ICMS

Instituto de Ciencia de Materiales de Sevilla

Memoria de Actividades
Annual Report

Consejo
Superior de
Investigaciones
Científicas

Universidad
de Sevilla

Junta de
Andalucía

2015

Consejo Superior de Investigaciones Científicas
Universidad de Sevilla
Junta de Andalucía

INSTITUTO DE CIENCIA DE MATERIALES DE SEVILLA

Memoria de Actividades
Annual Report
2015

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

Edita

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

Diseño y Maquetación

José Carlos Rivero Cabello

Comisión Memoria-ICMS

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

INDICE

TABLE OF CONTENTS

El ICMS en 2015
ICMS in 2015

Presentación Presentation

A través de esta Memoria 2015, el Instituto de Ciencia de Materiales de Sevilla (ICMS) pretende comunicar lo mejor de las actividades científicas realizadas a lo largo del periodo.

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

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

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

Through this Activity Report 2015, the Institute of Materials Science of Seville (ICMS) is trying to communicate the best of its scientific activities for the period.

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

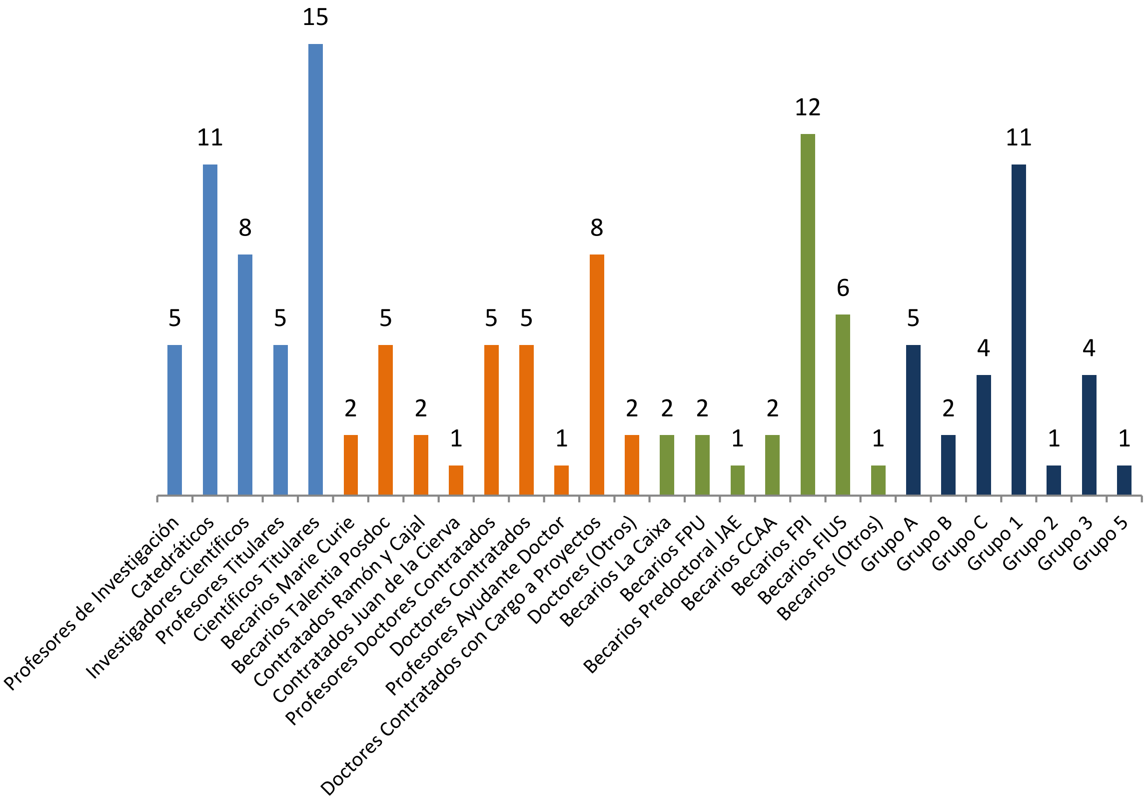
The current Strategic Plan 2014-2017 organizes our Center in 5 lines of research, which related with disciplines of solid state chemistry and physic, as well as materials engineering subjects, are intended to cover the most modern and innovative aspects of the current material science. Our basic commitment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

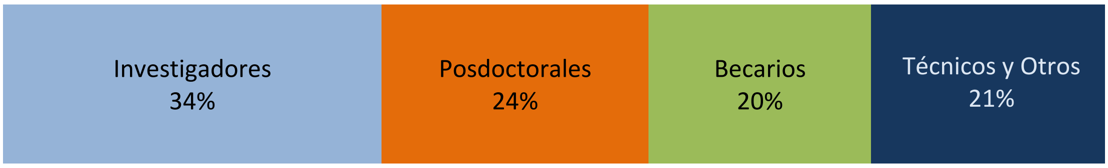
Dr. **Alfonso Caballero Martínez**
Director del Instituto de Ciencia de Materiales de Sevilla

Datos Estadísticos del ICMS
Statistical Data of ICMS

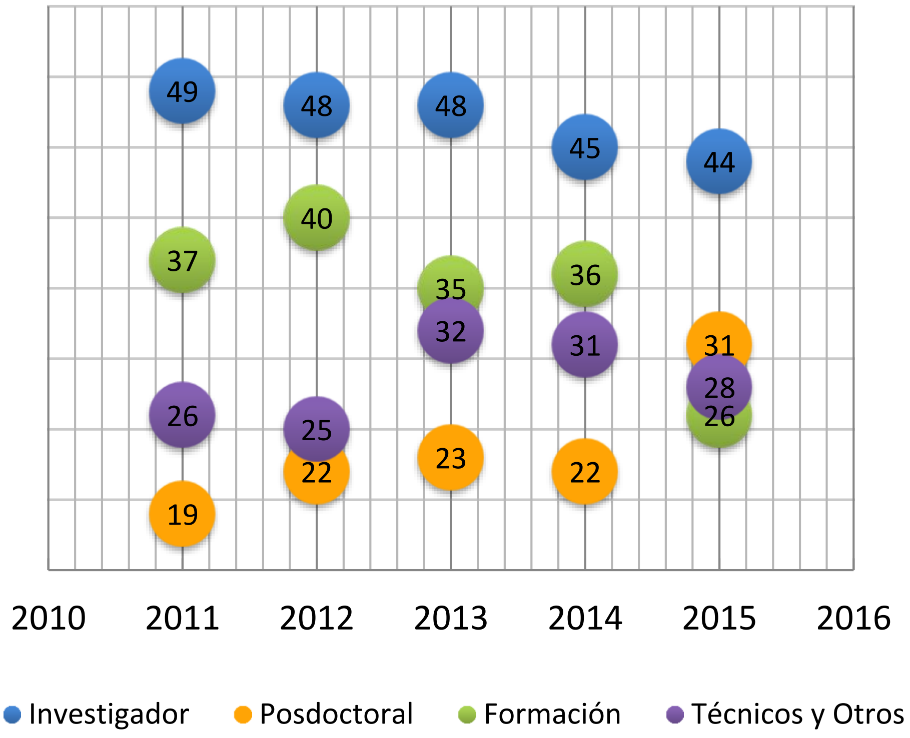
RECURSOS HUMANOS / HUMAN RESOURCES

Distribución del personal por categorías - 2015
Distribution by professional category - 2015

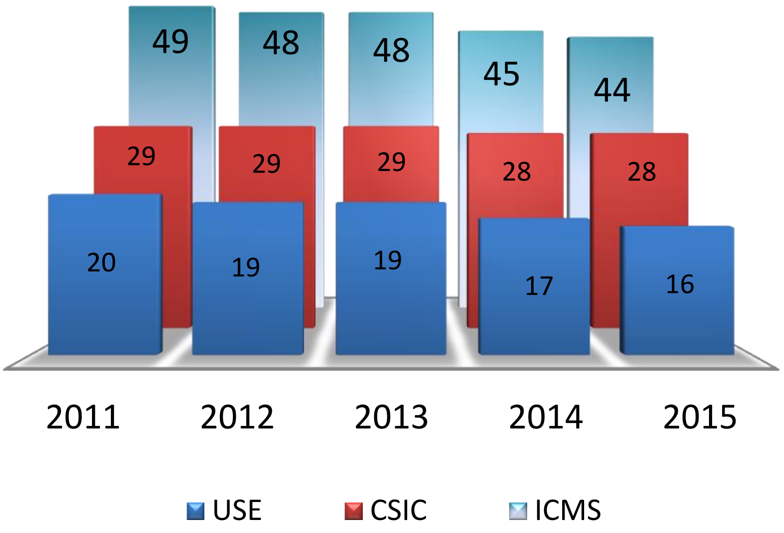




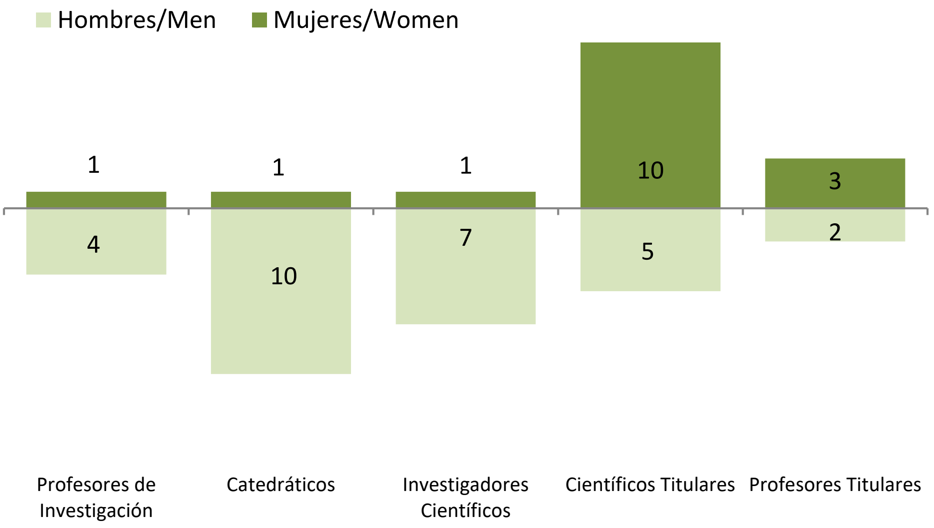
Evolución 2011-2015 del personal
Evolution of Staff

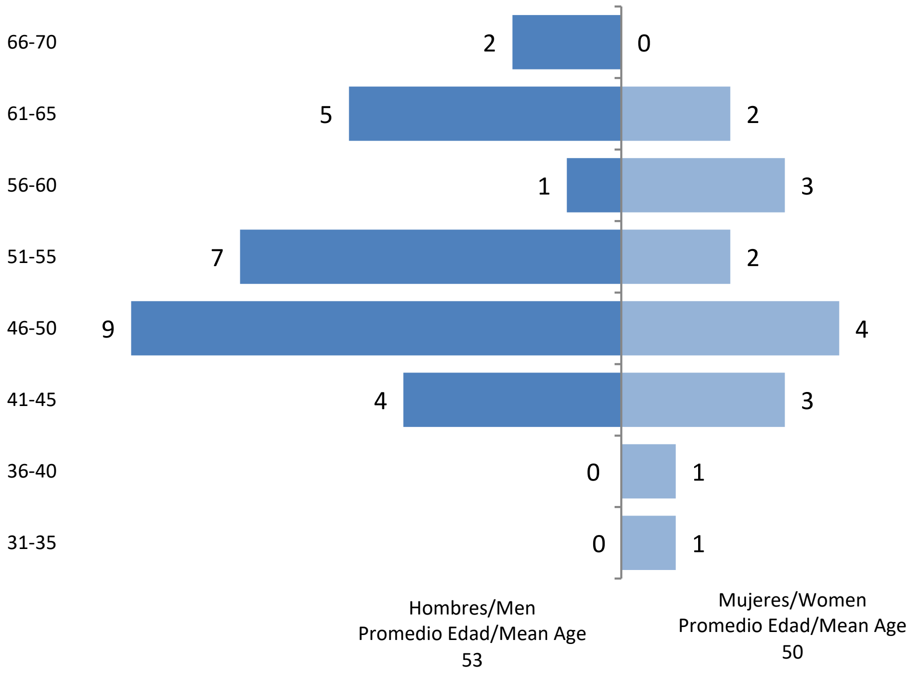


Evolución 2011-2015 del personal investigador
Evolution of Research Staff



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





FINANCIACIÓN / FUNDING

Evolución 2011-2015 de la Financiación conseguida por año
Evolution of the funding of the ICMS

