativa irreversibilidad de la transición microestructural. Esfuerzos combinados de modificación de la composición y modelado de la transición de fase deben permitirnos minimizar esta histéresis térmica y mejorar la aplicabilidad de las aleaciones Heusler. El estudio experimental de materiales magnetocalóricos requiere de técnicas apropiadas para la caracterización de la variación de entropía magnética y de la variación de temperatura adiabática. La primera puede obtenerse mediante medidas indirectas de imanación, pero los protocolos de medida para materiales con una transición de fase de primer orden existentes en la actualidad implican procedimientos costosos en tiempo (y, consecuentemente, de alto coste económico). La modelización de las transiciones de fase nos permitirá diseñar algoritmos de medida más eficientes. Por otra parte, la caracterización de la respuesta dinámica de los materiales magnetocalóricos sólo puede hacerse mediante la medida directa de la variación de temperatura adiabática, existiendo un número muy limitado de dispositivos comerciales que permitan realizar estas medidas de manera fiable, especialmente para muestras de reducida masa. Realizaremos estudios para desarrollar y optimizar un equipo de medida directa de la variación de temperatura adiabática para el estudio de la respuesta dinámica de los materiales, lo que tendrá implicaciones directas en el análisis de la viabilidad de emplear estos materiales en dispositivos refrigeradores. En especial, se estudiará la posibilidad de realizar medidas con excitación AC para la detección de pequeñas respuestas. El tercer bloque de estudio de este proyecto se centra en el análisis de otros efectos cruzados en materiales con transiciones de fase magnéticas o magnetoestructurales. Mediante el desarrollo de materiales multiferroicos artificiales, preparados por procedimientos combinados de aleado mecánico y "spin coating", estudiaremos la viabilidad de la recuperación de energía por medios termomagnéticos.

In this project, we will study magnetic materials with applicability in systems for energy efficiency. Our interest will be focused in two interrelated topics: magnetic refrigeration and composite materials for energy harvesting. Concerning magnetocaloric effect, we will study materials with a first order phase transition, which thermomagnetic response can be controlled through tailoring microstructure and composition. Other external excitations, such as pressure and electric field, can also affect the thermomagnetic response. We will focus on some of the currently open problems of two families of magnetocaloric materials with high technological interest: LaFeSi-type compounds and Heusler alloys. In hydrogenated compounds of the former family, despite their promising industrial applicability, hydrogen migrates yielding a reduction of the peak of the magnetocaloric response, which splits in two smaller peaks. The control of the microstructure and the compositional tailoring should lead to an enhancement of the stability of the properties. Heusler alloys, despite their high magnetocaloric response, show a strong irreversibility at the microstructural transition, which hinders their technological application. Tailoring the composition along with modelling of the phase transition should allow us to minimize the detrimental thermal hysteresis and to enhance the applicability of Heusler alloys. The experimental study of magnetocaloric materials requires appropriate techniques for the characterization of the isothermal magnetic entropy change and the adiabatic temperature change. Although the former can be obtained from indirect measurements of magnetization, the measurement protocols for materials with a first order phase transition used nowadays demand long time measurements (and, consequently, have a high economical cost). Modeling of the phase transitions will allow us to design more efficient measurement algorithms. On the other hand, the characterization of the dynamic response of the magnetocaloric materials can only be performed by direct measurements of the adiabatic temperature change. However, there is a reduced number of commercial devices supplying reliable measurements of this magnitude, especially for samples with a low mass. We will carry out studies for developing and optimizing a setup for the characterization of the dynamic response of materials. This will have direct implications in the analysis of the viability to use these materials in refrigerators. Particularly, we will study the viability to perform AC measurements to detect weak reponses. Finally, the analysis of cross-effects in materials with magnetic or magnetostructural transitions will be undertaken. Especially, we will study the viability for thermomagnetic energy harvesting through the development of artificial multiferroic materials prepared by combined mechanical alloying and spin coating techniques.

Unión Europea
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MINISTERIO DE ECONOMÍA Y COMPETITIVIDAD

Procesado y caracterización microestructural, mecánica y eléctrica de compuestos cerámica-grafeno Processing and microstructural, mechanical and electrical characterization of ceramic-graphene composites

Código/Code:	MAT2015-67889-P
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Organismo Financiador/Financial source:	Ministerio de Ciencia e Innovación
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Investigador responsable/Research head:	Ángela Gallardo López / Rosalía Poyato Galán
Componentes/Research group:	Antonio Muñoz Bernabé, Ana Morales Rodríguez, Felipe Gutiérrez Mora.

RESUMEN / ABSTRACT

En la actualidad se plantean interesantes expectativas sobre los compuestos cerámica-grafeno, propuestos para aplicaciones en catálisis, almacenamiento y conversión de energía, protección del medio ambiente y biotecnología. Pero aún se requieren importantes esfuerzos para dar respuesta a cuestiones abiertas. Hay que incidir en aspectos como la resistencia a la cizalla de las intercaras cerámica-grafeno -esencial para asegurar la transferencia de carga efectiva sobre las láminas de grafeno-, la distribución homogénea del grafeno en la matriz cerámica y la relación de tamaños entre matriz y refuerzo, para maximizar el aumento de tenacidad y de conductividad eléctrica, así como también explorar sus propiedades mecánicas a alta temperatura.

En este proyecto se plantea un estudio sistemático de compuestos de matrices cerámicas con grafeno, desde la fabricación hasta la caracterización microestructural, mecánica y eléctrica, con el objetivo de mejorar la comprensión de los mecanismos que controlan estas propiedades al incorporar nanoestructuras de grafeno a una matriz cerámica. Se procesarán compuestos de

dos matrices cerámicas diferentes, de alúmina y de circona tetragonal dopada con óxido de itrio (3YTZP), con grafeno mediante técnicas coloidales, prestando especial atención a la dispersión del grafeno en la matriz cerámica, aspecto no exento de dificultades y que es clave para conseguir la mejora de las propiedades. La sinterización se realizará en un horno de descarga de plasma (SPS, spark plasma sintering) de última generación, optimizando las condiciones para conseguir compuestos densos y de tamaño de grano nanométrico. Para el análisis microestructural se utilizarán técnicas como la difracción de rayos X, la espectroscopía Raman, y la microscopía electrónica de barrido y transmisión. Con ellas se evaluarán las fases cristalográficas presentes, el tamaño de grano, la distribución de las nanoestructuras de grafeno, etc.

Desde el punto de vista del diseño de materiales avanzados, es fundamental investigar la relación entre microestructura y propiedades mecánicas y eléctricas. Las propiedades mecánicas a temperatura ambiente (dureza, tenacidad a la fractura y resistencia a flexión) se abordarán mediante indentación y ensayos de flexión, a escalas macro y microscópica. A alta temperatura, se estudiará la deformación plástica de los compuestos cerámica-grafeno mediante ensayos de fluencia en atmósferas controladas. También se estudiará el comportamiento tribológico de los compuestos y se evaluará su conductividad eléctrica, una de las propiedades más interesantes ya que se modifica de forma notoria como resultado de la incorporación del grafeno a estos sistemas cerámicos. La respuesta eléctrica se analizará en un amplio rango de temperaturas, bien mediante espectroscopía de impedancia compleja, bien mediante medidas de conductividad en corriente continua en el caso de los compuestos menos resistivos.

Nowadays, interesting prospects are proposed for ceramic-graphene composites, in application fields such as catalysis, energy storage and conversion, environment protection and biotechnology. A great effort is still required to answer open questions. Issues such as shear resistance of the ceramic-graphene interface –essential to obtain an effective load transfer to the graphene sheets-, distribution of graphene in the ceramic matrix -to maximize the reinforcement mechanisms and electrical conductivity- and the high temperature mechanical properties in these composites need special attention.

A systematic study of ceramic matrix graphene composites, including processing and microstructural, mechanical and electrical characterization is proposed in this project, with the aim of improving the comprehension of mechanisms controlling these properties when adding graphene nanostructures to a ceramic matrix. Both alumina and yttria tetragonal zirconia (3YTZP) graphene composites will be processed by means of colloidal techniques. Special attention will be devoted to the dispersion of graphene in the ceramic matrix which is not a straightforward aspect, but is key to improve mechanical and functional properties. Sintering will be carried out by spark plasma sintering, SPS. Conditions will be optimized in order to obtain fully dense composites with nanometric grain size. Microstructural analysis will be performed by X ray diffraction, Raman spectroscopy, scanning and transmission electron microscopy (SEM and TEM). The present crystallographic phases, grain size and distribution of graphene nanostructures will be evaluated.

In order to design advanced materials, it is necessary to study the relationship between microstructure and mechanical or electrical properties. Room temperature mechanical properties (hardness, fracture toughness and flexural resistance) will be characterized by indentation and bending tests at macro and microscopic scales. At high temperature, the plastic behavior of these ceramic-graphene composites will be assessed by creep tests under controlled atmosphere. Tribological behavior of the composites will also be studied to evaluate their resistance to wear. The electrical response will be assessed in a wide range of temperatures by means of complex impedance spectroscopy or by direct current conductivity measurements in the composites with lower resistivity. This is a most interesting property since it can be strongly increased when incorporating graphene to these ceramic systems.

Procesado, caracterización y propiedades mecánicas de cerámicos nanoestructurados reforzados con nanotubos de carbono

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Organismo Financiador/Financial source: Junta de Andalucía
Investigador responsable/Research head: Arturo Domínguez Rodríguez
Componentes/Research group: Luis María Esquivias Fedriani, Angela Gallardo López, Diego Gómez García, Felipe Gutiérrez Mora, Victor Morales Flórez, Ana Morales Rodríguez, Rodrígo Moreno Botella, Antonio Muñoz Bernabé, Rosalía Poyato Galán, Eugenio Zapata Solvas

PROYECTOS EN LOS QUE PARTICIPAN INTEGRANTES DE LA UNIDAD

Avalanchas en Biofísica, Materiales y Plasmas

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Investigador responsable/Research head: Eduard Vives-Santa-Eulalia (U. Barcelona)
Investigador responsable (UEI) Research head (UEI): María del Carmen Gallardo Cruz
Componentes/Research group: José María Martín Olalla, Francsico Javier Romero Landa

RESUMEN / ABSTRACT

Bajo la denominación genérica de “avalanchas” se incluyen una serie de fenómenos físicos que consisten en la respuesta discontinua, estocástica e intermitente de un sistema cuando se conduce de forma suave y continua: variaciones repentinas del parámetro de orden ocurren separadas por intervalos sin ninguna actividad aparente. Existen ejemplos muy variados y a distintas escalas espaciales y temporales que incluyen la fractura de un material poroso nanoestructurado bajo compresión, las transiciones de fase estructurales en sólidos, el ruido Barkhausen en un material ferromagnético cuando se imanta, algunos fenómenos de condensación y ebullición, la respuesta de los tejidos neuronales, las verdaderas avalanchas (aludes) de nieve, los terremotos o incluso los plasmas de fusión y las llamaradas solares. Las dimensiones espaciales, energéticas y las duraciones de las avalanchas suelen presentar distribuciones estadísticas que, si no son de tipo ley de potencias, en general tienen colas largas. Es decir su comportamiento se aleja del comportamiento gaussiano esperable. El objetivo de esta red es discutir desde una aproximación multidisciplinar y holística las diferencias y similitudes entre estos fenómenos y la posibilidad que se puedan describir bajo un marco físico-estadístico común. Tambien se pretende importar y exportar las técnicas experimentales y estadísticas para el estudio de las correlaciones temporales y espaciales de las avalanchas entre las diferentes disciplinas: física de materiales, biofísica, geofísica, física de plasmas. El reto final sería contribuir de forma importante al conocimiento y a la mejora de los métodos de caracterización, evaluación de riesgos, predicción y alarma de estos fenómenos, desde las fracturas en materiales hasta los terremotos y las llamaradas solares.