PRODUCCIÓN CIENTÍFICA / SCIENTIFIC PRODUCTION

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

REVISTA JOURNAL	ARTÍCULOS PAPERS	FACTOR DE IMPACTO IMPACT FACTOR (*)
NANO LETTERS	1	13,779
JOURNAL OF THE AMERICAN CHEMICAL SOCIETY	1	13,038
ANGEWANDTE CHEMIE-INTERNATIONAL EDITION	1	11,709
JOURNAL OF PHYSICAL CHEMISTRY LETTERS	2	8,539
APPLIED CATALYSIS B-ENVIRONMENTAL	5	8,328
SMALL	1	8,315
JOURNAL OF MATERIALS CHEMISTRY A	1	8,262
NANOSCALE	1	7,760
PROGRESS IN PHOTOVOLTAICS	1	7,365
JOURNAL OF CATALYSIS	1	7,354
ACS APPLIED MATERIALS & INTERFACES	4	7,145
CHEMSUSCHEM	1	7,116
JOURNAL OF POWER SOURCES	2	6,333
ACTA BIOMATERIALIA	1	6,008
ADVANCED HEALTHCARE MATERIALS	1	5,760
APPLIED ENERGY	1	5,746
ACS PHOTONIC	1	5,404
CHEMICAL ENGINEERING JOURNAL	5	5,310
CATALYSIS SCIENCE & TECHNOLOGY	1	5,287
SCIENTIFIC REPORTS	1	5,228
ADVANCED OPTICAL MATERIALS	2	5,188
INORGANIC CHEMISTRY	1	4,820
ELECTROCHIMICA ACTA	1	4,803
SENSORS AND ACTUATORS B-CHEMICAL	1	4,758
ANALYTICA CHIMICA ACTA	1	4,712
JOURNAL OF PHYSICAL CHEMISTRY C	5	4,509
CATALYSIS TODAY	4	4,312
ANALYST	1	4,033
APPLIED CATALYSIS A-GENERAL	4	4,012

REVISTA JOURNAL	ARTÍCULOS PAPERS	FACTOR DE IMPACTO IMPACT FACTOR (*)
MATERIALS & DESIGN	1	3,997
LANGMUIR	2	3,993
JOURNAL OF MOLECULAR CATALYSIS A-CHEMICAL	1	3,958
JOURNAL OF CHROMATOGRAPHY A	1	3,926
COMPOSITES PART B-ENGINEERING	1	3,850
CRYSTENGCOMM	1	3,849
FUEL	1	3,611
NANOTECHNOLOGY	1	3,573
SCIENCE AND TECHNOLOGY OF ADVANCED MATERIALS	1	3,433
MICROPOROUS AND MESOPOROUS MATERIALS	1	3,349
RSC ADVANCES	5	3,289
INTERNATIONAL JOURNAL OF HYDROGEN ENERGY	3	3,205
APPLIED SURFACE SCIENCE	4	3,150
PLOS ONE	1	3,057
JOURNAL OF ALLOYS AND COMPOUNDS	1	3,014
CATALYSTS	1	2,964
JOURNAL OF THE EUROPEAN CERAMIC SOCIETY	3	2,933
JOURNAL OF THE MECHANICAL BEHAVIOR OF BIOMEDICAL MATERIALS	1	2,876
ULTRAMICROSCOPY	1	2,874
ENERGY & FUELS	1	2,835
PLASMA PROCESSES SCIENCE & TECHNOLOGY	1	2,808
JOURNAL OF THE AMERICAN CERAMIC SOCIETY	1	2,787
CERAMICS INTERNATIONAL	2	2,758
PLASMA PROCESSES AND POLYMERS	1	2,713
EUROPEAN JOURNAL OF INORGANIC CHEMISTRY	1	2,686
SPECTROCHIMICA ACTA PART A-MOLECULAR AND BIOMOLECULAR SPECTROSCOPY	1	2,653
APPLIED CLAY SCIENCE	5	2,586
INTERNATIONAL JOURNAL OF ENVIRONMENTAL SCIENCE AND TECHNOLOGY	1	2,344
OPTICS EXPRESS	1	2,295
INTERNATIONAL JOURNAL OF REFRACTORY METALS & HARD MATERIALS	3	2,263
PHOTOCHEMICAL & PHOTOBIOLOGICAL SCIENCES	1	2,235
OPTICAL MATERIALS	1	2,183
SURFACE & COATINGS TECHNOLOGY	1	2,139
MATERIALS CHEMISTRY AND PHYSICS	1	2,101

REVISTA JOURNAL	ARTÍCULOS PAPERS	FACTOR DE IMPACTO IMPACT FACTOR (*)
JOURNAL OF APPLIED PHYSICS	1	2,101
JOURNAL OF NANOPARTICLE RESEARCH	1	2,101
SENSORS	1	2,033
ANALYTICAL METHODS	1	1,915
JOURNAL OF APPLIED POLYMER SCIENCE	1	1,866
MICRON	2	1,838
RESEARCH ON CHEMICAL INTERMEDIATES	1	1,833
COMPTES RENDUS CHIMIE	1	1,798
JOURNAL OF THERMAL ANALYSIS AND CALORIMETRY	2	1,781
THIN SOLID FILMS	1	1,761
JOURNAL OF MATERIALS RESEARCH	1	1,579
INTERNATIONAL JOURNAL OF APPLIED CERAMIC TECHNOLOGY	1	1,534
SOLID STATE COMMUNICATIONS	1	1,458
JOURNAL OF NANOSCIENCE AND NANOTECHNOLOGY	2	1,338
SOFT MATERIALS	1	1,333
SURFACE AND INTERFACE ANALYSIS	1	1,018
PHYSICS OF THE SOLID STATE	2	0,831
OIL & GAS SCIENCE AND TECHNOLOGY-REVUE D IFP ENERGIES NOUVELLES	1	0,750
Total	**127**	**4,155**

(*) Factor de Impacto correspondiente al año 2015
Journal Citation Reports of 2015

COMPOSICIÓN Y ESTRUCTURA

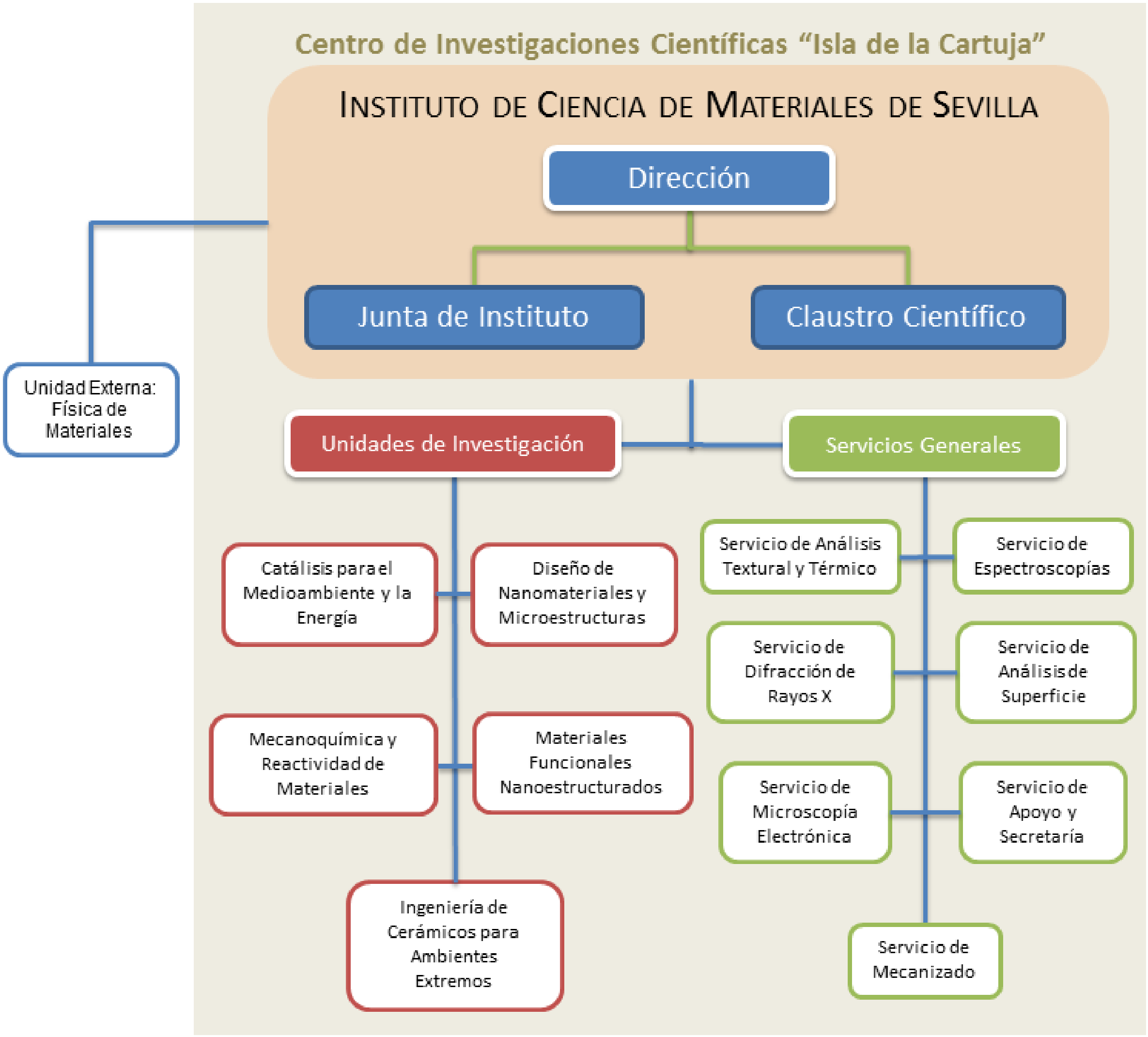
STRUCTURE AND ORGANISATION

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



DIRECCIÓN / DIRECTORATE

Director / Director:	D. Alfonso Caballero Martínez
Vicedirector / Vicedirector:	Dª Ana Isabel Becerro Nieto

JUNTA DE INSTITUTO / INSTITUTE BOARD

Presidente / Chairman:	D. **Alfonso Caballero Martínez**
Vicedirector / Vicedirector:	D. **Ana Isabel Becerro Nieto**

Vocales / Members:

D. **Juan Pedro Holgado Vázquez**
Representante U.I. Catálisis para el Medioambiente y la Energía
D. **José Jesús Benítez Jiménez**
Representante U.I. Ingeniería de Cerámicos para Ambientes Extremos
D. **Luis A. Pérez Maqueda**
Representante U.I. Mecanoquímica y Reactividad de Materiales
D. **Alberto Palmero Acebedo**
Representante U.I. Materiales Funcionales Nanoestructurados
Dª **Asunción Fernández Camacho**
Representante U.I. Diseño de Nanomateriales y Microestructuras
D. **Alejandro Conde Amiano**
Representante U.E.I. Física de Materiales
D. **Julián Martínez Fernández**
Representante del Profesorado de plantilla de la Universidad de Sevilla
D. **Juan Carlos Sánchez López**
Representante del Personal Científico de plantilla del CSIC
D. **Juan Carlos Martín Sánchez**
Representante del Personal No Científico y No Profesorado de Plantilla