Under the name of “avalanches” we designate many physical phenomena that consist in the discontinuous, stochastic and intermittent response of a system under smooth and continuous external driving: sharp variations of the order parameter occur separated by intervals with apparently no activity. There are many examples at different spatial and temporal scales including: the failure of nanostructured porous materials under compression, the structural transitions in solids, Barkhausen noise in ferromagnetic materials, some condensation and boiling phenomena, the response of neural tissues, the real snow avalanches, earthquakes and even fusion plasmas and solar flares. The spatial and energetic dimensions as well as the durations of the avalanches display often power-law behavior or, at least, fat tails. Their behaviour clearly differs from the expected gaussian behaviour. The goal of this network is to discuss from a multidisciplinar and holistic approximation the differences and similarities of these phenomena and the possibility to describe all of them in a comon statistical mechanics framework. We also pretend to export and import the experimental and statistical analysis techniques for the study of the spatial and temporal correlations of the avalanches in the different disciplines: materials physics, biophysics, geophysics and plasma physics. The final goal will be to contribute to the knowledge and improvement of the methods for characterization, risk evaluation, forecast and alarm of these phenomena, from materials failure to earthquakes and solar flares.

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

Nanostructuring as a procedure to control the field dependence of the magnetocaloric effect
D. Doblas, L. M. Moreno-Ramirez, V. Franco, A. Conde, A. V. Svalov, G. V. Kurlyandskaya
Materials & Design, **114** (2017) 214-219
DOI: 10.1016/j.matdes.2016.11.085

In this work, the field dependence of the magnetocaloric effect of Gd bulk samples has been enhanced through nanostructuring of the material. Nanostructuring consists in multilayers preparation by alternative rf-sputtering deposition of Gd layers and Ti spacers onto glass substrates. The results obtained for the multilayers were compared to those obtained for the Gd bulk. Assuming a power law for the field dependence of the magnetic entropy change (Delta S-M alpha H-n), higher field dependences close to the transition in a wider temperature range are obtained for the multilayer material (n=1.0) with respect to the bulk counterpart (n=0.78). The effect of a Curie temperature distribution in the multilayer material (due to variations of the layer thickness) has been studied through numerical simulations to explain the observed field dependence of the magnetocaloric effect, obtaining a remarkable agreement between experiments and results.

Time evolution of mechanical amorphization: A kinetic model
J.S. Blázquez, J.J. Ipus, A. Conde
Scripta Materialia, **130** (2017) 260-219
DOI: 10.1016/j.scriptamat.2016.12.019

A model is proposed to describe the evolution of the amorphization process driven by mechanical grinding. This model considers the interface controlled growth of the disordered regions at the boundaries between the crystallites (unlike the dassical nucleation and growth theory, the growing particles are thus concave which affects the kinetic equation). The validity of this model has been tested for the amorphization process of mechanically alloyed Fe-Nb-B system at different frequencies. Predictions of the model are in agreement with the experimental results and values of the linear growth rate and the thickness of the starting disordered layer are obtained.

Two different critical regimes enclosed in the Bean-Rodbell model and their implications for the field dependence and universal scaling of the magnetocaloric effect
C. Romero-Muniz, V. Franco, A. Conde
Physical Chemistry Chemical Physics, **19** (2017) 3582-3595
DOI: 10.1039/c6cp06291a

In the last few years power laws and universal scaling have been extensively used to study the field dependence of the magnitudes involved in the magnetocaloric effect of materials. They are key tools which allow us to compare the performing properties of different materials regardless of their nature, processing or experimental conditions during measurements. It was proved that power laws and universal scaling are a direct consequence of critical phenomena in the neighborhood of phase transitions. However, there remains some controversy about the reliability of these procedures. In this work we use the well-known Bean-Rodbell model to confirm that these features are unmistakably related to the critical behavior of the continuous phase transitions. In this specific model, universal scaling occurs either at a purely mean field second order transition or at a tricritical point. Finally, we analyze in detail if the universal scaling is compatible with materials at the tricitical point, making a comprehensive comparison with available experimental data from the literature. We conclude that it is really difficult to know with full certainty if a sample really is in the tricritical regime.

Normal and inverse magnetocaloric effects in structurally disordered Laves phase Y1-xGdxCo2 (0 <= x <= 1) compounds

N. Pierunek, Z. Sniadecki, M. Werwinski, B. Wasilewski, V. Franco, B. Idzikowski

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Magnetic and magnetocaloric properties of Y1-xGdxCo2 compounds, where x = 0.2, 0.4, 0.6, 0.8 and 1.0, were investigated experimentally and theoretically. Crystal structures were characterized by X-ray diffraction (Rietveld analysis) and investigated samples possess the MgCu2-type single phase with Fd-3m space group. Melt-spinning process introduced a chemical and topological disorder, which directly affected the magnetic properties. Refrigerant capacity (RC), strictly connected to the full width at half maximum delta T-FWHM of the Delta S-M(T) curve and the maximum of magnetic entropy changes Delta S-Mpk(T,Delta H), increases from 29 to 148 J/kg with replacement of Y by Gd atoms from x = 0.2 to x = 0.8. RC and delta T-FWHM indicate the presence of disorder. Temperature dependences of magnetic entropy change Delta S-M(T,Delta H) and RC were measured in as-quenched and annealed state for Y0.4Gd0.6Co2. This particular composition was chosen for detailed investigation mainly due to its Curie point (T-c = 282 K), which is close to the room temperature. After isothermal annealing (T-a = 60 min, T-a = 700 degrees C) RC decreased from 122 to 104 J/kg, which clearly indicates the homogenization of the heat treated sample. Furthermore, observed inverse magnetocaloric effect is associated with the presence of antiferromagnetically coupled Gd and Co magnetic moments. The phase transition temperature increases with increasing Gd content from 74 to 407 K for Y0.8Gd0.2Co2 and GdCo2, respectively. Within the FPLO-LDA DFT method the non-magnetic ground state for YCo2 and the magnetic ground state for GdCo2 are predicted in agreement with experiment. The dependence of calculated total and species-resolved magnetic moments on Gd concentration reasonably agrees with available experimental data.

J.Y. Law, J. Rial, M. Villanueva, N. Lopez, J. Camarero, L.G. Marshall, J.S. Blazquez, J.M. Borrego, V. Franco, A. Conde, L.H. Lewis, A. Bollero

N. Pierunek, Z. Sniadecki, M. Werwinski, B. Wasilewski, V. Franco, B. Idzikowski

Journal of Alloys and Compounds, **712** (2017) 373-378

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Gas-atomized Mn54Al46 particles constituted nominally of only epsilon- and gamma 2-phases, i.e. no content of the ferromagnetic Llo-type tau-phase, have been used to study the evolution of phases during short time of high-energy milling and subsequent annealing. Milling for 3 min is sufficient to begin formation of the tau-MnAl phase. A large coercivity of 4.9 kOe has been obtained in milled powder after annealing at 355 degrees C for 10 min. The large increase in coercivity, by comparison with the lower value of 1.8 kOe obtained for the starting material after the same annealing conditions, is attributed to the combined formation of the tau-MnAl and beta-Mn phases and the creation of a very fine microstructure with grain sizes on the order of 20 nm. Correlation between morphology, microstructure and magnetic properties of the rapidly milled MnAl powders constitutes a technological advance to prepare highly coercive MnAl powders.

Grinding and particle size selection as a procedure to enhance the magnetocaloric response of La(Fe, Si)(13) bulk samples

J.J. Ipus, J.M. Borrego, L.M. Moreno-Ramírez, J.S. Blázquez, V. Franco, A. Conde
Intermetallics, **84** (2017) 30-34
DOI: 10.1016/j.intermet.2016.12.022

The magnetocaloric effect of La(Fe, Si)(13) samples deteriorates with the presence of secondary phases. However, it is highly challenging to produce single phase samples by conventional procedures and purification requires long annealing time. We propose grinding and particle size selection of non-optimal starting samples as procedure to enhance the magnetocaloric response. In this study a starting multi-phase LaFe11.8Si1.2 ingot was grinded and sieved to select three different particle size ranges. X-ray diffraction and Mossbauer spectrometry reveal that all samples mainly contain fcc-La(Fe, Si)(13) and bcc-Fe(Si) phases. Microstructural and magnetic results show that the fcc-La(Fe, Si)(13) phase fraction increases for samples with the smallest average particle size, increasing their magnetocaloric response in a factor larger than three with respect to that of the bulk sample.

Dense graphene nanoplatelet/yttria tetragonal zirconia composites: Processing, hardness and electrical conductivity

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Ceramics International, **43(15)** (2017) 11743-11752
DOI: 10.1016/j.ceramint.2017.06.007

Yttria tetragonal zirconia ceramic composites with 1, 2.5, 5 and 10 vol% nominal contents of graphene nanoplatelets (GNPs) were fabricated and characterized. First, the GNP dispersion in isopropanol was optimized to de-agglomerate the GNPs without damaging their structure. Then, submicrometric fully dense composites were obtained via spark plasma sintering (SPS) at 1250 degrees C with a 5 min holding time. The processing routine produced a nearly homogeneous GNP dispersion in the ceramic matrix, and the GNPs preferential orientation was perpendicular to the sintering compression axis. A ceramic grain refinement due to the GNPs was also detected. The Vickers hardness measured on the plane perpendicular to the sintering compression axis (basal plane) was lower than on the cross sections. This anisotropy increased with the increasing GNP content, while the average hardness decreased. The electrical conductivity was also highly anisotropic, up to seven times higher for the basal planes. The electrical percolation threshold for these composites was estimated to be between 2.2 and 4.4 vol% of the GNP measured content.

Effect of acid-treatment and colloidal-processing conditions on the room temperature mechanical and electrical properties of 3YTZP/MWNT ceramic nanocomposites

R. Poyato, A. Morales-Rodríguez, F. Gutiérrez-Mora, A. Muñoz, A. Gallardo-López
Ceramics International, **43(18)** (2017) 16560-16568
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Different colloidal powder processing routines have been used to prepare composites of 3 mol% Y2O3-ZrO2 (tetragonal zirconia polycrystals, 3YTZP) with 2.5 vol% multiwall carbon nanotubes (MWNT) with the aim of achieving a homogeneous distribution of the MWNTs in the ceramic, eliminating agglomerates but also minimizing carbon nanotube (CNT) damage during processing. Modifications of the acid treatment applied to the nanotubes, including subjecting them to stirring or ultrasonic agitation, and use of acid or basic pH during composite powder mixing have been approached.

No MWNT damage during processing was detected by Raman spectroscopy. CNT bundles were found in all the composites forming different patterns depending on the processing route. Similar values of hardness were obtained for all the composites, while different anisotropy in fracture propagation was found when studying parallel and perpendicular directions to the sintering pressing axis on the cross sections of the composites due to the MWNT preferential alignment. The CNT bundles were found to act as fracture short paths. A similar anisotropic behavior was observed for the electrical conductivity. These results have been correlated to the different microstructures obtained in the composites prepared with different processing routines.

Tunable magnetocaloric effect around room temperature by Fe doping in Mn0.98Cr(0.02-x)FexAs compound

Journal of Magnetism and Magnetic Materials, **436** (2017) 85-90
DOI: 10.1016/j.jmmm.2017.04.030

In this work, we present an investigation of the magnetic and magnetocaloric properties of Mn0.98Cr((0.02-x))FexAs compounds with x = 0.002, 0.005 and 0.010. Our findings show that as Fe content increases the unit cell volume decreases, which indicates that Fe doping emulates the pressure effect on the crystalline structure. The transition temperature T-C decreases as x increases and it can be set at approximate value of room temperature by changing the doping level. In addition, the magnetic entropy change Delta S-M was determined using a discontinuous measurement protocol, and realistic values from the magnetocaloric effect presented by MnAs-type compounds under pressure (emulated pressure) could be obtained. The values of Delta S-M(MAX) are very large, around -11 J kg(-1) K-1 with Delta H = 15 kOe, which is higher than that observed for most compounds with TC around room temperature. However, Delta S-M is confined to a narrow temperature range of 11 K. To overcome this drawback, the composition of a theoretical composite formed by our samples was calculated in order to obtain a table-shaped Delta S-M curve. The simulated composite showed a high value of full width at half maximum delta T-FWHM of 33 K, which is much higher than that of single sample.

Ball milling as a way to produce magnetic and magnetocaloric materials: a review

J.S. Blázquez, J.J. Ipus, L.M. Moreno-Ramírez, J.M. Álvarez-Gómez, D. Sánchez-Jiménez, S. Lozano-Pérez, V. Franco, A. Conde
Journal of Materials Science, **52 (20)** (2017) 11834-11850
DOI: 10.1007/s10853-017-1089-3

Ball milling (BM) is a well-established technique for producing different materials in powder shape. Dynamical analysis of BM helps to optimize the process through simple but general relations (e.g., definition of an equivalent milling time). Concerning the field of study of magnetocaloric effect (MCE), BM is used in different ways: as a single step process (mechanical alloying), as an initial step to enhance mixing of the elements (e.g., to speed up the formation of the desired intermetallic phase) or as a final step (e.g., hydriding of La-Fe-Si). In this contribution, besides a simple description of the effects of some geometrical parameters on the power released during BM and a short review of the BM contribution to the research field of MCE, we will discuss the effect of the microstructure of the starting material and the granular shape inherent to BM on magnetic materials exhibiting MCE.

Mechanochemistry of copper sulphides: phase interchanges during milling
M. Balaz, A. Zorkovska, J.S. Blazquez, N. Daneu, P. Balaz
Journal of Materials Science, **52 (20)** (2017) 11947-11961
DOI: 10.1007/s10853-017-1189-0

Covellite, CuS and chalcocite, Cu2S nanoparticles prepared in the explosive manner from elemental precursors were further ball-milled in order to observe additional changes caused by mechanical action. Three phases of chalcocite were interchanging during milling, monoclinic one being major at the equilibrium after 30 min. In the case of covellite synthesis, milling for 15 min brought about a significant diminishment in the content of digenite, Cu1.8S, impurity. Covellite powder exhibited finer character than chalcocite, as documented by crystallite size, grain size and specific surface area analysis. Finally, the effect of milling speed on the explosive character of the reaction and phase composition of chalcocite was investigated. The most drastic conditions favored the formation of the monoclinic phase with the lowest symmetry and the time and intensity of the explosion was found to depend on the milling speed. The whole process is mechanically driven.

Predicting the tricritical point composition of a series of LaFeSi magnetocaloric alloys via universal scaling
V. Franco, J.Y. Law, A. Conde, V. Brabander, D.Y. Karpenkov, I. Radulov, K. Skokov, O. Gutfleisch
Journal of Physics D-Applied Physics, **50** (41) (2017) 414004
DOI: 10.1088/1361-6463/aa8792

Typically, the most common techniques to determine the order of the phase transition of magnetocaloric materials do not allow a quantitative prediction of the composition that corresponds to the change from first to second order phase transition, i.e. the so-called tricritical point of an alloy series. In this work, we study a series of LaFeSi alloys in which the different Fe/Si ratio produces a change in the order of the transition. We show the quantitative prediction of the tricritical composition analyzing the field dependence of the peak magnetic entropy change. The proposed procedure is also simple to implement, as it does not require any modelling of the experimental data and it only requires temperature and field dependent magnetization experiments. It is shown that the obtained value is in agreement with the results emerging from the most commonly used qualitative techniques.

Influence of noise on the determination of Curie temperature from magnetocaloric analysis
L.M. Moreno-Ramírez, V. Franco, M. Pekala, A. Conde
IEEE Transactions on Magnetics, **53** (11) (2017) 2502004
DOI: 10.1109/TMAG.2017.2705421

In this paper, we study the effect of the signal-to-noise ratio of magnetization measurements on the determination of the Curie temperature from the analysis of the magnetocaloric response. The procedure has been compared with the method of the inflection point of the magnetization versus temperature curves. Magnetization data have been simulated using the Arrott-Noakes equation of state, with the addition of different noise levels (either 1%of the measured signal or 0.3% of the measurement range). It is shown that the obtained values of the Curie temperature are more accurate in the case of the magnetocaloric procedure, although this method requires more data analysis than the inflection point method. Moreover, the field independence of the Curie temperature obtained from the magnetocaloric procedure allows us to perform a statistical analysis of the obtained values, reducing the associated error in the Curie temperature determination.

Scaling Analysis of the Magnetocaloric Effect in Co/Au Nanoparticles
P. Hrubovcak, A. Zelenakova, V. Zelenak, V. Franco
Acta Physica Polonica A, **131** (4) (2017) 795-797
DOI: 10.12693/APhysPolA.131.795

The system of superparamagnetic Co/Au bimetallic nanoparticles of average diameter 7 nm was investigated with respect to its magnetocaloric properties. DC magnetic measurements revealed the presence of field dependent zero field cooled M(T) maximum (6-8 K) and significant zero field cooled/ field cooled irreversibility at low temperatures in the system. Documented thermal hysteresis disallow standard magnetic entropy change calculation from isothermal M(H) data, thus we attempted to employ zero field cooled M(T) data for this purpose. Magnetic entropy change was calculated employing the Maxwell relation. In maximal field variation of 1 T relative high magnetic entropy change for nanoparticles triangle S-M 0.7J/(kg K) at T = 9 K was observed. The data collapsed onto single universal curve after proper axis rescaling.

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

European Advanced Materials Congress 2017

22-24 Agosto [Estocolmo, Suecia]

Enhancing processing, microstructure and properties of graphene/3YTZP ceramic composites.
Gallardo-López, A; Muñoz-Ferreiro, C; Morales-Rodríguez, A; Gutiérrez-Mora, F; Poyato, R.
Oral

Influence of graphene nanoplatelets orientation in a ceramic matrix on its scratch behavior.
Gutiérrez-Mora, F; Gallardo-López, A; Muñoz-Ferreiro, C; Morales-Rodríguez, A; Muñoz, A; Poyato, R.
Poster

15th Conference & Exhibition of the European Ceramic Society

9-13 julio [Budapest, Hungría]

Effect of graphene content on the tribological behaviour of 3YTZP reinforced with graphene nanoplatelets.
Gutiérrez-Mora, F; Gallardo-López, A; Morales-Rodríguez, A; Poyato, R.
Oral

Graphene nanoplatelet/3 mol% yttria doped zirconia composites with high electrical conductivity.
Poyato, R; Osuna, J; Morales-Rodríguez, A; Gallardo-López, A.
Oral

Graphene-ceramic composites with 3YTZP matrix.
Gallardo-López, A; Muñoz-Ferreiro, C; Morales-Rodríguez, A; Poyato, R.
Poster

Microstructure and properties of pressureless sintered graphene/3YTZP composites.
López-Pernia, C; Poyato, R; Morales-Rodríguez, A; Gallardo-López, A.
Poster

62nd Annual Conference on Magnetism and Magnetic Materials

4-10 Noviembre 2017 [Pittsburgh (EEUU)]

A new procedure of the determination of the order of thermomagnetic phase transition
J. Y. Law, V. Franco, A. Conde, V. Brabander, D. Y. Karpenkov, I. A. Radulov, K. Skokov, O. Gutfleisch
Invitada

INCOME

3-7 Septiembre 2017 [Kosice (Eslovaquia)]

Mössbauer study of the kinetics of mechanical amorphization in Fe70Zr30
A. F. Manchón-Gordón, J. J. Ipus, J. S. Blázquez, C. F. Conde, A. Conde
Oral

Intermag 2017

24-28 Abril 2017 [Dublín (Irlanda)]

The influence of noise on the Determination of the Curie Temperature from magnetocaloric measurements
L. M. Moreno-Ramírez; V. Franco; M. Pekala; A. Conde
Póster

Modification of the field dependence and scaling of the magnetocaloric effect in LaFeSi across the tricritical point
V. Franco, J. Y. Law, A. Conde, V. Brabander, D. Y. Karpenkov, L. A. Radulov, K. Skokov, O. Gutfleisch
Oral