CLAUSTRO CIENTÍFICO / SCIENTIFIC BOARD

Presidente / Chairman: D. Alfonso Caballero Martínez

Vocales
Members

Alba Carranza, María Dolores
Alvarez Molina, Rafael
Arzac Di Tomaso, Gisela M.
Ayala Espinar, María Regla
Becerro Nieto, Ana Isabel
Benítez Jiménez, José Jesús
Borrego Moro, Josefa
Calvo Roggiani, Mauricio
Castillo Hernández, Paula M.
Centeno Gallego, Miguel Angel
Colón Ibáñez, Gerardo
Córdoba Gallego, José Manuel
Cruz Carrillo, Miguel Antonio
Díaz Cuenca, María Aranzazu
Domínguez Rodríguez, Arturo
Espinós Manzorro, Juan Pedro
Estrada de Oya, María Dolores
Fortio Godinho, Vanda C.
Franco García, Victorino
García García, Francisco J.
Geng, Dongling
Gómez García, Diego
Gotor Martínez, Francisco José
Holgado Vázquez, Juan Pedro
Jiménez de Haro, M. Carmen
Justo Erbez, Angel
Laguna Espitia, Oscar Hernando
López Santos, María del Carmen
Macías Azaña, Manuel
Martínez Fernández, Julián
Míguez García, Hernán
Navío Santos, José Antonio
Ocaña Jurado, Manuel
Orta Cuevas, M. Mar
Pavón González, Esperanza
Perejón Pazo, Antonio
Pérez Maqueda, Luis Allan
Ramírez de Arellano-López, Antonio
Real Pérez, Concepción
Rodríguez González-Elipe, Agustín
Romero Sarria, Francisca
Sánchez Jiménez, Pedro E.
Sánchez Soto, Pedro José
Salazar Carballo, Pedro A.
Yubero Valencia, Francisco

Alcalá González, María Dolores
Aparicio Rebollo, Francisco J.
Avilés Escaño, Miguel Angel
Barranco Quero, Angel
Beltrán Custodio, Ana María
Borrás Martos, Ana Isabel
Bravo León, Alfonso
Carretero Palacios, Sol
Castro Arroyo, Miguel Angel
Clauss Klamp, Caroline
Conde Amiano, Clara F.
Cotrino Bautista, José
Diánez Millán, María Jesús
Domínguez Leal, María Isabel
Escudero Belmonte, Alberto
Esquivias Fedriani, Luis M.
Fernández Camacho, Asunción
Galisteo López, Juan Francisco
Gallardo Cruz, Carmen
García Navarro, Ana
Gil Rostra, Jorge
Gómez Ramírez, Ana María
Hidalgo López, M. Carmen
Ivanova, Svetlana
Jiménez Melendo, Manuel
Li, Yuelong
López Flores, Victor
Lozano Barbero, Gabriel
Malet Maenner, Pilar
Martínez Tejada, Leidy Marcela
Muñoz Bernabé, Antonio
Núñez Alvarez, Nuria
Odriozola Gordón, José Antonio
Palmero Acebedo, Alberto
Penkova, Anna Dimitrova
Pereñíguez Rodríguez, Rosa
Poyato Galán, Rosalía
Ramírez Rico, Joaquín
Rico Gavira, J. Victor
Rojas Ruiz, T. Cristina
Ruiz Conde, Antonio
Sánchez López, Juan Carlos
Sánchez Valencia, Juan Ramón
Sayagués De Vega, M. Jesús

Unidades de Investigación / Research Units

Catálisis para el medioambiente y la Energía
Catalysis for Enviroment and energy

Personal / Personnel

Catedráticos

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

Investigadores Científicos

Dr. Miguel Angel Centeno Gallego
Dr. Gerardo Colón Ibáñez

Científicos Titulares

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

Profesores Titulares

Dr. Manuel Macías Azaña
Dra. Francisca Romero Sarria

Investigadores Contratados

Dr. Oscar Hernando Laguna Espitia

Profesores Contratado Doctor

Dra. María Isabel Domínguez Leal
Dra. Leidy Marcela Martínez Tejada
Dra. Rosa Pereñíguez Rodríguez
Dra. Svetlana Lyuvimirova Ivanova
Dra. Anna Dimitrova Penkova

Becarios Predoctorales

Ldo. Rafael Castillo Barrero
Lda. Victoria Garcilaso de la Vega-González
Lda. Elena María Jiménez Barrera
Ldo. Alberto Rodríguez Gómez
Lda. Nuria García Moncada
Lda. Miriam González Castaño
Lda. Cristina Megías Sayago
Ldo. José Luis Santos Muñoz

Personal Contratado

Lda. Ángeles López Martín

INGENIERÍA DE CERÁMICOS PARA AMBIENTES EXTREMOS
ENGINEERED CERAMICS FOR EXTREME ENVIRONMENTS

PERSONAL / PERSONNEL

Catedráticos

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

Científicos Titulares

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

Profesores Titulares

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

Investigadores Contratados

Dr. Miguel Antonio Cruz Carrillo
Dra. Esperanza Pavón González

Profesor Contratado Doctor

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

Becarios Predoctorales

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

Personal Contratado

Dra. Paula M. Castillo Hernández
Lda. Aurora Gómez Marín

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

MATERIALES FUNCIONALES NANOESTRUCTURADOS NANOSTRUCTURED FUNCTIONAL MATERIALS

PERSONAL / PERSONNEL

Profesores de Investigación

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

Catedráticos

Dr. José Cotrino Bautista

Investigadores Científicos

Dr. Francisco Yubero Valencia

Científicos Titulares

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

Profesor Visitante

Dr. Richard M. Lambert

Investigadores Contratados

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

Becarios Predoctorales

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

Personal Contratado

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

DISEÑO DE NANOMATERIALES Y MICROESTRUCTURAS
TAILORED NANOMATERIALS AND MICROSTRUCTURE

PERSONAL / PERSONNEL

Profesores de Investigación

Dra. Asunción Fernández Camacho

Catedráticos

Dr. Luis M. Esquivias Fedriani
Dr. Diego Gómez García

Investigadores Científicos

Dr. Juan Carlos Sánchez López

Científicos Titulares

Dra. Rosalía Poyato Galán
Dra. T. Cristina Rojas Ruiz

Investigadores Contratados

Dra. Gisela M. Arzac Di Tomaso
Dra. Ana María Beltrán Custodio
Dra. Vanda C. Fortio Godinho

Becarios Predoctorales

Lda. Mariana Paladini San Martin

Personal Contratado

Ing. Tec. M. Rocío García Gil
Ldo. Dirk Hufschmidt
D. Miguel Nieto Redondo

SERVICIOS GENERALES / GENERAL SERVICES

Servicio de Apoyo y Secretaría
Dª Margarita Adorna Muñoz
Dª Ana García Martín

Informática – Apoyo Dirección
D. José Carlos Rivero Cabello

Servicio de Análisis Textural y Térmico
Dª Cristina Gallardo López

Servicio de Difracción de Rayos X
Ldo. José María Martínez Blanes

Servicio de Espectroscopías
Dr. Miguel Angel Avilés Escaño

Servicio de Mecanizado
Ing. D. Enrique Jiménez Roca
D. Juan Carlos Martín Sánchez
D. Manuel Perea Domínguez

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

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

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

Unidad “Materiales Funcionales Nanoestructurados”
Dr. Victor J. Rico Gavira

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

PERSONAL / PERSONNEL

Catedrático

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

Profesor Titular

Dr. Javier S. Blázquez Gámez
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

UNIDADES DE INVESTIGACIÓN
RESEARCH UNITS

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

PERSONAL / PERSONNEL

Catedráticos

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

Investigadores Científicos

Dr. Miguel Angel Centeno Gallego
Dr. Gerardo Colón Ibáñez

Científicos Titulares

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

Profesores Titulares

Dr. Manuel Macías Azaña
Dra. Francisca Romero Sarria

Investigadores Contratados

Dr. Oscar Hernando Laguna Espitia

Profesores Contratado Doctor

Dra. María Isabel Domínguez Leal
Dra. Leidy Marcela Martínez Tejada
Dra. Rosa Pereñíguez Rodríguez
Dra. Svetlana Lyuvimirova Ivanova
Dra. Anna Dimitrova Penkova

Becarios Predoctorales

Ldo. Rafael Castillo Barrero
Lda. Victoria Garcilaso de la Vega-González
Lda. Elena María Jiménez Barrera
Ldo. Alberto Rodríguez Gómez
Lda. Nuria García Moncada
Lda. Miriam González Castaño
Lda. Cristina Megías Sayago
Ldo. José Luis Santos Muñoz

Personal Contratado

Lda. Ángeles López Martín

Proyectos de Investigación / Research Projects

Unión Europea
Fondo Europeo de Desarrollo Regional
"Una manera de hacer Europa"

MINISTERIO DE ECONOMÍA Y COMPETITIVIDAD

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

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

RESUMEN / ABSTRACT

Existen en la actualidad fuentes de gas que podríamos agrupar bajo el calificativo de no-convencional que incluyen el que se encuentra confinado en formaciones geológicas de baja permeabilidad, el gas asociado al crudo, los pequeños yacimientos en lugares remotos, el biogas producido en la digestión anaerobia de residuos y los denominados product gas generados en la gasificación de biomasa y en la combustión de alquitran. La composición de todas estas fuentes de gas es similar estando constituidas por una mezcla de metano y dióxido de carbono con cantidades inferiores de otros gases permanentes. La concentración de CO_2 puede llegar a ser de hasta el 40% en volumen como ocurre con el gas asociado de algunos campos off-shore y el biogas producido por fermentación de residuos agrícolas. La valorización de este gas mediante el proceso GTL (Gas to Liquid) es una alternativa cuando su localización remota o distante de los gaseoductos no permite ser agregado a las fuentes convencionales o no puede ser consumido in situ, ya que los combustibles líquidos son más fáciles de almacenar y transportar y tienen aplicación directa en el transporte.

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

En el presente proyecto se pretende desarrollar la tecnología de microcanales para el proceso GTL utilizando mezclas metano-dióxido de carbono para simular las fuentes de gas no convencional. Los estudios que hemos venido realizando sobre reactores de microcanales deberán ampliarse a condiciones de presión y temperatura más drásticas, lo que debe permitir validar y mejorar la selección de materiales para la construcción y las técnicas de unión.

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

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

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

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

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

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

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).

Unión Europea
Fondo Europeo de Desarrollo Regional
"Una manera de hacer Europa"
JUNTA DE ANDALUCIA

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

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

RESUMEN / ABSTRACT

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

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

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

Código/Code:	P11-TEP-8196 (Proyecto de Excelencia)
Periodo/Period:	01-02-2013 / 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

Catalizadores nanoestructurados para aplicaciones ambientales y producción de compuestos de química fina

Periodo/Period:	29-10-2014 / 30-04-2015
Oganismo Financiador/Financial source:	COLCIENCIAS. Convocatoria complementos para proyectos con cofinanciación internacional para la comunidad COLCIENCIAS en el exterior
Investigador responsable/Research head:	Oscar Hernando Laguna Espitia

CONVENIOS Y CONTRATOS / CONTRACTS AND AGREEMENTS

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

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

EXPERIMENTOS EN GRANDES INSTALACIONES / LARGE FACILITY EXPERIMENTS

Sincrotrón SOLEIL / SOLEIL Synchrotron

The role of the ionic conductor in the water activation in WGS reaction for H_2 production

Código/Code:	20140594
Periodo/Period:	23-11-2015 / 30-11-2015
Organismo Financiador/Financial source:	SOLEIL Synchrotron
Instalación Científica:	SOLEI L-Línea SAMBA GIF-sur-YVETTE (Francia)
Investigador principal/Research head:	Víctor López Flores
Investigadores/Researchers:	Nuria García Moncada, Cristina Megías

Influence of activation temperature and heating ramp on the metal surface distribution related to catalytic activity in Pt-Fe-Cu/Al_2O_3 catalysts for PROX reactions

Código/Code:	20141399
Periodo/Period:	02-11-2015 / 09-11-2015
Organismo Financiador/Financial source:	SOLEIL Synchrotron
Instalación Científica:	SOLEI L-Línea TEMPO GIF-sur-YVETTE (Francia)
Investigador principal/Research head:	Víctor López Flores
Investigadores/Researchers:	Rafael Castillo Barrero, José Antonio Odriozola

The role of water activation boosted in water gas shift reaction for pure H_2 production