11th Symposium on Hysteresis Modeling and Micromagnetics (HMM 2017)

29-31 Mayo 2017 [Barcelona (España)]

T-FORC as a tool for the characterization of magnetocaloric materials
V. Franco, A. Conde
Oral

62nd Annual Conference on Magnetism and Magnetic Materials

4-10 Noviembre 2017 [Pittsburgh (EEUU)]

How low is the temperature that we need to reach in calorimetric measurements to accurately obtain the magnetocaloric effect? The Gd and GdSiGe cases
L.M. Moreno-Ramírez; V. Franco; A. Conde; H. Neves Bez; Y. Mudryk; V.K. Pecharsky
Oral

23rd Soft Magnetic Materials Conference

10-13 Septiembre 2017 [Sevilla (España)]

Optimization of the supersaturated solid solution produced by mechanical alloying as a precursor of the La(Fe,Si)13 phase
J.J. Ipus, J.S. Blázquez, L.M. Moreno-Ramírez, V. Franco, A. Conde
Póster

Magnetocaloric response and critical exponents of Fe70Zr30 mechanically alloyed systems
A.F. Manchón-Gordón, J.J. Ipus, L.M. Moreno-Ramírez, J.S. Blázquez, C.F. Conde, V. Franco, A. Conde
Póster

Magnetocaloric effect and Demagnetizing factor in La0.7Ca0.3Mn1-xNixO3 (x = 0, 0.02, 0.07, 0.1)
A. Gómez, O. Morán, E. Chavarriaga, N. R. Rojas, J. S. Blázquez, A. Conde, J. L. Izquierdo, C. Parra, I. Supelano
Póster

Effect of low temperature truncated calorimetric measurements on the magnetocaloric properties of biphasic materials
L. M. Moreno-Ramírez;V. Franco; A. Conde
Póster

FORMACION / TRAINING

FORMACIÓN DE GRADUADOS / MASTER DEGREE THESIS

Título:	Estudio de materiales 3D fabricados a partir de nanoplaquetas de grafeno
Autor:	Manuel García Palma
Directores:	Ángela Gallardo López/Rosalía Poyato/Ana Morales Rodríguez.
Grado:	Trabajo Fin de Grado en Ingeniería de Materiales
Año Académico:	2017-2018 (27 Noviembre 2017)

DOCENCIA / TEACHING

Máster Profesorado de ESO y Bachillerato, FP
Complementos de formación disciplinar en Física y Química
Dr. Javier S. Blázquez
Lugar: Universidad de Sevilla

EQUIPAMIENTO CIENTÍFICO AVAILABLE EQUIPMENT

- Calorímetro de barrido diferencial (Perkin-Elmer DSC7)
- Criostato para espectrómetro Móssbauer
- Balanza termogravimétrica (Perkin-Elmer TGA-7)
- Espectrómetro Mössbauer (Wissel MB-500) con horno y criostato.
- Coercímetro (desarrollado en el laboratorio)
- Magnetómetro de muestra vibrante (LakeShore 7000) con horno y criostato.
- Equipo de medida directa de temperatura adiabática (Advanced Magnetic Technologies).
- Molino Planetario (Fritszch Pulverisette Vario 4)
- Equipo de solidificación por enfriamiento ultrarrápido (melt spinning, Bühler)
- Equipo de fusión por arco (MAM1, Bühler)
- Calorímetro de conducción, resolución en la medida del flujo de calor mejor que 0,1 W, fluctuaciones en temperatura del orden de 10-6 K, velocidad de barrido menor de 0,01K/h, rango 80-320K, es posible aplicar tensión uniaxial hasta 30 kg/cm2 y campo eléctrico hasta 800V/cm
- Calorímetro de conducción, rango 80-400K, campo eléctricos 2000V/cm
- Medida de constante dieléctrica, en los mismos calorímetros,
- Medida de ciclo de histéresis en Ferroeléctricos.
- Analizador de Impedancia

SERVICIOS GENERALES

GENERAL SERVICES

SERVICIO DE ESPECTROSCOPIAS / SPECTROSCOPY SERVICE

El Servicio de Espectroscopias incluye las Unidades de Espectroscopía 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: Raman Spectroscopy, Infrared Spectroscopy and Ultraviolet-Visible Spectroscopy. It is devoted to the determination of molecular structure of chemical compounds and materials.

ESPECTROSCOPÍA MICRO-RAMAN / MICRO-RAMAN SPECTROSCOPY

La espectroscopía Raman se basa en un proceso fotónico en el que la radiación incidente es dispersada por la muestra, produciéndose transiciones de tipo vibracional y rotacional. En general, el espectro Raman se interpreta como un espectro vibracional que ofrece información muy similar al espectro de infrarrojo, aunque las vibraciones que se ven reflejadas en el espectro Raman no son siempre las mismas que en aquél. Para que un modo vibracional sea activo en espectroscopía Raman es necesario que se produzcan cambios en la polarizabilidad de los enlaces químicos o la molécula considerada, lo que conlleva la producción de momentos dipolares inducidos. Su campo de aplicación es muy extenso: semiconductores, compuestos del carbono (grafito, diamante, nanotubos, fibras…), catalizadores, pigmentos, etc.

Raman spectroscopy is based on a photonic process in which the incident radiation is dispersed by the sample. This latter is perturbed leading to vibrational and rotational transitions. In general, the Raman spectrum is interpreted like a vibrational one, providing information very similar to the infrared spectroscopy, although the Raman active vibrations are not always the same as those excited with infrared radiation. A Raman vibration mode is active if there is a change of polarizability of the chemical bonds or the considered molecule, which in turn results in the generation of induced dipolar momentam. Its application fields are very broad: semiconductors, carbon compounds (graphite, diamond, nanotubes, fibers…), catalysts, pigments, etc.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- LabRAM Horiba Jobin Yvon dotado de un microscopio confocal y 3 longitudes de excitación (785 cm^{-1} rojo, 532 cm^{-1} verde, y 325 cm^{-1} UV)
  LabRAM Horiba Jobin Yvon equipped with a confocal microscope and 3 excitation lasers (785 cm^{-1} red, 532 cm^{-1} green, and 325 cm^{-1} UV)

Responsables Científicos/ Scientific Responsibles: Dr. Juan Carlos Sánchez López, Dr. Hernán Míguez García y Dr. Miguel Angel Centeno Gallego
Personal Técnico/ Technical Assistant: Dr. Miguel Angel Avilés Escaño

ESPECTROSCOPÍA INFRARROJA / INFRARED SPECTROSCOPIES

La espectroscopía de Infrarrojos (FT-IR) se basa en la absorción de radiación infrarroja por parte de los materiales. Esta absorción supone un cambio en la energía vibracional de los enlaces, siempre que se produzca un cambio en la polarización de dicho enlace. El resultado obtenido es un espectro en el que se representa la radiación absorbida o transmitida en función del número de onda de la radiación, lo cual permite identificar el enlace correspondiente.
El equipo en el ICMS cubre un rango de número de ondas que va desde 5000 a 250 cm-1 (óptica de CsI) y se puede trabajar con purga o en vacío. Se halla equipado con accesorios para trabajar en los modos de Reflectancia Difusa (DRIFT), Reflectancia Total Atenuada (ATR) y Reflexión Especular. Dispone de un microscopio de Infrarrojos que tiene una resolución lateral de 10 µm.

Infrared spectroscopy (FT-IR) is based on the selective absorption of the infrared radiation by the materials. This absorption means a change in the vibrational energy of the chemical bonds, whenever it occurs a change in the polarization. The result is a spectrum showing the absorbed or transmitted radiation as a function of the wavenumber of the radiation, which can be assigned to the corresponding chemical bound.
The equipment at the ICMS works in a wavenumber range from 5000 to 250 cm^{-1} (CsI optic), and can operate with a gas purge or in vacuum. It is equipped with several accessories to do Diffuse Reflectance (DRIFT), Attenuated Total Reflectance (ATR) or Specular Reflectance. It has got an Infrared Microscope with a lateral resolution of 10 µm.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- JASCO FT/IR-6200 IRT-5000
  JASCO FT/IR-6200 IRT-5000

Responsables Científicos/ Scientific Responsibles: Dr. Juan Carlos Sánchez López, Dr. Hernán Míguez García y Dr. Miguel Angel Centeno Gallego
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 equipos que cubren el rango de longitudes de onda que va desde 190 nm hasta 900 nm. Se puede trabajar tanto en el modo de Transmisión como en el de Reflectancia Difusa.

The Ultraviolet-Visible Spectroscopy (UV-Vis) reports on the existing energy differences between the more external occupied electronic levels and the nearer unoccupied ones.
There are 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
- Cary 5000 + UMA (Universal Measurement Accesory)
  Cary 5000 + UMA (Universal Measurement Accesory)
- Cary 300
  Cary 300

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

SERVICIO DE ANÁLISIS TEXTURAL Y TÉRMICO / TEXTURAL AND THERMAL ANALYSIS SERVICE

Este servicio incluye las siguientes Unidades: Análisis Térmico, Fisi-quimisorción, Análisis de Tamaño de Partícula y Potencial Z. Está dedicado a la determinación de la textura, estructura y comportamiento térmico de los materiales.

This Service includes the following units: Thermal Analysis, Physisorption and Chemisorption, Particle Size and Z-potential determination. It is devoted to the characterization of texture, microstructure and thermal behavior of advanced materials.

FISI-QUIMISORCIÓN / PHYSISORPTION-CHEMISORPTION

Este servicio constituye una herramienta básica para la caracterización microestructural de sólidos pulverulentos de distinta naturaleza, en cuanto a porosidad, superficie específica y superficie químicamente activa.

En el servicio se dispone de un analizador de adsorción de gases (Micromeritics, ASAP 2020) que proporciona isotermas de adsorción y desorción, a partir de los cuales se obtienen de ellas la superficie específica y distribución del tamaño de poro y de microporo de estos materiales, incorporando también los accesorios necesarios para medidas de quimisorción.

This service constitutes a basic tool for the microstructural characterization of powdered solids of different natures, regarding to their porosity, specific surface area and chemically active surface.