Código/Code:	201502121
Periodo/Period:	02-11-2015 / 10-11-2015
Organismo Financiador/Financial source:	ALBA Synchrotron
Instalación Científica:	Synchrotron ALBA, BL22-CLAESS Line Barcelona (España)
Investigador principal/Research head:	Víctor López Flores
Investigadores/Researchers:	Sara Navarro Jaén, José Luis Santos, Svetlana Ivanova, Nuria García Moncada

PATENTES / PATENTS

Procedimiento de obtención de un fotocatalizador heteroestructurado de $BiPO_4$-TiO_2-GC_3N_4

Inventores: Gerardo Colón Ibáñez, Sergio Alberto Obregón Alfaro
Tipo de Patente: Nacional
Solicitud número: 201530925
Fecha Solicitud: 26 Junio 2015
Entidad Titular: CSIC

Complejo catalítico conformado por la mezcla de catalizador y conductor iónico

Inventores: José Antonio Odriozola Gordón, Svetlana Ivanova, Francisca Romero-Sarria, Miriam González-Castaño, Nuria García Moncada, Miguel Angel Centeno Gallego
Tipo de Patente: Nacional
Solicitud número: P201500441
Fecha Solicitud: 02 Junio 2015
Entidad Titular: CSIC

UNIDAD ASOCIADA / ASSOCIATED UNIT

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

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

El responsable de esta Unidad Asociada, por parte de la Universidad de Las 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

Theory and Practice: Bulk Synthesis of C_3B and its H_2- and Li-Storage Capacity

King, TC; Matthews, PD; Glass, H; Cormack, JA; Holgado, JP; Leskes, M; Griffin, JM; Scherman, OA; Barker, PD; Grey, CP; Dutton, SE; Lambert, RM; Tustin, G; Alavi, A; Wright, DS

Angewandte Chemie International Edition, **54** (2015) 5919-5923

May, 2015 | **DOI:** 10.1002/anie.201412200

Previous theoretical studies of C_3B have suggested that boron-doped graphite is a promising H_2- and Li-storage material, with large maximum capacities. These characteristics could lead to exciting applications as a lightweight H_2-storage material for automotive engines and as an anode in a new generation of batteries. However, for these applications to be realized a synthetic route to bulk C_3B must be developed. Here we show the thermolysis of a single-source precursor (1,3-$(BBr_2)2C_6H4$) to produce graphitic C_3B, thus allowing the characteristics of this elusive material to be tested for the first time. C_3B was found to be compositionally uniform but turbostratically disordered. Contrary to theoretical expectations, the H_2- and Li-storage capacities are lower than anticipated, results that can partially be explained by the disordered nature of the material. This work suggests that to model the properties of graphitic materials more realistically, the possibility of disorder must be considered.

Synthesis and application of layered titanates in the photocatalytic degradation of phenol

Ivanova, S; Penkova, A; Hidalgo, MD; Navio, JA; Romero-Sarria, F; Centeno, MA; Odriozola, JA

Applied Catalysis B: Environmental, **163** (2015) 23-29

February, 2015 | **DOI:** 10.1016/j.apcatb.2014.07.048

This study proposes a direct synthetic route to single titanate sheets through the mild and versatile conditions of the “chimie douce”. The stages of the production include the complexation of the titanium alkoxide precursor by benzoic acid, the formation of titanium oxo-clusters and their controlled transformation into single sheet titanates during the hydrolysis stage. The resulted material appears to be an excellent precursor for self-organized TiO_2 nanotubes formation which presents an excellent activity as photocatalyst in the photo-degradation of phenol.

Evolution of H-2 photoproduction with Cu content on $CuOx$-TiO_2 composite catalysts prepared by a microemulsion method

Kubacka, A; Munoz-Batista, MJ; Fernandez-Garcia, M; Obregon, S; Colon, G

Applied Catalysis B: Environmental, **163** (2015) 214-222

Febrero, 2015 | **DOI:** 10.1016/j.apcatb.2014.08.005

Copper oxides in contact with anatase correspond to promising materials with high activity in the photo-production of hydrogen by aqueous reforming of alcohols. By a single pot microemulsion method we obtained a series of Cu-Ti composite systems with controlled copper content in the 0-25 wt.% range. The scanning of such a wide range of composition led to the discovery of two well differentiated maxima in the photo-reaction performance. These maxima present rather high and relatively similar reaction rates and photonic efficiencies but are ascribed to the presence of different copper species. A multi-technique analysis of the materials indicates that the maxima obtained comes from optimizing different steps of the reaction; while the first would be connected with a positive effect on anatase charge handling performance the second seems exclusively related to electron capture by surface copper species.

Synergy between gold and oxygen vacancies in gold supported on Zr-doped ceria catalysts for the CO oxidation

Laguna, OH; Perez, A; Centeno, MA; Odriozola, JA
Applied Catalysis B: Environmental, **176** (2015) 385-395
October, 2015 | **DOI:** 10.1016/j.apcatb.2015.04.019

The CO oxidation activity of 1 wt.% gold catalysts prepared by deposition-precipitation on a series of ceria doped with Zr supports was studied. The supports (10, 25 and 50 Zr at.%) were synthesized by a pseudo sol-gel method through the thermal decomposition of the corresponding metallic propionates. All the prepared solids were characterized by means of XRF, BET, XRD, Raman spectroscopy, SEM, and H-2-TPR. Solid solution was obtained in all mixed systems, while the segregation of different Ce-Zr oxides was observed for the solid with the 50 Zr at.%. The oxygen vacancies population and the amount of easier reducible Ce4+ species in the solids increase with the Zr content. No major textural or structural modifications were detected after gold deposition, although a strong Au-support interaction was generated. Such interaction is strongly influenced by the nucleation of gold deposits on the oxygen vacancies and consequently the amount of Zr inserted in the ceria network also determines the dispersion of gold. The presence of gold eases the surface reduction at lower temperatures, and as higher the amount of Zr in the gold catalysts, higher the CO conversion at low temperatures, probably due to the enhancement of the electronic transfer at the surface of the catalysts.

Cu–TiO_2 systems for the photocatalytic H_2 production: Influence of structural and surface support features

Obregon, S; Munoz-Batista, MJ; Fernandez-Garcia, M; Kubacka, A; Colon, G
Applied Catalysis B: Environmental, **179** (2015) 468-478
December, 2015 | **DOI:** 10.1016/j.apcatb.2015.05.043

The influence of different TiO_2 supports on the Cu active species has been studied. It was found that the photocatalytic H_2 evolution is highly affected by the structural and electronic features of surface Cu species. Thus, metal dispersion and oxidation state appears strongly conditioned by the structural and surface properties of the TiO_2 support. We have examined three TiO_2 supports prepared by different synthetic methods; sol–gel, hydrothermal and microemulsion. In addition, we have induced structural and surface modifications by sulfate pretreatment over freshly prepared TiO_2 precursors and subsequent calcination. Notably different copper dispersion and oxidation state is obtained by using these different TiO_2 supports. From the wide structural and surface analysis of the catalysts we are able to propose that the occurrence of highly disperse Cu^{2+} species, the sample surface area as well as the crystallinity of the TiO_2 support are directly related to the photocatalytic activity for H2 production reaction.

Study of the phenol photocatalytic degradation over TiO_2 modified by sulfation, fluorination, and platinum nanoparticles photodeposition

Murcia, JJ; Hidalgo, MC; Navio, JA; Arana, J; Dona-Rodriguez, JM
Applied Catalysis B: Environmental, **179** (2015) 305-312
December, 2015 | **DOI:** 10.1016/j.apcatb.2015.05.040

In this work, titanium dioxide has been modified by sulfation, fluorination and simultaneous Pt nanoparticles deposition; the influence of these treatments on the photocatalytic activity of this oxide has been studied. A complete characterization study was carried out and it was observed that sulfation, fluorination and metallization were important factors influencing the TiO_2 properties. The photocatalytic activity of the materials prepared was evaluated in the phenol degradation and it was found that TiO_2 fluorination significantly increased the phenol photodegradation rate, compared with bare TiO_2, sulfated TiO_2 or the commercial TiO_2 Degussa P25. It was also found that Pt photodeposition on sulphated TiO_2 notably increased the photocatalytic activity of this oxide, while Pt on fluorinated TiO_2 did not modify significantly the phenol photodegradation rate.

H-2 oxidation as criterion for PrOx catalyst selection: Examples based on Au-Co-O-x-supported systems

Reina, TR; Megias-Sayago, C; Florez, AP; Ivanova, S; Centeno, MA; Odriozola, JA
Journal of Catalysis, **326** (2015) 161-171
June, 2015 | **DOI:** 10.1016/j.jcat.2015.03.015

A new approach for understanding PrOx reaction over gold catalysts is proposed in this work. The competition between H-2 and CO oxidation has been studied over a series of $Au/MOx/Al_2O_3$ (M = Ce and Co) catalysts in simulated post-reforming gas stream, containing H_2O and CO_2 for H-2 cleanup goals. The catalysts' behavior is correlated to their oxygen storage capacity, redox behavior, and oxidation ability. The estimation of the reaction rates reveals that in these solids the H-2 combustion, the selectivity limiting factor in the PrOx process, is mainly controlled by the support and not by the gold presence. The possible use of the hydrogen oxidation reaction as a catalyst selection criterion is discussed.

Microreactors technology for hydrogen purification: Effect of the catalytic layer thickness on $CuOx/CeO_2$-coated microchannel reactors for the PROX reaction

Laguna, O. H.; Castano, M. Gonzalez; Centeno, M. A.; Odriozola, J. A.
Chemical Engineering Journal, **271** (2015) 45-52
September, 2015 | DOI: 10.1016/j.cej.2015.04.023

Two blocks of microreactors composed by 100 microchannels and coated, respectively, with 150 and 300 mg of a $CuOx/CeO_2$ catalyst, were prepared and tested in the preferential oxidation of CO in presence of H_2 (PROX). The deposition of different amount of catalyst resulted in different catalytic layer thicknesses thus modifying the catalytic performances of the microreactor. The evaluation of the main reaction variables (the space velocity, the O_2-to-CO ratio and the presence of H_2O and/or CO_2 in the stream) was performed over both microreactors and compared to that of the parent powder catalyst. The least loaded microreactor, with a coating thickness around 10 µm, presented the highest CO conversion and selectivity levels at temperatures below 160 °C. This result evidences (i) the improvement of the catalytic performances got by the structuration of the powder catalyst and (ii) the importance of the selection of the adequate thickness of the catalytic layer on the microreactor, which have not to exceed and optimal value. An adequate coating thickness allows minimizing the mass and heat transport limitations, thus resulting in the enhancement of the catalytic performance during the PROX reaction.

Enhancement of stability and photoactivity of TiO_2 coatings on annular glass reactors to remove emerging pollutants from waters

Espino-Estevez, MR; Fernandez-Rodriguez, C; Gonzalez-Diaz, OM; Navio, JA; Fernandez-Hevia, D; Dona-Rodriguez, JM
Chemical Engineering Journal, **279** (2015) 488-497
November, 2015 | **DOI:** 10.1016/j.cej.2015.05.038

TiO_2 coatings of highly photoactive lab-made titania were prepared on the outer wall of the inner tube of a glass tubular reactor by dip-coating method. The effect of decreasing the size of the aggregates to improve adhesion and photoactivity of the coatings to degrade phenol, diclofenac and isoproturon was also investigated. Chemical disaggregation of the TiO_2 particles resulted in a lower aggregate size, between 0.1 and 1 µm, than mechanical disaggregation, between 1 and 10 µm. The results of the adhesion tape test showed that either milling of aggregate material with a planetary mill or chemical stabilization of the particles were necessary to obtain TiO_2 coatings on glass tube with acceptable quality to be used in water treatment applications. SEM images showed that coatings prepared after milling the TiO_2 suspension were more homogeneous without surface aggregates. The degree of adhesion of the coatings after increasing the roughness of the support by abrasive blasting was also evaluated. Adhesion to the substrate was slightly lower when using the modified support. The photoactivity results showed that the coatings prepared after wet milling of catalyst during 30 min and after chemical disaggregation were more efficient in terms of degradation and mineralization when using phenol as model molecule. Subsequent studies with two emerging pollutants, diclofenac and isoproturon, also showed enhanced efficiency of these coatings. The reusability of the TiO_2 coatings was also evaluated and a promising photocatalytic performance was observed with a very low variation of the decay rate after five consecutive usages.