This service is composed by a phyisisorption analyser (Micromeritics, ASAP 2020) which provides the complete adsorption/desorption isotherms, from which the specific surface area, pore and micropore size distribution and concentration of reactive sites are obtained. The instrument is also equipped for carrying out chemisorption of different reactive molecules, as O_2, H_2, CO, etc.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- Analizador científico de fisisorción ASAP2010 (Micromeritics)
  Physisorption analyser ASAP 2010 (Micromeritics)
- Analizador de quimisorción ASAP2010 (Micromeritics)
  Chemisorption analyser ASAP 2010 (Micromeritics)
- Analizador de fisisorción multimuestra TRISTAR II (Micromeritics)
  Multisample physisorption analyser TRISTAR II (Micromeritics)
- Analizador de fisisorción multimuestra TRISTAR II-Kr (Micromeritics)
  Multisample physisorption analyser TRISTAR II-Kr (Micromeritics)

Responsables Científicos/ Scientific Responsibles: Dr. Gerado Colón Ibáñez y Dr. Miguel Angel Centeno Gallego
Personal Técnico/ Technical Assistant: Dª Cristina Gallardo López

ANÁLISIS TÉRMICO / THERMAL ANALYSIS

Las técnicas de análisis térmico permiten estudiar aquellos cambios físicos o químicos que ocurren en los sólidos en función de la temperatura y que conlleven modificaciones en su masa o intercambios de calor con su entorno.
En el servicio se pueden realizar experimentos desde temperatura ambiente hasta 1500ºC, tanto en atmósfera inerte (N_2) como reactiva (aire, O_2,…).
Se dispone de dos técnicas: Análisis Termogravimétrico (TG) y Análisis Térmico Diferencial (ATD).

Thermal analysis techniques allow to studying physical or chemical changes occurring in solid in samples as a function of the temperature. Those changes should involve either a mass change or a heat flow.
The experiments can be performed in the range from room temperature to 1500ºC, both under inert (N_2), or reactive (air, O_2,…) atmospheres.
Two different techniques are available: Thermogravimetry (TG) and Differential Thermal Analysis (DTA)

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- Equipo de análisis térmico simultáneo TG/ATD/CDB TA Instruments Q600
  TA Instruments Q600 Simultaneous TG/DTA/DSC instrument
- Equipo termogravimétrico TG, TA Instruments Q5000
  Thermogravimetric instrument TG, TA Instruments Q5000

Responsable Científico/ Scientific Responsible: Dr. Luis A. Pérez Maqueda
Personal Técnico/ Technical Assistant: Dª Cristina Gallardo López

TAMAÑO DE PARTÍCULAS Y POTENCIAL Z / PARTICLE SIZE AND Z POTENTIAL ANALYSIS

Se dispone de las técnicas de Dispersión Dinámica de Luz y de Difracción Láser (LD), que permiten determinar la distribución de tamaños de partícula de sistemas coloidales en suspensión (disolvente acuoso u orgánico) en los rangos que van de 3 a 3000 nanómetros (DLS) o de 0.05 a 900 micras (LD).

Así mismo, se dispone de la técnica de Análisis de Movilidad Electroforética para la evaluación del potencial "Z" de sistemas coloidales en suspensión (disolvente acuoso u orgánico).

Dynamic light scattering (DLS) and Laser diffraction (LD) are available for the determination of particle size distributions of colloidal systems (dispersed in aqueous or organic dispersions solutions) in the range 3-3000 nanometers (DLS) and 0.05-900 microns (LD).

Electrophoretic mobility measurements can be also performed for the evaluation of Z potential in colloidal systems (aqueous or organic dispersions).

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- Malvern modelo Zetamaster (DLS) y MalvernSizer (LD)
  Malvern model Zetamaster (DLS) and MalvernSizer (LD)

Responsable Científico/ Scientific Responsible: Dr. Manuel Ocaña Jurado
Personal Técnico/ Technical Assistant: Dª Cristina Gallardo López

SERVICIO DE MICROSCOPÍA ELECTRÓNICA / ELECTRON MICROSCOPY SERVICE

El servicio está dedicado a la caracterización química y estructural de muestras sólidas mediante técnicas de microscopía electrónica. Las técnicas de caracterización disponibles en el servicio son la Microscopía Electrónica de Transmisión (TEM) y la Microscopía Electrónica de Barrido (SEM), con el equipamiento anexo de preparación de muestras para TEM y SEM.

This Service is devoted to the chemical and structural characterization of solid samples by means of electron microscopies. The characterization techniques available at ICMS are Transmission

Electron Microscopy (TEM) and Scanning Electron Microscopy (SEM), with additional equipment for TEM and SEM sample preparation.

MICROSCOPÍA ELECTRÓNICA DE BARRIDO / SCANNING ELECTRON MICROSCOPY

La microscopía electrónica de barrido proporciona información microestructural, morfológica y de composición química en escala microscópica. Se puede aplicar a todo tipo de materiales y problemáticas de estudio en ciencia de materiales: cerámicas, plásticos, metales, minerales, catalizadores, muestras de patrimonio histórico, capas finas, recubrimientos, interfases, nanopartículas, etc. El equipo SEM es un microscopio de emisión de campo de cátodo frío que permite realizar imágenes de la morfología y textura superficial de las muestras con una resolución de 1 nm a 15kV. También permite trabajar a bajo voltaje en muestras sin metalizar y en modo transmisión (STEM-in-SEM) en muestras electrón-transparentes. Acoplado al detector de rayos-X (EDX) permite análisis elementales y mapas composicionales.

The scanning electron microscopy provides information about the microstructure, morphology and chemical composition at the microscopic scale of solid samples. It can be applied to all type of materials including ceramics, polymers, metals, minerals, catalysts, samples from cultural heritage, thin films, coatings, interfaces, nanoparticles, etc. The SEM microscope is a field emission cold cathode equipment which enables images of the surface morphology and texture of samples with a resolution of 1 nm at 15kV. It also allows working at low voltages with non-metalized samples and in transmission mode for electron-transparent samples (STEM-in-SEM). Coupled to the X-ray detector (EDX) enables compositional analysis and elemental mapping.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- Microscopio SEM, modelo Hitachi S4800 SEM-FEG: cañón de emisión de campo de cátodo frío y voltaje de 0.5-30 kV, resolución de 1 nm a 15kV. Dotado de analizador EDX Bruker-X Flash-4010 con una resolución de 133 eV (en la línea MnKα) y detector con portamuestras para trabajar en modo transmisión (STEM-in-SEM).
  Hitachi S4800 SEM-FEG microscope: cold cathode field emission gun with voltage from 0.5 to 30 kV, resolution of 1nm at 15 kV. Equipped with a Bruker-X Flash-4010 EDX detector with a resolution of 133 eV (at the MnKα line), and a detector with sample holder to work in transmission mode (STEM-in- SEM).
- Equipamiento adicional en el “laboratorio de preparación de muestras para microscopía electrónica” (ver sección abajo)
  Additional equipment in the “electron microscopy samples preparation laboratory” (see the section below)

Responsable Científico/ Scientific Responsible: Dra. Asunción Fernández Camacho
Personal Técnico/ Technical Assistant: Dra. M. Carmen Jiménez de Haro

MICROSCOPÍA ELECTRÓNICA DE TRANSMISIÓN / TRANSMISSION ELECTRON MICROSCOPY

La microscopía electrónica de transmisión es una técnica ampliamente utilizada para la caracterización estructural y química de materiales a escala microscópica y nanoscópica, proporcionando imágenes bidimensionales de la textura de la muestra, forma y tamaño de grano y/o de partícula, grado de homogeneidad a escala microscópica, grado de cristalinidad de la muestra, identificación de fases cristalinas, e imágenes de alta resolución que identifican dominios cristalinos. El equipo está dotado de un analizador EDX para el análisis composicional. Puede aplicarse a todo tipo de materiales y campos de estudio en ciencia y tecnología de materiales trabajando sobre muestras electrón-transparentes preparadas en su caso ad-hoc para este fin. El servicio realiza microscopía en modo transmisión: Imágenes en campo claro y campo oscuro, difracción de electrones de área selecta y microscopía electrónica de alta resolución así como análisis elemental de area selecta. No se dispone de modo STEM.

The transmission electron microscopy is a widely used technique for the microstructural and chemical characterization at micro and nanoscales, providing two-dimensional images of the sample texture and shape as well as grain and/or particle size, degree of homogeneity at the microscopic scale, degree of crystallinity of the sample, identification of crystalline phases, and high resolution images to identify the crystalline domains. The microscope is equipped with an EDX analyzer for compositional analysis. It can be applied to all type of materials and research topics in materials science and technology working with electron-transparent samples prepared ad-hoc for this end. The service performs transmission electron microscopy: Imaging in bright and dark field, selected area electron diffraction and high resolution electron microscopy, as well as elemental analysis of selected areas. It does not provide STEM mode.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- Microscopio Philips CM20 (200kV) con una resolución estructural de 0.14 nm entre líneas y 0.23 nm entre puntos, portamuestras de uno y dos giros y de calentamiento. Acoplados al equipo se dispone de un sistema de análisis por Energías Dispersivas de Rayos X (EDX X-Max 80T, Oxford Instruments) y una cámara CCD (Gatan) para registro de imágenes.
  Philips CM200 microscope (200kV) with a structural resolution of 0.14 nm between lines and 0.23 nm between points. Sample holders with one and two angles and heating. It is equipped with a X-ray Energy Dispersive Analyzer (EDX X-Max 80T, Oxford Instruments) and a CCD GATAN camera for image acquisitions.
- Equipamiento adicional en el “laboratorio de preparación de muestras para microscopía electrónica” (ver sección abajo)
  Additional equipment in the “electron microscopy samples preparation laboratory” (see the section below)

Responsable Científico/ Scientific Responsible: Dra. Asunción Fernández Camacho
Personal Técnico/ Technical Assistant: Dª Olga Montes Amorín (CicCartuja) y Dª María Inmaculada Roja Cejudo

LABORATORIO DE PREPARACIÓN DE MUESTRAS PARA MICROSCOPÍA ELECTRÓNICA / ELECTRON MICROSCOPY SAMPLES PREPARATION LABORATORY

El laboratorio de preparación de muestras para TEM y SEM dispone de metalizador de oro, evaporador de carbón, metalizador de Cr y carbón, cortadora de disco, pulidora, "disc-grinder", cortadora ultrasónica, pulidora cóncava (dimple) y adelgazador iónico (Fischione 1010).

The laboratory for TEM and SEM samples preparation has a gold coater, a carbon evaporator, a metallization system for Cr and carbon, a diamond wheel cutter, a grinder with disc-grinder device, an ultrasonic cutter, a concave polishing (dimple) and ion thinning (Fischione 1010).