Role of ruthenium on the catalytic properties of CeZr and CeZrCo mixed oxides for glycerol steam reforming reaction toward H_2 production

Martinez, LM; Araque, M; Centeno, MA; Roger, AC
Catalysis Today, **242** (2015) 80-90
March, 2015 | **DOI:** 110.1016/j.cattod.2014.07.034

The effect of ruthenium on the physico-chemical properties of CeZr and CeZrCo mixed oxides for H_2 production by glycerol steam reforming reaction has been studied. The combination of in situ Raman spectroscopy under both reductive and oxidative conditions, H_2/O_2 pulses and XRD, Raman, BET analysis, H_2-TPR and TPD-TPO analyses contributed to the determination of the structural and textural properties, redox behavior, re-oxidation capacity and resistance to carbon deposition of the synthesized catalysts. The results show that the catalytic activity is improved by the (positive) cooperative and complementary effect between cobalt and ruthenium that favors the selectivity toward the steam reforming, selective to H_2, with respect to the unselective thermal decomposition of glycerol. Ruthenium stabilizes the cobalt cations inserted in the fluorite structure preventing its rejection as Co_3O_4; and provides the necessary hydrogen to reduce Ce^{4+}. The combination cobalt–ruthenium modifies positively the redoxproperties of the catalysts, increases the re-oxidation capacity (O_SC) and promotes the gasification of the carbon deposits. Under the reaction conditions, the decrease in glycerol conversion came along with a change of selectivity. The formation of H_2 and CO_2 were strongly decreased, while the formation of CO, C_2H_4 and condensable products (mainly hydroxyacetone) increase. The differences in the catalytic stability and activity of the catalysts are related to the capability of the catalysts to activate H_2O under the reaction conditions, favoring the steam reforming reaction over the thermal decomposition.

Photocatalytic activity of bismuth vanadates under UV-A and visible light irradiation: Inactivation of Escherichia coli vs oxidation of methanol

Adan, C; Marugan, J; Obregon, S; Colon, G
Catalysis Today, **240** (2015) 93-99
February, 2015 | DOI: 10.1016/j.cattod.2014.03.059

Four bismuth vanadates have been synthesized by using two different precipitating agents (NH_3 and triethylamine) following a hydrothermal treatment at 100 °C for 2 h and at 140 °C for 20 h. Then, solids were characterized by X-ray diffraction, BET surface area, UV–vis spectroscopy and scanning microscopy techniques. The characterization of the synthesized materials showed a well crystallized scheelite monoclinic structure with different morphologies. All materials display optimum light absorption properties for visible light photocatalytic applications. The photocatalytic activity of the catalysts was investigated for the inactivation of Escherichia coli bacteria and the oxidation of methanol under UV–vis and visible light irradiation sources. Main results demonstrate that $BiVO_4$ are photocatalytically active in the oxidation of methanol and are able to inactivate bacteria below the detection level. The activity of the catalyst decreases when using visible light, especially for methanol oxidation, pointing out differences in the reaction mechanism. In contrast with bacteria, whose interaction with the catalyst is limited to the external surface, methanol molecules can access the whole material surface.

Boosting the activity of a $Au/CeO_2/Al_2O_3$ catalyst for the WGS reaction

Reina, T. R.; Ivanova, S.; Centeno, M. A.; Odriozola, J. A.
Catalysis Today, **253** (2015) 149-154
September, 2015 | **DOI:** 10.1016/j.cattod.2015.01.041

Herein a strategy to design highly efficient $Au/CeO_2/Al_2O_3$ based WGS catalysts is proposed. The inclusion of transition metals, namely Fe, Cu and Zn as CeO_2 dopant is considered. All the promoters successfully increased the WGS performance of the undoped sample. The activity improvement can be correlated to structural and/or redox features induced by the dopants. The comparative characterization of the doped samples by means of XRD, Raman spectroscopy and OSC evaluation permits an accurate understanding of the boosted WGS activity arising from the

Ce-promoter interaction. This study establishes distinction among both, structural and redox sources of promotion and provides a useful strategy to develop highly active Au/CeO_2 based catalysts for the WGS reaction.

Oxodiperoxomolybdenum complex immobilized onto ionic liquid modified SBA-15 as an effective catalysis for sulfide oxidation to sulfoxides using hydrogen peroxide

Carrasco, Carlos J.; Montilla, Francisco; Bobadilla, Luis; Ivanova, Svetlana; Antonio Odriozola, Jose; Galindo, Agustin

Catalysis Today, **255** (2015) 102-108

October, 2015 | **DOI:** 10.1016/j.cattod.2014.10.053

A supported ionic-liquid-phase (SILP) was prepared by the reaction of 1-methyl-3-(3-(triethoxysilyl) propyl)-1H-imidazol-3-ium chloride with a mesoporous SBA-15 silica and then an oxodiperoxomolybdenum complex was immobilized onto the obtained SILP. The resulting material, identified as SBA-15 + ImCl+ MoO5, was characterized by solid state NMR (H-1, C-13 and Si-29), and their textural and thermogravimetric properties were determined. The SBA-15 + ImCl+ MoO5 material was investigated as catalyst for the oxidation of methylphenylsulfide, as model reaction, with aqueous hydrogen peroxide as oxidant at room temperature. The presence of the molybdenum species was crucial for achieving good conversions and methanol was selected as the best solvent (conversion of 95% and selectivity toward sulfoxide 98%). The optimized reaction conditions were applied for the oxidation of several selected sulfides. In general, good catalytic activity and selectivity to sulfoxide were obtained and, remarkably, the selectivity toward sulfoxide is higher than those observed in the study of the same process carried out in [C(4)min][PF6] (C(4)mim = 1-buty1-3-methylimidazolium) and catalyzed by a molecular molybdenum complex, under the same reaction conditions. The importance of the IL-functionalization in the SBA-15 material was evidenced by recycling experiments. The SBA-15 + ImCl+ MoO5 catalyst was used for the sulfoxidation of the methylphenylsulfide substrate for ten reaction cycles without a significant change in conversion, selectivity to sulfoxide and molybdenum content.

Glycerol steam reforming on bimetallic NiSn/CeO_2-MgO-Al_2O_3 catalysts: Influence of the support, reaction parameters and deactivation/regeneration processes

Bobadilla, LP; Penkova, A; Alvarez, A; Dominguez, MI; Romero-Sarria, F; Centeno, MA; Odriozola, JA

Applied Catalysis A: General, **492** (2015) 38-47

February, 2015 | **DOI:** 10.1016/j.apcata.2014.12.029

NiSn bimetallic catalysts supported over Al_2O_3 modified with different promoter (Mg and/or Ce) were prepared and characterized by powder X-ray diffraction (XRD), N_2 sorptometry, and temperature programmed reduction (TPR). Hydrogen production by glycerol steam reforming over these catalysts was investigated. Among the catalysts, NiSn/AlMgCe catalyst shows the highest hydrogen yield as well as the best stability during the reaction. The effect of reaction temperature, water/glycerol molar ratio and space velocity on the glycerol steam reforming over NiSn/AlMgCe were also investigated. Finally, it was verified that the catalyst can be regenerated by oxidation of carbonaceous deposits.

Mono and bimetallic Cu-Ni structured catalysts for the water gas shift reaction

Arbelaadez, O; Reina, TR; Ivanova, S; Bustarnante, F; Villa, AL; Centeno, MA; Odriozola, JA
Applied Catalysis A: General, **497** (2015) 1-9
May, 2015 | **DOI:** 10.1016/j.apcata.2015.02.041

The water-gas shift (WGS) reaction over structured Cu, Ni, and bimetallic Cu-Ni supported on active carbon (AC) catalysts was investigated. The structured catalysts were prepared in pellets form and applied in the medium range WGS reaction. A good activity in the 180–350 °C temperature range was registered being the bimetallic Cu-Ni:2-1/AC catalyst the best catalyst. The presence of Cu mitigates the methanation activity of Ni favoring the shift process. In addition the active carbon gasification reaction was not observed for the Cu-containing catalyst converting the active carbon in a very convenient support for the WGS reaction. The stability of the bimetallic Cu-Ni:2-1/AC catalyst under continuous operation conditions, as well as its tolerance towards start/stop cycles was also evaluated.

On the origin of the photocatalytic activity improvement of BIVO4 through rare earth tridoping

Obregon, S; Colon, G
Applied Catalysis A: General, **501** (2015) 56-62
July, 2015 | **DOI:** 10.1016/j.apcata.2015.04.032

Rare earth (Tm^{3+}/Er^{3+},Yb^{3+},Y^{3+}) tri-doped BiVO4 have been synthesized by means of a surfactant free hydrothermal method having good photoactivities under sun-like excitation for the degradation of methylene blue and O-2 evolution reactions. The improved photocatalytic performance has been attained by multiple approach of the overall photocatalytic mechanism. From the structural and morphological characterization it has been stated that the presence of Y^{3+} induces the stabilization of the tetragonal phase probably due to its substitutional incorporation in the $BiVO_4$ lattice. Moreover, the extensive doping with rare earth ions such as Yb^{3+} and Er^{3+}/Tm^{3+} t-$BiVO_4$ evidenced that important structural, electronic changes as well as the luminescence properties were also exalted. Ternary doping clearly prompts the higher photocatalytic activities. A more packed tetragonal structure in conjunction leading to improved charge carriers mobility, with the observed visible and NIR photoactivities of t-$BiVO_4$ could be the responsible of the improved photocatalytic activity under solar-like irradiation.

Boosting the visible-light photoactivity of Bi_2WO_6 using acidic carbon additives

Carmona, RJ; Velasco, LF; Hidalgo, MC; Navio, JA; Ania, CO
Applied Catalysis A-General, **505** (2015) 467-477
September, 2015 | **DOI:** 10.1016/j.apcata.2015.05.011

We have explored the role of the physicohemical properties of carbon materials as additives to bismuth tungstate on its structure, optical properties, and photocatalytic activity for the degradation of rhodamine B under visible light. For this purpose, C/Bi_2WO_6 hybrid composites were prepared following two different routes: (i) physical mixture of the catalyst components, and (ii) one-pot hydrothermal synthesis of the semiconductor in the presence of the carbon additive. Three carbons with different properties were selected as additives: biomass-derived activated carbon, carbon nanotubes and carbon spheres obtained from polysaccharides. Data has shown the outstanding role of the acidic/basic nature of the carbon additive, and of the synthetic method on the photocatalytic performance of the resulting composites. For a given additive, the degradation rate of RhB is greatly improved for the catalysts prepared through a one-step hydrothermal synthesis, where there is low shielding effect of the carbon matrix. Carbon additives of acidic nature boost the surface acidity of the hybrid photocatalyst, thereby enhancing the photodegradation of RhB under visible light via a coupled mechanism (photosensitization, semiconductor photocatalysis and carbon-photon mediated reactions).

A comparative study of Bi_2WO_6, CeO_2, and TiO_2 as catalysts for selective photo-oxidation of alcohols to carbonyl compounds

Lopez-Tenllado, FJ; Murcia-Lopez, S; Gomez, DM; Marinas, A; Marinas, JM; Urbano, FJ; Navio, JA; Hidalgo, MC; Gatica, JM
Applied Catalysis A-General, **505** (2015) 375-381
September, 2015 | **DOI:** 10.1016/j.apcata.2015.08.013

Several semiconductors based on ceria or bismuth tungstate were tested for selective oxidation of alcohols to carbonyl compounds in a search for photocatalysts more selective than TiO_2. Gas-phase selective photo-oxidation of propan-2-ol to acetone and liquid-phase transformation of 2-buten-1-ol (crotyl alcohol) to 2-butenal (crotonaldehyde) were studied as test reactions. In both processes the highest selectivities were achieved with Bi_2WO_6-based solids. Further studies on crotyl alcohol transformation evidenced the lower adsorption of the aldehyde on these systems which could minimize the decrease in crotyl alcohol yield observed for TiO_2 or CeO_2 at high conversions. Incorporation of titania (5% molar) to the Bi_2WO_6 system increased the reaction rate significantly whereas the aldehyde yield remained high.

A novel two-steps solvothermal synthesis of nanosized $BiPO_4$ with enhanced photocatalytic activity

Zhang, YF; Sillanpaa, M; Obregon, S; Colon, G
Journal of Molecular Catalysis A-Chemical, **402** (2015) 92-99
June, 2015 | **DOI:** 10.1016/j.molcata.2015.03.011

Nano-sized $BiPO_4$ has been successfully synthesized via a novel designed two-steps solvothermal route using ethylene glycol as solvent. Comparing with traditional hydrothermal method, the novel approach could readily prepare $BiPO_4$ with shorter time. The photocatalytic activity of prepared $BiPO_4$ has been tested via degradation of methylene blue (MB) under light irradiation. The experimental results show that the $BiPO_4$ prepared by novel route had enhanced photocatalytic activity and the synthetic parameters also impact the reaction rate meaningfully. Finally, the obtained samples have been widely characterized by means of powder X-ray diffraction (XRD), Brunauer–Emmett–Teller (BET) surface area, scanning electron microscopy (SEM), UV–vis diffuse reflectance spectra (DRS) and Fourier transformed infrared (FTIR) spectra.

$BiPO_4$ prepared by this novel approach have a particles size below 100 nm, which is a big improvement by comparing with previous works (few micrometer). The effect of EG during the formation of $BiPO_4$ has been discussed and a possible formation mechanism is proposed.

Effective photoreduction of a nitroaromatic environmental endocrine disruptor by AgNPs functionalized on nanocrystalline TiO_2

Hernandez-Gordillo, A; Obregon, S; Paraguay-Delgadod, F; Rodriguez-Gonzalez, V
RSC Advances, **5** (2015) 15194-15197
February, 2015 | DOI: 10.1039/c5ra00094g

Unprecedented photoactivity of silver nanoparticles photodeposited on nanocrystalline TiO_2 for the efficient reduction of 4-nitrophenol at room temperature is reported. The use of Na_2SO_3 as a harmless scavenger agent for the reduction of a nitroaromatic endocrine disruptor yields a valuable 4-aminophenol reagent.

The role of silver nanoparticles functionalized on TiO_2 for photocatalytic disinfection of harmful algae

Lee, Soo-Wohn; Obregon, S.; Rodriguez-Gonzalez, V.
RSC Advances, **5** (2015) 44470-44475
May, 2015 | **DOI:** 10.1039/C5RA08313C

Silver loaded TiO_2 samples were prepared by photodeposition of different amounts of Ag^+ ions over commercial titanium dioxide (Evonik TiO_2 P25) in aqueous media without the presence of sacrificial agents. The obtained photocatalysts were characterized by several techniques such as X-ray powder diffraction (XRD), UV-vis diffuse reflectance spectroscopy (DRS), transmission electron microscopy (TEM) and X-ray photoelectron spectroscopy (XPS) in order to correlate the effect of the silver amount on the photocatalytic properties of the final nanocomposite. The effect of the silver nanoparticles on the photocatalytic behaviour of TiO_2 was evaluated by means of the photodegradation of methyl orange dye and the inactivation of noxious algae Tetraselmis suecicaand Amphidium carterae under continuous exposure of low power irradiation UV-light. The sample with 1.5% wt of silver nanoparticles showed the highest photocatalytic elimination of the azo dye and both algae types. According to the results, the cells were deformed during the photocatalytic process by the attack of highly reactive species such as hydroxyl radicals, H_2O_2 and superoxide ions generated on the TiO_2 surface. The algae cells were not regenerated by themselves after the photocatalytic process due the high degree of fragmentation that they suffered during the light irradiation.

Catalytic screening of $Au/CeO_2\text{-}MO_x/Al_2O_3$ catalysts (M = La, Ni, Cu, Fe, Cr, Y) in the CO-PrOx reaction

Reina, TR; Ivanova, S; Centeno, MA; Odriozola, JA
International Journal of Hydrogen Energy, **40** (2015) 1782-1788
January, 2015 | **DOI:** 10.1016/j.ijhydene.2014.11.141

In this work, a series of Au/CeO_2-MOx/Al_2O_3 catalysts has been prepared and evaluated in the PrOx reaction. Within the series of dopants Fe and Cu containing samples enhanced the catalytic performance of the parent $Au/CeO_2/Al_2O_3$ catalyst being copper the most efficient promoter. For both samples an enhanced oxygen storage capacity (OSC) is registered and accounts for the high CO oxidation activity. More particularly, the Au/CeO_2-CuO_x/Al_2O_3 catalyst successfully withstands the inclusion of water in the PrOx stream and presents good results in terms of CO elimination. However to achieve a good selectivity toward, CO_2 formation properly adjusting of the reaction parameters, such as oxygen concentration and space velocity is needed. Within the whole screened series the Cu-containing catalyst can be considered as the most interesting alternative for H-2 clean-up applications.

Ni-CeO_2/C Catalysts with Enhanced OSC for the WGS Reaction

Pastor-Perez, L; Ramirez-Reina, T; Ivanova, S; Centeno, MA; Odriozola, JA; Sepulveda-Escribano, A
Catalysts, **5** (2015) 298-309
March, 2015 | **DOI:** 10.3390/catal5010298

In this work, the WGS performance of a conventional Ni/CeO_2 bulk catalyst is compared to that of a carbon-supported Ni-CeO_2 catalyst. The carbon-supported sample resulted to be much more active than the bulk one. The higher activity of the Ni-CeO_2/C catalyst is associated to its oxygen storage capacity, a parameter that strongly influences the WGS behavior. The stability of the carbon-supported catalyst under realistic operation conditions is also a subject of this paper. In summary, our study represents an approach towards a new generation of Ni-ceria based catalyst for the pure hydrogen production via WGS. The dispersion of ceria nanoparticles on an activated carbon support drives to improved catalytic skills with a considerable reduction of the amount of ceria in the catalyst formulation.

Photocatalytic reduction of CO_2 over platinised Bi_2WO_6-based materials

Murcia-Lopez, S; Vaiano, V; Hidalgo, MC; Navio, JA; Sannino, D
Photochemical & Photobiological Sciences, **14** (2015) 678-685
April, 2015 | **DOI:** 10.1039/c4pp00407h

The photocatalytic reduction of CO_2 with H_2O to produce CH_4 in the gas phase was carried out in the presence of two Bi_2WO_6-based materials. For this purpose, single Bi_2WO_6 and a coupled Bi_2WO_6-TiO_2 system were synthesised and metallised with Pt, through a Pt photodeposition method. Then, the samples were characterised and the photocatalytic activity was evaluated in a continuous fluidised-bed reactor irradiated with UV light. Single Bi_2WO_6 presents an interesting behaviour under H_2O rich conditions. In particular, the metallisation improves the material's performance for CH_4 formation, while the TiO_2 addition to Bi_2WO_6 increases the CH4 yield only at low H_2O/CO_2 ratio. The Bi_2WO_6-TiO_2 system metallised with a Pt photocatalyst displayed the highest CH_4 yield among all the prepared photocatalysts. The stability of the system can be enhanced through the addition of a blue phosphor to the reactant mixture, especially under H_2O rich conditions.

Structural and chemical reactivity modifications of a cobalt perovskite induced by Sr-substitution. An in situ XAS study

Hueso, JL; Holgado, JP; Pereniguez, R; Gonzalez-DelaCruz, VM; Caballero, A
Materials Chemistry and Physics, **151** (2015) 29–33
February, 2015 | **DOI:** 10.1016/j.matchemphys.2014.11.015

$LaCoO_3$ and $La_{0.5}Sr_{0.5}O_3O_3$-delta perovskites have been studied by in situ Co K-edge XAS. Although the partial substitution of La(III) by Sr(II) species induces an important increase in the catalytic oxidation activity and modifies the electronic state of the perovskite, no changes could be detected in the oxidation state of cobalt atoms. So, maintaining the electroneutrality of the perovskite requires the generation of oxygen vacancies in the network. The presence of these vacancies explains that the substituted perovskite is now much more reducible than the original $LaCoO_3$ perovskite. As detected by in situ XAS, after a consecutive reduction and oxidation treatment, the original crystalline structure of the $LaCoO_3$ perovskite is maintained, although in a more disordered state, which is not the case for the Sr doped perovskite. So, the $La_{0.5}Sr_{0.5}CoO_3$-delta perovskite submitted to the same hydrogen reduction treatment produces metallic cobalt, while as determined by in situ XAS spectroscopy the subsequent oxidation treatment yields a Co(III) oxide phase with spinel structure. Surprisingly, no Co(II) species are detected in this new spinel phase.

Photocatalytic propylene epoxidation on Bi_2WO_6-based photocatalysts

Murcia-Lopez, S.; Vaiano, V.; Sannino, D.; Hidalgo, M. C.; Navio, J. A.
Researchon Chemical Intermediates, **41** (2015) 4199-4212
July, 2015 | **DOI:** 10.1007/s11164-013-1523-3

The photocatalytic epoxidation of propylene (PR) with molecular oxygen was carried out in a fluidized-bed reactor with several Bi_2WO_6-based materials under UV-A illumination. Three different photocatalysts were tested: one of single Bi_2WO_6 and two of coupled Bi_2WO_6-TiO_2 heterostructures, thus showing that a mixed system of Bi_2WO_6 with commercial TiO_2 Degussa-P25 leads to the best combination of conversion and PO selectivity. Then, direct support on glass spheres and silica gel was made, being a good alternative for improving the Bi_2WO_6 performance. Additionally, several reaction conditions of temperature and PR to O-2 feed ratio were studied.

Ionic liquid immobilization on carbon nanofibers and zeolites: Catalyst design for the liquid-phase toluene chlorination

Losch, Pit; Martinez Pascual, Antonio; Boltz, Marilyne; Ivanova, Svetlana; Louis, Benoit; Montilla, Francisco; Antonio Odriozola, Jose
Comptes Rendus Chimie, **18** (2015) 324-329
March, 2015 | **DOI:** 10.1016/j.crci.2014.06.006

The environmental-friendly chlorination reaction of toluene by trichloroisocyanuric acid (TCCA, $C_3N_3O_3Cl_3$) was investigated applying immobilized ionic liquids (ILs) on different supports. Ionic liquids were grafted either on carbon nanofibers (CNF) or encapsulated in zeolites. Their influence on the chlorination activity as well as on the selectivity in different chlorinated products was studied. An unusually high selectivity toward meta-chlorotoluene was achieved, up to 36%. Hence, the selectivity could be tuned to produce either expected ortho-/para-chlorotoluene or meta-chlorotoluene with a proper support choice.

Facile Synthesis of Decahedral Particles of Anatase TiO_2 with Exposed {001} Facets

Perales-Martinez, IA; Rodriguez-Gonzalez, V; Obregon-Alfaro, S; Lee, SW
Journal of Nanoscience and Nanotechnology, **15** (2015) 7351-7356
September, 2015 | **DOI:** 10.1166/jnn.2015.10578

This paper reports a facile synthesis of decahedral particles of anatase TiO_2 dominated by {101} and {001} faces. The decahedral particles has been enhanced by means a microwave-assisted hydrothermal method using TiF_4 as a titanium precursor and HF as capping agent to promote oriented growth and formation of {001} faces in only 4 h. The prepared samples were characterized by scanning electron microscopy, high resolution of transmission electron microscopy and X-ray diffraction. The morphology of anatase TiO_2 particles is consisted of near-perfect-truncated-bipyramid-shape. Reaction time is a key factor to obtain truncated-bipyramid-shaped particles with sharp and well-defined edges. Reaction times longer than 4 h induce irregular particles. Decahedral anatase TiO_2 particles are truncated bypiramid crystals which have eight {101} and two {001} facets at top/bottom surfaces. The average size of decahedral anatase TiO_2 particles are similar to 250 nm for the samples obtained without applying the microwave irradiation and similar to 350 nm for reaction 4 h.