Responsable Científico/ Scientific Responsible: Dra. Asunción Fernández Camacho
Personal Técnico/ Technical Assistants: Dª María Inmaculada Roja Cejudo, Dª Olga Montes Amorín (CicCartuja) y Dra. M. Carmen Jiménez de Haro

SERVICIO DE DIFRACCIÓN DE RAYOS X
X-RAY DIFFRACTION LABORATORY SERVICE

La difracción de rayos-X permite la identificación cualitativa y cuantitativa de sustancias cristalinas y su caracterización microestructural y textural.
El servicio dispone en la actualidad de cuatro difractómetros independientes, configurados específicamente para abordar el análisis de muestras policristalinas de muy distinta naturaleza, en lo referente a su composición, estabilidad química, cristalinidad, etc.
Asimismo, con alguno de ellos se pueden llevar a cabo, además de los análisis rutinarios (θ-2θ), otros varios mas avanzados, como pueden ser:

- Seguir las transformaciones de fase "in situ" provocadas por calentamientos en atmósfera inerte (vacio, Ar) o reactiva (H2, O2,..).
- Caracterizar materiales en la nanoescala (1-100 nm) mediante el estudio de la dispersión de rayos-X a ángulos bajos (SAXS).
- Determinar el grosor, densidad y rugosidad de películas delgadas, mediante Reflectometría de rayos-X.
- Obtener la estructura cristalina de materiales inestables a la atmósfera o muy transparentes a los rayos-X, mediante el empleo de capilares.

X-ray diffraction allows the qualitative and quantitative identification of crystalline substances and their microstructural and textural characterization.
At present, four independent diffractometers are available in this service, specifically configured to analyze the composition, chemical stability, crystallinity and many other properties in polycrystalline samples of a varied nature. Besides ordinary analyses (θ-2θ), part of the equipment can perform some advanced studies as:

- Direct monitoring of transformations undergone in materials under heating, such as phase changes, under inert o reactive atmosphere.
- To characterize materials at the nanoscale (1-100 nm) through X-ray scattering at low angles, using the SAXS technique.
- To measure some physical parameters of layers such as density, thickness and surface roughness with the reflectometry setup.
- To obtain the diffraction patterns of samples either sensitive to the atmosphere or highly transparent to X-rays (organic compounds) employing the capillary configuration.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- Difractómetro Panalytical X'PERT PRO con cargador automático de muestras
  Diffractometer PANALYTICAL X'PERT PRO with automatic sample charger
- Difractómetro PHILIPS X'PERT PRO con cámara de alta temperatura (1200 ºC) ANTON PAAR HTK 1200
  Diffractometer PHILIPS X'PERT PRO with high temperature chamber (1200ºC) ANTON PAAR HTK 1200
- Difractómetro Panalytical X'PERT PRO (reflectometría, SAXS, ángulo rasante y capilares)
  Diffractometer PANALYTICAL X'PERT PRO (reflectometry, SAXS, low angle scattering and capillary)
- Difractómetro de polvo SIEMENS D5000 DUAL (reflexión y transmisión)
  Diffractometer SIEMENS D5000 DUAL (reflection and transmission)

Responsable Científico/ Scientific Responsible: Dra. Concepción Real Pérez
Personal Técnico/Technical Assistant: D. José María Martínez Blanes

SERVICIO DE ANÁLISIS DE SUPERFICIE/ SURFACE ANALYSIS SERVICE

El Servicio de Análisis de superficie consta de un espectrómetro de Espectroscopía de Fotoelectrones de Rayos X (XPS). Este servicio está dedicado al análisis químico y electrónico de superficies sólidas. También permiten conocer la composición en profundidad (desde la superficie hacia el interior) de los sólidos.

The surface analysis service consists of an X-ray Photoelectron Spectrometer (XPS). This service is devoted to the electronic and chemical analysis of solid surfaces. It also provides information about the compositional depth profile of solids (from their surface toward their bulk).

ESPECTROSCOPÍA DE FOTOEMISIÓN DE ELECTRONES / X-RAY PHOTOELECTRON SPECTROSCOPY

Las "Espectroscopías de Fotoelectrones" (XPS/ESCA y AES) son unas poderosas técnicas de análisis cuantitativo no destructivo, sensibles exclusivamente a las primeras capas de la superficie de los sólidos (20-30 Å), lo que permite obtener información sobre las propiedades químicas, físicas y electrónicas de las mismas.

El interés técnico de esta información es enorme en campos tales como corrosión, catálisis, tratamientos de superficies, fenómenos de flotación y adherencia, segregación de fases, etc.

La característica más importante de la Espectroscopía de Fotoelectrones (XPS/ESCA) es que permitir diferenciar distintos estados de oxidación y/o situaciones del entorno (coordinación) de los átomos en las muestras sólidas analizadas. El límite de detección es del 0.5% para cada especie química.

El servicio dispone actualmente de dos instrumentos independientes.

Typically, “photoelectron spectroscopies” are a powerful set of non-destructive analysis techniques, exclusively sensitive to the more superficial few atomic layers (20-30 Å), allowing to obtain valuable information about their chemical, physical and electronic properties.

The technical interest of the resulting information is huge in fields such as catalysis, corrosion, surface treatments,, floating and adhesion phenomena, or segregation processes, among others. The most remarkable characteristic of X-Ray Photoelectron Spectroscopy (XPS/ESCA) is that it allows to discriminate, for a given element, between different oxidation states or chemical surroundings (coordination).

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

Espectrómetro de Fotoelectrones VG ESCALAB 210, compuesto de:
Photoelectron Spectrometer VG ESCALAB 210, consisting on:

- Cámara de análisis, analizador hemiesférico multicanal VG ESCALAB 210, manipulador de cuatro ejes, y fuentes de excitación de rayos X (dual, AlKα y MgKα, acromático), de luz ultravioleta y de haces de electrones, lo que permite realizar análisis superficiales mediante técnicas de XPS, UPS y REELS, así como estudios angulares.
  Analysis Chamber, equipped with a hemispheric multichannel analyser VG ESCALAB 210, a four axis manipulator, a dual X-ray source (achromatic AlKα, Mg Kα), a UV lamp, and a electron gun, allowing to perform surface analysis by XPS, UPS and REELS, including angular resolved studies.
- Dos Precámaras de tratamientos, con vacío residual de 10^{-8} y 10^{-9} mbar respectivamente, en las que es posible someter a las muestras a tratamientos diversos como: calentamientos a alta temperatura (T<800ºC) bajo atmósfera controlada, desbastado iónico con gases inertes o reactivos, exposición a plasmas, iluminación con laser, deposición de metales, óxidos y compuestos sencillos, exfoliación in situ, etc.
  Two prechambers for different treatments, with ultimate vacuum levels of 10-8 and 10-9 mbar respectively, where samples can be subjected to diverse treatments and transferred to the analysis chamber without exposure to the atmosphere. The possible treatments include heating at high temperature (< 800C) under controlled atmosphere, ion

sputtering with inert or reactive gases, exposure to plasma, laser treatments, deposition of metals, oxides or simple compounds, exfoliation, etc.

Espectrómetro de Fotoelectrones SPECS, compuesto de:
Photoelectron Spectrometer SPECS, consisting on:

- Cámara de análisis, dotada de analizador hemiesférico multicanal PHOIBOS 100, manipulador de tres ejes, y fuentes de excitación de rayos X (dual, Alkα y MgKα).
  Analysis Chamber, equipped with a hemispheric multichannel analyser PHOIBOS 100, three axis manipulator and dual X-ray source (achromatic Al Kα, Mg Kα).
- Precámara de tratamiento de alta Presión y alta Temperatura (HPHT Cell). En esta Cámara es posible someter a las muestras a tratamientos térmicos en presencia de gases hasta una presión de 20 atm y 800 ºC, tanto en estático como en dinámico (simultáneamente).
  Pre-chamber for High Pressure/High Temperature treatments (HPHT Cell).Samples can be subjected to treatments in the presence of gases up to 20 bar and 800ºC (simultaneously). These treatments can be performed either under static or flowing gas conditions. After treatments, samples can be transferred to the analysis chamber without exposure to the atmosphere.
- Una cámara de inserción rápida dotada de sistema de aparcamiento/ desgasificado, que permite evacuar las muestras a temperatura reducida (T < 150ºC). También es posible la realización de tratamientos de desbatado iónico o la incorporación de otros sistemas (iluminación con luz Uv-Vis, evaporación de metales, u otros compuestos, etc.)
  A Fast entry chamber, equipped with a parking and degassing system, allowing the samples to be evacuated at moderate temperature (T < 150ºC). It is also possible to sputter the samples under an accelerated ion beam (0.5- 5.0 kV) using inert or reactive gases. Incorporation of some other equipment (Visible light illumination, metal evaporators) is also contemplated.

Responsables Científicos/ Scientific Responsibles: Dr. Juan Pedro Espinós Manzorro y Dr. Juan Pedro Holgado Vázquez

SERVICIO DE MECANIZADO/ MECHANIZED WORKSHOP

Se trata de un servicio horizontal fundamental para el Instituto y unidades externas adscritas al mismo. Ya que permite mejorar, modificar y adecuar el material y equipamiento científico a las necesidades de cada investigador y/o investigación en curso. Incluso llegando a su fabricación partiendo de una necesidad concreta. Ofreciendo asesoramiento técnico, diseño y fabricación de todos los elementos anteriormente descritos.
Además brinda la posibilidad de realizar pequeñas reparaciones y parte del mantenimiento general del equipamiento científico y de laboratorio.

This is a service essential for the Institute and external drives attached to the same. Because it allows you to improve, modify and adapt the material and scientific equipment to the needs of each researcher and/or research in progress. Even going to the extent of their manufacture on the basis of a specific need. Offering technical advice, design and manufacture of all elements described above.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

El servicio cuenta con herramientas manuales y herramientas eléctricas para la conformación de materiales muy diversos. Contando con la posibilidad unión de diversos materiales mediante los procesos de soldeo que se pueden realizar en el servicio:

The service account with hand tools and power tools for the formation of very diverse materials. With the possibility union of various materials by welding processes that can be performed in the service:

- Soldadura fuerte con diferentes aportes
  Brazing with different contributions
- Soldadura por arco eléctrico
  Electric arc welding
- Soldadura TIG sobre aceros
  TIG welding on steel

Para los procesos de mecanizado por arranque de viruta se cuenta con las siguientes máquinas-herramientas:

For the processes of machining by chip is has the following machine-tools:

- Centro de mecanizado,HAAS TM 1P
  HAAS machining center,TM 1P
- Taladro vertical, ERLO TSAR32
  Vertical drill, ERLO TSAR32
- Torno paralelo convencional PINACHO SC200
  Conventional lathe PINACHO SC200
- Torno paralelo semiautomático PINACHO SMART TURN180
  Semi-automatic lathePINACHO SMART TURN180

Responsable/ Responsible: Dra. Ana Isabel Becerro Nieto
Personal Técnico: D. Juan Carlos Sánchez Martín y D. Manuel Perea Domínguez

ACTIVIDADES DIVULGATIVAS Y FORMATIVAS

OUTREACH AND TEACHING ACTIVITIES

DOCENCIA / TEACHING

MÁSTER / MASTER

MÁSTER EN CIENCIA Y TECNOLOGÍA DE NUEVOS MATERIALES

Organizado por la Universidad de Sevilla
Coordinador: Dr. Joaquín Ramírez Rico
Fecha de Celebración: Curso Académico 2016-17
Créditos necesarios: 60
Dirigido a: Licenciados en Química, Física, así como, los Titulados en Ingeniería afines (Ingenieros Químicos, de Materiales, etc.)
Especialidades: Materiales para la Energía y el Medio Ambiente. Ingeniería de Superficies, Materiales Estructurales y Funcionales.
Mención de Calidad

Química del Estado Sólido (Créditos: 5) | Dra. Pilar Malet Maenner
Fisica del Estado Sólido (Créditos: 5) | Dra. Angela Gallardo López | Dra. Josefa Borrego | Dr. Manuel Jiménez Melendo
Técnicas de Caracterización de Materiales (Créditos: 8) | Dr. Julián Martínez Fernández | Dr. Juan M. Montes Martos (US)
Síntesis de Materiales y Nanoestructuras (Créditos: 7) | Dra. María Dolores Alcalá | Dra. Svetlana Ivanova | Dr. Francisco J. Gotor Martínez
Catalizadores para la Energía y el Medio Ambiente (Créditos: 5) | Dr. José Antonio Odriozola Gordón
Materiales con Funcionalidad Superficial (Créditos: 5) | Dra. Rosa María Pereñiguez Rodríguez | Dra. Leidy Marcela Martínez Tejada
Recuperación y Transformación de Materiales (Créditos: 5) | Dra. Svetlana Ivanova | Dr. Leidy Marcela Martínez Tejada
Corrosión y Recubrimientos Protectores (Créditos: X) | Dr. Leidy Marcela Martínez Tejada
Procesado de Materiales Estructurales (Créditos: 5) | Dr. Alfonso Bravo León | Dr. José Antonio Rodríguez Ortíz
Comportamiento Mecánico (Créditos: 5) | Dr. Arturo Domínguez Rodríguez | Dr. Diego Gómez García
Comportamiento Magnético (Créditos: 5) Dr. Javier S. Blázquez Gámez | Dr. Victorino Franco García
Comportamiento Térmico, Dieléctrico y Óptico de Materiales (Créditos: 5) | Dr. Hernán Míguez García | Dr. Francisco Javier Romero Landa | Dra. Carmen Gallardo Cruz
Computación en Ciencia de Materiales (Créditos: 5) | Dra. Regla Ayala Espinar | Dr. Antonio Córdoba Zurita (US) | Dr. Felipe Gutiérrez Mora

Asimismo, el personal del ICMS ha impartido cursos regulares de primer y segundo ciclos de las correspondientes Licenciaturas y/o Grados en Química, Física y Farmacia e Ingeniería de Materiales y Biología en la Universidad de Sevilla.

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

8 de agosto | **Applications of ordered and disordered optical materials in optoelectronics**
Hernán Míguez
Lugar: Departmental Seminar. Nonlinear Physics Cente. Research School of Physics & Engineering (Australia)

15 de agosto | **Multifunctional Photonic Materials for Energy, Sensing and Biomedical Applications**
Hernán Míguez
Lugar: CBMS Seminar. Macquarie University, Department of Molecular Sciences (Australia)

15 de noviembre | **Synthesis and applications of 1D and 3D supported nanostructures developed by plasma assisted vacuum deposition methods**
Ana Borrás
Lugar: MRI-Seminar Queen Mary University of London. School of Engineering and Materials Science (Reino Unido)

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

16 de febrero | **The Versatility of Mesoscopic Solar Cells**
Prof. Dr. Anders Hagfeld
Swiss Federal Institute of Technology Lausanne (EPFL), CH-1015, Lausanne, Switzerland

In our work on solid-state dye-sensitized solar cells (ssDSSC) we have recently [1] shown that copper phenanthroline complexes can act as an efficient hole transporting material. We prepared ssDSCs with the organic dye LEG4 and copper(I/II)-phenantroline as redox system and achieved power conversion efficiencies of more than 11%. Our follow up work on electron transfer studies and device optimization will be presented at the meeting.

In our work on perovskite solar cells (PSC) we have achieved efficiencies above 20% with a mixed composition of iodide/bromide and methyl ammonium/formamidinium [2]. For cells larger than 1 cm2 we recently certified a record efficiency of 19.6% [3], replacing the anti-solvent step in the perovskite film formation with a vacuum flash treatment. With the use of SnO2 compact underlayers as electron acceptor contacts we have constructed planar perovskite solar cells with a hysteresis free efficiency above 20% [4]. Recently, we have taken the cation mixing of the perovskite film further by including the Cs+ in a so-called 'triple cation' composition, i.e. Cs/FA/Ma. Larger grains grown in a monolithic manner are observed and for example reproducibility and device stability are improved [5]. At the meeting we will discuss our follow up works [6] and present our champion data; up to 22% efficiency with an external electroluminescence of 4%, and an outstanding open-circuit voltage of 1.24 V at a band gap of 1.63 eV entailing one of the smallest loss-in-potential of 0.39 V ever measured for any solar cell material. Furthermore, we will report a breakthrough in stability at 85 oC for 500 h under full solar illumination and maximum power point tracking (during which 95% of the initial performance was retained).

References

[1] Freitag et al., Energy & Envir. Sci., DOI: 10.1039/C5EE1204J
[2] Bi et al., Science Advance, DOI: 10.1126/sciadv.1501170
[3] X. Li et al., Science, DOI:10.1126/science.aaf8060
[4] Correa et al., Energy & Envir. Sci., DOI:10.1039/C5EE02608C
[5] M. Saliba et al., Energy & Envir. Sci., 2016, DOI: 10.1039/C5EE03874J
[6] M. Saliba et al., Science 10.1126/science.aah5557 (2016)

16 de marzo | **Materials for catalysis: challenges and opportunities**
Prof. Dr. Cuong Pham-Huu
Institute of Chemistry and Processes for Energy, Environment and Health (ICPEES, UMR 7515) CNRS and University of Strasbourg, France

New catalytic materials have been extensively developed since the last decades for application in several research fields including sensors, drug delivery, light-weight high mechanical strength composites and catalysis. In this presentation, we will discuss about last developments on the use of (i) confinement effect in 1D carbon channel, (ii) nanostructuration of 2D carbon via catalytic patterning along with the uncovering of the patterning mechanism by operand transmission electron microscopy, and (iii) development of new bio-sourced mesoporous carbon doped with nitrogen as an active and selective metal-free catalyst for oxidation process. The first example reports on the use of confinement effect to modify and selectively cast, metal oxides, inside 1D carbon material for applications in the field of drug delivery, biological imaging and catalysis. In the second example, catalysis has been used to perform nanopatterning of few-layer graphene, leading to the generation of higher reactive edge sites, for the subsequence anchorage of metal oxide nanoparticles with improved sintering resistance for application in the field of sensor and liquid-phase reactions. The direct analysis of the process by operando TEM at ambient conditions allows one to uncover the different mechanisms operated during the patterning process for future optimization step. The last example focus on the synthesis of nitrogen-doped mesoporous carbon, issued from bio-sourced raw materials, decorated silicon carbide as metal-free catalyst for the selective oxidation of trace amount of H2S into elemental sulfur with improved activity and stability. The presentation will end-up with some future perspectives about the use of carbon-based metal-free catalysts in some relevant catalytic processes as well as the role of operando TEM investigation for unraveling catalytic mechanisms.

20 de abril | **Plasma-synergistic effects: catalysing cross-disciplinary collaborations**
Prof. Dr. Kostya (Ken) Ostrikov
Queensland University of Technology (QUT) and CSIRO, QUT-CSIRO Joint Sustainable Processes and Devices Laboratory. Australia

This presentation will introduce the key features of low-temperature plasmas that make them a versatile tool in materials science and engineering and other areas such as chemical engineering and health sciences. Particular attention will be paid on synergistic effects of plasmas with common materials and processing methods and what difference it makes in diverse applications, with particular focus where nanoscale features of materials play a role. These localized interactions have opened opportunities for fundamental research and applications in the plasma nanoscience field.

The focused “what can plasma do for you” examples will be used to stimulate collaborative efforts even between researchers normally working in completely disparate fields.

18 de mayo | **Luminescent assemblies, imaging and cellular lasing**
Prof. Dra. Luisa De Cola
Institute de Science et d'Ingénierie Supramoléculaires (I.S.I.S.), Université de Strasbourg and KIT, Germany

Luminescent molecules that can undergo self-assembly are of great interest for the development of new materials, sensors, biolabels…. The talk will illustrate some of the recent results on soft structures based on metal complexes able to aggregate in fibers, gels and soft mechanochromic materials [1]. The use of platinum complexes as building block for luminescent reversible piezochromic and mechanochromic materials, down to the nanoscale, will be illustrated [2]. The emission of the compounds can be tuned by an appropriate choice of the coordinated ligands as well as of their aggregation in different structures. The formation of soft assemblies allows the tuning of the emission color, by pressure and temperature leading to a new class of materials possessing reversible properties. We demonstrate how even small changes in molecular design can completely inhibit or enhance the formation of organized supramolecular architectures, leading to a deep understanding of the key factor affecting the whole self-assembly process.
The monitoring of the different emission properties, used as fingerprint for each of the assembled species, allowed an unprecedented real-time visualization of the evolving self-assemblies [3].
In the final part of the talk a new concept, mirrorless cellular lasing, i.e. to generation of strong laser action from stained biological cells and, importantly, in absence of any external resonators is described. Interestingly, such phenomenon is connected to the biological components that constitute the structure of the cells involved in laser generation [4].