Simultaneous Production of CH_4 and H-2 from Photocatalytic Reforming of Glucose Aqueous Solution on Sulfated Pd-TiO_2 Catalysts

Vaiano, V; Iervolino, G; Sarno, G; Sannino, D; Rizzo, L; Mesa, JJM; Hidalgo, MC; Navio, JA
Oil & Gas Science and Technology-Revue D IFP Energies Nouvelles, **70** (2015) 891-902
November, 2015 | **DOI:** 10.2516/ogst/2014062

In this work, the simultaneous production of CH_4 and H-2 from photocatalytic reforming of glucose aqueous solution on Pd-TiO_2 catalysts under UV light irradiation by Light-Emitting Diodes (LED) was investigated. The Pd-TiO_2 catalysts were prepared by the photodeposition method. The Pd content was in the range 0.5-2 wt% and a photodeposition time in the range 15-120 min was used. Pd-TiO_2 powders were extensively characterized by X-Ray Diffraction (XRD), SBET, X-Ray Fluorescence spectrometry (XRF), UV-Vis Diffuse Reflectance Spectra (UV-Vis DRS), TEM and X-Ray Photoelectron Spectroscopy (XPS). It was found that the lower Pd loading (0.5 wt%) and 120 min of photodeposition time allowed us to obtain homogeneously distributed metal nanoparticles of small size; it was also observed that the increase in the metal loading and deposition time led to increasing the Pd-0 species effectively deposited on the sulfated TiO_2 surface. Particle size and the oxidation state of the palladium were the main factors influencing the photocatalytic activity and selectivity. The presence of palladium on the sulfated titania surface enhanced the H-2 and CH_4 production. In fact, on the catalyst with 0.5 wt% Pd loading and 120 min of photodeposition time, H-2 production of about 26 lmol was obtained after 3 h of irradiation time, higher than that obtained with titania without Pd (about 8.5 lmol). The same result was obtained for the methane production. The initial pH of the solution strongly affected the selectivity of the system. In more acidic conditions, the production of H-2 was enhanced, while the CH_4 formation was higher under alkaline conditions.

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

12th European Congress on Catalysis | EuropaCat 2015

30 agosto – 4 septiembre [Kazan, Rusia]

Gold supported on Fe-doped ceria supports prepared through the microemulsion method for the PROX reaction
O.H. Laguna, M.A. Centeno, M.Boutonnet, J.A. Odriozola
Comunicación oral

Influence of the ionic liquid presence on the selective oxidation of glucose over molybdenum based catalysts
C.M. Sayago, C.J. Carrasco, S. Ivanova, F.J. Montilla Ramos, A. Galindo del Pozo, J.A. Odriozola
Comunicación oral

Selectivity control in oxidation of 1-tetradecanol on supported nano Au catalysts
S. Martínez-González, S. Ivanova, M.I. Domínguez, V. Cortés Corberán
Comunicación Oral

Biogas steam reforming for syngas production with structured catalyst
A. Alvarez M, L.M. Martínez, M.A. Centeno, J.A. Odriozola
Poster

Structured Pt(2%)/CeO_2/Al_2O_3 WGS catalyst design: introduction of buffer layer
M. González Castaño, S. Ivanova, M.A. Centeno, J.A. Odriozola
Poster

Pt, Rh and Pt-Rh-deposited on $MgAl_2O_4$ spinels catalysts for dry and steam reforming of CH_4
V. Garcilaso, M.A. Centeno, O.H. Laguna, J.A. Odriozola
Poster

3rd International Congress on Catalysis for Biorefineries | CatBior2015

27 – 30 septiembre [Rio de Janeiro, Brasil]

Gold catalysis screening in the selective aerobic oxidation of glucose
C. Megías-Sayago, S. Ivanova, M.A. Centeno, J.A. Odriozola
Comunicación oral

5th Asian Symposium on Advanced Materials: Chemistry, Physics & Biomedicine of Functional and Novel Materials

1 – 4 noviembre [Busan, República de Corea]

Clay-based nanostructured materials containing anatase and noble metal nanoparticles for photocatalytic hydrogen production
J. Pérez-Carvajal, P. Aranda, S. Obregón, G. Colón, E. Ruiz-Hitzky
Comunicación oral

Eleventh International Symposium on Heterogeneous Catalysis | EISHC XI

6 – 9 septiembre [Varna, Bulgaria]

Structured and microstructured catalytic reactors for hydrogen cleanup
J.A. Odriozola
Conferencia Plenaria

Polioxometalate/ionic liquids hybrids as a catalyst for the isomerisation of glucose to fructose in water
C. Megías-Sayago, S. Ivanova, M.A. Centeno, J.A. Odriozola
Comunicación oral

Gold modified hydrocalcite Cu/ZnO/Al_2O_3 catalysts for pure hydrogen production
J.L. Santos, T.R. Reina, I. Ivanov, S. Ivanova, T. Tabakova, M.A. Centeno, V. Idakiev, J.A. Odriozola
Poster

🟩 CONGRESOS Y REUNIONES NACIONALES / NATIONAL CONGRESSES AND MEETINGS

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

Catálisis, Confluencia Interdisciplinar: Modelos, Catalizadores y Reactores SECAT'15

13 – 15 julio [Barcelona, España]

José Antonio Odriozola Gordón [Miembro del Comité Científico)
Miguel Angel Centeno Gallego [Miembro del Comité Científico)

COMUNICACIONES / COMMUNICATIONS

Catálisis, Confluencia Interdisciplinar: Modelos, Catalizadores y Reactores SECAT'15

13 – 15 julio [Barcelona, España]

Diseño y caracterización de catalizadores basados en oro para la reacción de water gas shift
T. Ramírez Reina
Conferencia invitada

Uso de catalizadores estructurados en la producción de gas de síntesis a partir de biogas
A. Alvarez, L.M. Martínez, M.A. Centeno, J.A. Odriozola
Comunicación oral

Oxidación selectiva de glucosa en presencia de O_2 sobre catalizadores de oro soportado
C. Megías-Sayago, S. Ivanova, M.A. Centeno, J.A. Odriozola
Comunicación oral

Catalizadores Pt/Na/C para la reacción de wáter gas shift
J.L. Santos, M.A. Centeno, J.A. Odriozola
Comunicación oral

Sistemas catalíticos Ru/Co/$CexZr_{1-x}O_2/Al_2O_3$ basados en cobalto para el reformado de CH_4 con vapor: efecto de la modificación del soporte y la introducción de rutenio
V. Garcilaso de la Vega, O.H. Laguna, M.A. Centeno, J.A. Odriozola
Comunicación oral

Óxidos mixtos basados en zirconio como conductores iónicos
N. García Moncada, R. Poyato Galán, F. Romero Sarria, M.A. Centeno Gallego, J.A. Odriozola Gordón
Poster

Intensificación de la producción de hidrógeno por reformado de metanol
O. Sanz, I. Velasco, I. Pérez-Miqueo, G. Arzamendi, L.M. Gandia, J.A. Odriozola, M. Montes
Poster

Catalizadores de Pt, Rh y Pt-Rh, soportados sobre espinela $MgAl_2O_4$ para las reacciones de reformado húmedo y reformado seco de CH_4
V. Garcilaso de la Vega, O.H. Laguna, M.A. Centeno, J.A. Odriozola
Poster

Efecto del estado de oxidación del manganeso en la oxidación prefencial de CO en presencia de hidrógeno
L.M. Martínez T., O.H. Laguna, M.A. Centeno, J.A. Odriozola
Poster

Primera etapa de la síntesis de Fischer-Tropsch sobre un catalizador modelo: Ru/Al_2O_3
E.M. Jiménez-Barrera, P. Bazin, F. Romero-Sarria, M. Daturi, J.A. Odriozola
Poster

Efecto promotor del O_2 sobre catalizadores estructurados en WGS: Pt vs. Au
M. González Castaño, T.R. Reina, S. Ivanova, L.M. Martínez, M.A. Centeno, J.A. Odriozola
Poster

XIV Congreso Nacional de Tratamientos Térmicos y de Superficie | TRATERMAT 2015

30 septiembre – 1 octubre [Pontevedra, España]

Preparación de membranas de SAPO-34 soportadas sobre mallas metálicas
M. Romero, M.I. Domínguez, R. Poyato, J.A. Odriozola
Poster

Soldadura por difusión de microreactores metálicos de aceros inoxidables ferríticos
J. Pahissa, L.M. Martínez T., M.A. Centeno, J.A. Odriozola
Poster

FORMACION / TRAINING

TESIS DOCTORALES/ DOCTOR DEGREE THESIS

Título: **Reformado catalítico de biomasa. Ajuste de la estequiometría de los productos para la reacción de Fischer Tropsch**
Autor: Andrea Alvarez Moreno
Directores: José Antonio Odriozola Gordón, Miguel Angel Centeno Gallego
Calificación: Sobresaliente “Cum Laude”
Centro: Universidad de Sevilla
Fecha: 13 de febrero de 2015

Título: **Obtención de zeolitas utilizando líquidos iónicos como agentes directores de estructura**
Autor: José María Martínez Blanes
Directores: Svetlana Lybomirova Ivanova, José Antonio Odriozola Gordón
Calificación: Sobresaliente “Cum Laude”
Centro: Universidad de Sevilla
Fecha: 2 de diciembre de 2015

Título: **Design, characterization and structuring of Pt based catalysts for WGS reaction**
Autor: Miriam González Castaño
Directores: José Antonio Odriozola Gordón, Svetlana Lybomirova Ivanova
Calificación: Sobresaliente “Cum Laude”
Centro: Universidad de Sevilla
Fecha: 10 de diciembre de 2015

FORMACIÓN DE GRADUADOS / MASTER DEGREE THESIS

Título: **Soldadura por difusión de Microreactores metálicos de aceros inoxidables ferríticos**
Autor: Javier Pehissa García
Directores: Leidy Marcela Martínez Tejada
Grado: Trabajo Fin de Grado
Centro: Universidad de Sevilla
Año Académico: 2014-2015 (19 febrero 2015)

Título: **Síntesis y caracterización de catalizadores basados en Hidroxiapatitos con Wolframio**
Autor: Juan León Ramos
Directores: Miguel Angel Centeno Gallego, María Isabel Domínguez Leal
Grado: Trabajo Fin de Grado
Centro: Universidad de Sevilla
Año Académico: 2014-2015 (15 julio 2015)

Título: **Diseño de conductores iónicos como soporte de catalizadores de WGS**
Autor: Alba María Fernández Sotillo
Directores: José Antonio Odriozola Gordón, María Isabel Dominguez Leal
Grado: Trabajo Fin de Master
Centro: Universidad de Sevilla
Año Académico: 2014-2015 (19 febrero 2015)

Título: **Influencia de la concentración de ácido acético en la preparación de catalizadores Pt/Al_2O_3 para la reacción PROX**
Autor: Sergio García Dalí
Directores: Svetlana Lyubomirova Ivanova, Francisco Romero Sarria
Grado: Trabajo Fin de Master
Centro: Universidad de Sevilla
Año Académico: 2014-2015 (2 julio 2015)

Título: **Nanoparticulas de rutenio como catalizadores para metanación de CO y CO_2**
Autor: Sara Navarro Jaén
Directores: Oscar H. Laguna Espitia, José Antonio Odriozola Gordón
Grado: Trabajo Fin de Master
Centro: Universidad de Sevilla
Año Académico: 2014-2015 (2 julio 2015)

Título: **Tipos de sitios superficiales en catalizadores de óxido de molibdeno (VI) para la síntesis de biodiesel**
Autor: María Domínguez Gómez
Directores: Francisca Romero Sarria
Grado: Trabajo Fin de Grado
Centro: Universidad de Sevilla
Año Académico: 2014-2015 (15 julio 2015)

Título: **Estudio de la estructuración de un catalizador Pt/Al_2O_3 en micro-monolitos metálicos para procesos de combustión**
Autor: María Rodríguez Rodríguez
Directores: Oscar H. Laguna Espitia, José Antonio Odriozola Gordón
Grado: Trabajo Fin de Master
Centro: Universidad de Sevilla
Año Académico: 2014-2015 (3 agosto 2015)