References

[1] C. A. Strassert, L. De Cola et al. Angew. Chem. Int. Ed., 2011, 50, 946; M. Mauro, L. De Cola et al. Chem. Commun. 2014, 50, 7269
[2] D. Genovese, L. De Cola et al. Adv. Funct. Mater., 2016, 26, 5271–5278
[3] A. Aliprandi, M. Mauro, L. De Cola Nature Chemistry , 2016, 8, 10-15
[4] D. Genovese, V. Barna, L. De Cola submitted

16 de junio| **The influence of non-stoichiometry and chemical doping on the electrical properties of Na1/2Bi1/2TiO3 ceramics**
Prof. Dr. Derek C. Sinclair
Departamento de Ciencia de Materiales e Ingeniería University of Sheffield, UK

ABO3-type perovskite oxides exhibit a diverse range of useful functional properties from ferroelectric BaTiO3 materials for dielectric applications in multilayer ceramic capacitors to solid electrolytes and mixed oxide-ion, electronic conducting electrodes for solid oxide fuel cells. The ferroelectric perovskite Na1/2Bi1/2TiO3 (NBT) is known to exhibit interesting and diverse structure-composition-property relationships. A combination of A-site (Na, Bi) disorder, Bi- and Ti-displacements and in-phase and out-of-phase octahedral tilting ensures the crystal chemistry and polymorphism of NBT remain a challenging subject, especially below ca. 520 oC where distortions from the ideal 'cubic' cell are reported to occur. Furthermore, the electrical conductivity of undoped NBT materials are known to be critically dependent on low levels of A-site non-stoichiometry.

Here we review the structure-composition-property relationships of NBT materials based on a combination of A-site non-stoichiometry and chemical doping. We use results to show nominally Na-rich or Bi-deficient NBT ceramics to be excellent oxide-ion conductors whereas nominally Na-deficient or Bi-excess NBT ceramics are electrically insulating but retain. This shows undoped NBT ceramics to be mixed ion-electron conducting materials.

Finally, we discuss the differences in the defect chemistry of NBT compared to conventional titanate perovskites, such as (Ba,Sr)TiO3.

[1] E. Aksel, J.S. Forrester, J.L. Jones, P.A. Thomas, K. Page, and M.R. Suchomel, Appl. Phys. Lett., 98, 152901 (2011)
[2] I. Levin and I.M. Reaney, Adv. Funct. Mater., 22, 3445–3452 (2012)
[3] Y. Hiruma, H. Nagata, and T. Takenaka, J. Appl. Phys., 105, 084112 (2009)
[4] M. Li, M.J. Pietrowski, R.A. De Souza, H. Zhang, I.M. Reaney, S.N. Cook, J.A. Kilner, and D.C. Sinclair, Nat. Mater., 13, 31–35 (2014)
[5] M. Li, H. Zhang, S.N. Cook, L. Li, J.A. Kilner, I.M. Reaney, and D.C. Sinclair, Chem. Mater., 27, 629–634 (2015)
[6] M. Li, L. Li, J. Zang, and D.C. Sinclair, Appl. Phys. Lett., 106, 102904 (2015)

15 de septiembre | **Tailoring crystallization in oxide glasses: Application to transparent polycrystalline ceramics and nanostructured glass-ceramics**

Prof. Dr. Prof. Mathieu Allix
Conditions Extrêmes et Matériaux: Haute Température et Irradiation (CEMHTI); CNRS - Centre national de la recherche scientifique, Orléans (France)

Crystallization from glass can be a powerful process to elaborate innovating transparent materials for optical and photonic applications if nucleation and crystal growth steps can be precisely controlled. This talk will focus on two main applications: transparent polycrystalline ceramics elaborated by full and congruent crystallization from glass and nanostructured glass-ceramics designed from nanoscale phase separated glasses.

Transparent polycrystalline ceramics elaborated by full crystallization from glass

Transparent ceramics are an emerging class of optical materials competing with single crystal technology for a broad range of applications. Ceramics offer several advantages, particularly in the fabrication of complex shapes and large-scale industrial production, and enable great and homogenous doping of optically active ions. However, up to date, only a limited number of cubic or nanocrystalline transparent polycrystalline ceramics requiring complex and expensive synthetic approaches has been reported. Our recent work shows the possibility to obtain new transparent ceramics by full and congruent crystallization from glass. This is demonstrated in the case of several new compositions, such as BaAl4O7, Sr3Al2O6 and Sr3Ga2O6, all showing high transparency in the visible and infra-red ranges. Lately, we have focused our work on large scale and highly transparent strontium aluminosilicate compositions. A crystallographic study coupled to NMR experiments and DFT calculations of the birefringence evidences the role of structural disorder (Al/Si substitution and presence of vacancies on strontium sites) to explain the optical isotropy observed in these hexagonal materials. These results propose an innovative concept, the addition of a controlled structural disorder within crystalline structures, in order to lower the birefringence and to elaborate new transparent ceramics.

Transparent nanostructured glass and glass-ceramics

New nanostructured gallogermanate- and gallosilicate-based glass materials exhibiting high transparency in the visible range have been fabricated by conventional melt-quenching. These materials can accommodate wide oxide compositions and present nanoscale phase separation. The size of the nanostructuring can be tailored depending on the nominal composition. A single heat treatment then allows selective crystallization of the phase separated glass, resulting in glass-ceramic materials exhibiting nanostructures and transparency similar to the parent glass [8, 9]. The wide possibilities of designing new nanostructured glass-ceramics with tunable optical properties will be illustrated in the case of a highly transparent ZnGa2O4 glass-ceramic exhibiting 50 wt% of nanocrystals with homogeneous and tunable sizes. High resolution scanning transmission electron microscopy analysis coupled with in situ high temperature X-ray diffraction and optical measurements led to a detailed description of the crystallization process. Remarkably, red long-lasting luminescence arising from the entire sample volume is observed in this Cr3+ doped material, opening the route to a wider range of performing applications for this famous zinc gallate persistent phosphor [10, 11].

1. M.Allix et al., Advanced Materials, 24 5570-5575 (2012)
2. International patent deposited 1/12/2011, published 6/6/2013
3. G.Patton et al., PCCP, 16 24824 (2014)
4. M.Boyer et al., J. Mater. Chem. C, 4 3238-3247 (2016)
5. S.Alahraché et al, Chemistry of Materials, 25 4017-4024 (2013)
6. M.Boyer et al. Cryst. Growth Des., 16 386-395 (2016)
7. K.Al Saghir et al., Chemistry of Materials, 27 508-514 (2015)
8. International patent deposited 28/02/2014, published 4/9/2014
9. S.Chenu et al. Advanced Optical Materials, 2 364 (2014)
10. S. Chenu et al. J. Mater. Chem. C, 2 10002-10010 (2014)
11. M.Allix et al. Chemistry of Materials, 25 1600–1606 (2013)

OTRAS ACTIVIDADES / OTHER ACTIVITIES

FERIA DE LA CIENCIA / FAIR OF SCIENCE

La 15ª Feria de la Ciencia (del 11 al 13 de mayo de 2017, Palacio de Congreso y Exposiciones de Sevilla, FIBES) constituye un punto de encuentro donde se desarrollaron actividades de divulgación de la Ciencia y la Tecnología, realizando demostraciones y experimentos para facilitar la comprensión de contenidos científicos. El Instituto de Ciencia de Materiales de Sevilla presentó la actividad: "Catálisis para un mundo mejor y más sostenible".
Coordinadora: Dra. T. Cristina Rojas Ruiz
Unidad "Catálisis para el medioambiente y la energía"
Investigador Responsable: Dr. Juan Pedro Holgado
Participaron un total de 24 monitores

The Fair of Science (11 to 13 May 2017, 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: "Catálisis para un mundo mejor y más sostenible"
Coordinator: Dra. T. Cristina Rojas Ruiz
Research Unit: Catalysis and Environmental Energy
Scientific Responsible: Dr. Juan Pedro Holgado
Participants: 24 monitors

SEMANA DE LA CIENCIA Y LA TECNOLOGÍA / SCIENCE AND TECHNOLOGY WEEK

Jornadas de puertas abiertas. La Semana de la Ciencia y la Tecnología está dirigida a todos los públicos. Este evento de carácter europeo está diseñado para demostrar cómo la ciencia y la tecnología nos afectan y cómo éstas pueden ser utilizadas para mejorar nuestras vidas y el mundo que nos rodea.

Coordinación: Dra. T. Cristina Rojas Ruiz

Café con Ciencia

El café con ciencia es una actividad que acerca de forma original y atractiva la ciencia y sus protagonistas. Esta actividad de divulgación genera un punto de contacto entre profesionales de la ciencia y alumnos de secundaria, promoviendo la reflexión sobre diversos asuntos en un entorno cercano y participativo.

En esta edición se compartirán desayuno con estudiantes de bachillerato para dialogar sobre temas concretos de sus estudios y sobre sus respectivas trayectorias profesionales. Esta actividad cumple el triple objetivo de comunicar la ciencia a través de sus propios protagonistas, promover la cultura científica y fomentar vocaciones investigadoras.

Las mesas de encuentro tienen una hora de duración, y se desarrollan con grupos reducidos de quince alumnos.

Mesa. Dr. Gabriel Lozano Barbero. Tema: “¿Puede una nanoestructura iluminar tu salón”

Conferencia

Dr. Hernán Míguez García. Tema: “Nanomateriales ópticos para dispositivos optoelectrónicos” Facultad de Física de la Universidad de Sevilla

PARTICIPACION EN LA NOCHE EUROPEA / European Researchers' Night

Dos Investigadores del Instituto han participado en la European Researchers' Night, una iniciativa promovida por la Comisión Europea en el marco del programa Horizonte 2020. El evento se celebró el 29 de septiembre de forma simultánea en trescientos cincuenta ciudades europeas.

En Sevilla han unido esfuerzos el Consejo Superior de Investigaciones Científicas (CSIC), la Universidad de Sevilla (US), la Universidad Pablo de Olavide (UPO) y el Ayuntamiento de Sevilla para ofrecer un programa conjunto compuesto por talleres, microencuentros y espectáculos diseñados para buscar un acercamiento distendido entre la ciudadanía y la comunidad científica, mediante la diversión y el lenguaje divulgativo.

Este singular formato ofrece una oportunidad a los ciudadanos de acercarse a los investigadores y vencer algunos tópicos sobre la profesión científica. Y a los investigadores ofrece una oportunidad de salir de sus laboratorios y demostrar que no viven ajenos a la realidad exterior.

LA NOCHE EUROPEA
DE L@S INVESTIGADOR@S
MUJERES Y HOMBRES QUE HACEN CIENCIA PARA TI

Dr. **Mauricio Calvo Roggiani**. Cristales Fotónicos, materiales nanotecnológicos para el control de la luz

Dr. **Juan Ramón Sánchez Valencia.** Nanomateriales, mucho más que miniaturación.

Visitas Guiadas / Guided Visits

Estudiantes universitarios de Chemical Engineering at Eindhoven. University of Technology (The Netherlands) han visitado el ICMS el día 27 de enero de 2017.

Alumnos de 4º de la ESO del Colegio BVM Irlandesas Ntra. Sra. De Loreto han visitado las instalaciones del ICMS el día 22 de marzo de 2017 organizada por l a Dra. Ana Isabel Becerro Nieto.

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