Título: **Modificación de la química superficial de un coque y su aplicación como soporte de catalizadores en la licuefacción directa de carbón**
Autor: Diego Arcelio Rico Siera
Directores: Miguel Angel Centeno, Yazmin Yaneth Agámez Pertuz
Grado: Trabajo de Tesis de Maestría de la Facultad de Ciencias. Dpto. Química
Centro: Universidad Nacional de Colombia, Sede Bogotá
Año Académico: 2014-2015 (3 agosto 2015)

Docencia / Teaching

Centro de investigación y extensión de la facultad de Ciencias - U. Pedagógica y Tecnológica de Colombia
Procesos de reformado: diseño y aplicacion tecnológica de soluciones catalíticas
Dr. Oscar Hernando Laguna Espitia
Lugar: Universidad Pedagógica y Tecnológica de Colombia,Tunja (Colombia), 13 abril 2015

XIII Escuela de Ciencia e Ingeniería de Materiales
Dr. José Antonio Odriozola Gordón
Lugar: Instituto de Materiales de la Universidad Nacional Autónoma de México (UNAM), México, 29 Junio-3 Julio 2015 (10 h)

Premios y Reconocimientos / Prizes and Acknowledgements

Tomás Ramirez Reina. “Diseño y caracterización de catalizadores basados en oro para la reacción de Water Gas Shift“ **Premio a la mejor Tesis Doctoral en Catálisis 2014**. Sociedad Española de Catálisis, SECAT. 2015

Journal of Catalysis. Editor-in-Chief’s Featured Article. "H2 oxidation as criterion for PrOx catalyst selection: Examples based on Au–CoOx-supported systems” Volumen 326, Junio 2015, 161-171. Tomás Ramírez Reina, Cristina Megías-Sayago, Alejandro Pérez Florez, Svetlana Ivanova, Miguel Ángel Centeno, José Antonio Odriozola

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

KTH		
Estocolmo, Suecia	**Victoria Garcilaso de la Vega**	30-03-2015 / 03-07-2015

Estancias y Visitas de Investigadores en el ICMS
Personnel of the Other Laboratories in the ICMS

Universidad Pedagógica y Tecnológica de Colombia (UPTC)		
Tunja, Colombia	**Julie Mesa**	06-03-2015 / 04-04-2015
	Hugo Rojas Sarmiento	21-03-2015 / 04-04-2015
Université d’Alger		
Alger, Algeria	**Meriem Chebout**	01-10-2015 / 30-10-2016
	Salima Rahou	18-09-2015 / 15-12-2015
Universidad Nacional		
Bogotá, Colombia	**Nicolás Rodríguez Riaño**	23-09-2015 / 22-09-2016
	Hugo Rojas Sarmiento	21-03-2015 / 04-04-2015

EQUIPAMIENTO CIENTÍFICO AVAILABLE EQUIPMENT

- Molino de bolas de movimiento planetario PM100 de RETSCH
- Espectrofotómetro Infrarrojos: Thermo-Nicolet Nexus FT-IR; Thermo-Nicolet 380 FT-IR
- Accesorio DRIFTS, celdas de alta y baja temperatura
- Sistema de vacío cuarzo/vidrio para adsorción de moléculas sonda seguido por FT-IR
- Espectrómetro de masas: Balzers Thermostar
- Sistema TPR-TPO (con posibilidad de realizar pulsos) seguido de espectrómetro de masas (Balzers) y detector de conductividad térmica. PID Eng&Tech.
- Equipos comerciales de actividad catalítica Microactivity Reference PID Eng&Tech (4)
- Microscopio metalográfico: Leica DMIRM
- Rotavapor: Heidolph Hei-VAP Value
- Equipo de ultra-alto vacío para espectroscopía XPS y Auger equipado con cañón de argón para realizar devastado iónico.
- Estufas: P-Selecta; P-Selecta digiheat
- Ph-metros: Crison pH burette 24; Crison phMeter Basic 20; Crison micropH2000.
- Cromatógrafos (2): Agilent Technologies 7890 A GC System; Agilent Technologies 6890 N Network GC System.
- Micro-cromatógragos, microGC (2): Micro Gas Cromatograph CP-4900 Varian (2)
- HPLC: Varian 356-LC, Solvent Delivery Module Varian ProStar.
- Horno de soldadura: Microtest Máquina de ensayos EM2/200/FR
- Baño de ultrasonidos: P-Selecta Ultrasons Medi-II
- Horno Energon
- Horno para tratamiento de aceros a alta temperatura equipado con medidores de flujo e inyector de agua.
- Planta Piloto de Integración de reacciones catalíticas Reference PID Eng&Tech
- Espectrómetro Uv-Vis (Varian Cary 100, con esfera integradora para
- muestras sólidas)
- Analizador de Carbón Orgánico (TOC-V CHP Shimadzu 5000A)
- Cromatógrafo (HPLC Agilent Technologies 1200)
- Espectrómetro IR (Varian 660-IR FTIR Spectrometer)
- Dip-Coater con cámara de temperatura (SS-00 AB Table Dry Oven MTI
- Corporation)
- Espectrofotómetro FTIR con celdas DRIFTS y ATR.
- Sistema de análisis TPR/TPO con detector TCD y espectrómetro de masas.
- 6 reactores catalíticos de gases con detección por cromatografía de gases y espectrometría de masas.
- 2 reactores catalíticos de líquidos que permiten el seguimiento de hasta 8 reacciones de forma simultánea con control de temperatura y flujo de gases.
- 4 reactores catalíticos de líquidos de alta presión y temperatura con agitación interna y control de flujo de gases.
- Reactores fotocatalíticos con lámparas de Xe y Hg.
- Espectrofotómetros FTIR con accesorios DRIFTS, ATR y sistema de vacío en cuarzo/vidrio para adsorción de moléculas sonda

- Espectrómetros de masas
- Sistemas TPR-TPO (con posibilidad de realizar pulsos) seguido de espectrómetro de masas y detector de conductividad térmica.
- Microscopio metalográfico
- Rotavapor
- Estufas
- Ph-metros
- Cromatógrafos de Gases
- Micro-cromatógragos, microGC
- Cromatografos HPLC
- Horno de soldadura: Microtest Máquina de ensayos EM2/200/FR
- Baño de ultrasonidos
- Hornos
- Horno para tratamiento de aceros a alta temperatura equipado con medidores de flujo e inyector de agua.
- Planta Piloto de Integración de reacciones catalíticas Reference PID Eng&Tech
- Espectrómetro Uv-Vis, con esfera integradora para muestras sólidas)
- Analizador de Carbón Orgánico
- Dip-Coater con cámara de temperatura
- Reactores catalíticos de gases con detección por cromatografía de gases y espectrometría de masas.
- Reactores catalíticos de líquidos que permiten el seguimiento de hasta 8 reacciones de forma simultánea con control de temperatura y flujo de gases.
- Reactores catalíticos de líquidos de alta presión y temperatura con agitación interna y control de flujo de gases.
- Reactores fotocatalíticos con lámparas de Xe y Hg.

INGENIERÍA DE CERÁMICOS PARA AMBIENTES EXTREMOS

ENGINEERED CERAMICS FOR EXTREME ENVIRONMENTS

PERSONAL / PERSONNEL

Catedráticos

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

Científicos Titulares

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

Profesores Titulares

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

Investigadores Contratados

Dr. Miguel Antonio Cruz Carrillo
Dra. Esperanza Pavón González

Profesor Contratado Doctor

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

Becarios Predoctorales

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

Personal Contratado

Dra. Paula M. Castillo Hernández
Lda. Aurora Gómez Marín

PROYECTOS DE INVESTIGACIÓN / RESEARCH PROJECTS

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

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

RESUMEN / ABSTRACT

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

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

Se trabajará en las siguientes líneas:

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

- Diseño de la unidad piloto y estudio de la integración y operación sobre el motor de referencia.
- Diseño final del filtro para su instalación industrial.

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

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

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

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

The following research lines will be addressed:

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

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

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

Unión Europea
Fondo Europeo de Desarrollo Regional
"Una manera de hacer Europa"
JUNTA DE ANDALUCIA

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

Código/Code:	P11-TEP-7418 (Proyecto de Excelencia)
Periodo/Period:	16-05-2013 / 15-05-2015
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	174.455 €
Investigador responsable/Research head:	José Jesús Benítez Jiménez
Componentes/Research group:	Antonio Heredia Bayona, Miguel Angel San Miguel Barrera, Jaime Oviedo López, J. Alejandro Heredia Guerrero, Santiago Domínguez Meister, Daniel Aguilera Puerto, Francisco Javier Navas Martos, José Manuel de la Torre Ramírez

RESUMEN / ABSTRACT

El presente proyecto de investigación tiene por objeto el estudio de la viabilidad de la implementación de un proceso a escala planta piloto que permita obtener un material bioplástico a partir de una materia prima asequible y de bajo coste como son los desechos de pieles (epidermis) de frutos. La oportunidad de la propuesta parte de proponer un nuevo tipo de material polimérico completamente inocuo, biodegradable y ecológico como sustituto de plásticos tradicionales obtenidos a partir del petróleo que conllevan un serio problema medioambiental, tanto en su producción industrial como en su posterior desecho. Por otro lado, el concepto de sostenibilidad medioambiental se extiende no sólo a la biodegradabilidad del producto final y al bajo impacto del proceso de producción propuesto, también a su obtención a partir de un recurso vegetal que no se retrae de la cadena alimenticia animal y humana, como es el caso de los

bioplásticos qu<e se vienen fabricando actualmente a partir de maíz o patata. Por otro lado, y en nuestro ámbito territorial andaluz, la materia prima es especialmente accesible dado el volumen de la actividad agroalimentaria. En este mismo sentido, el nuevo bioplástico podría paliar un importante problema de eliminación de residuos plásticos en explotaciones agrarias ya que se concibe como plenamente compostable y, por tanto, capaz de generar biomasa asimilable por la siguiente generación de plantas. El producto propuesto viene, además, a completar la gama de polihidroxialcanoatos (PHA) a los miembros de la serie obtenidos a partir de polihidroxiácidos de cadena larga. Las propiedades singulares de esta nueva familia puede ser un buen complemento de los ya conocidos y podría dar lugar a aplicaciones novedosas y de mayor valor añadido.

The main objective of this project is to evaluate the feasibility of scaling up a procedure to obtain fatty polyhydroxyalcanoate (PHA) bioplastics from a low-cost and abundant source like peels residues of commercial fruits. The strength of the proposal relies on the introduction of a new non-toxic and fully biodegradable polymeric material as a substitute for environmental-hostile petroleum-based plastics. The overall sustainability is extended to the use of a low-impact synthetic route and to the processing of a plant residue rather than crops intended for human or cattle feeding. The project is considered of additional interest in regions with an agricultural based economy like Andalusia and with an important environmental impact arising from the greenhouse activity. The proposal also covers the study of new and more specific applications of such bio-based fatty polyhydroxyalcanoates.

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 medioambiental. 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.

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

Unión Europea
Fondo Europeo de Desarrollo Regional
"Una manera de hacer Europa"
JUNTA DE ANDALUCIA

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

RESUMEN / ABSTRACT

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

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

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

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

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

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

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

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

Effect of catalytic graphitization on the electrochemical behavior of wood derived carbons for use in supercapacitors

Gutierrez-Pardo, A; Ramirez-Rico, J; Cabezas-Rodriguez, R; Martinez-Fernandez, J
Journal of Power Sources, **278** (2015) 18-26
March, 2015 | **DOI:** 10.1016/j.jpowsour.2014.12.030

Porous graphitic carbons were successfully obtained from wood precursors through pyrolysis using a transition metal as catalyst. Once the catalyst is removed, the resulting material mimics the microstructure of the wood and presents high surface area, open and interconnected porosity and large pore volume, high crystallinity and good electrical conductivity, making these carbons interesting for electrochemical devices. Carbons obtained were studied as electrodes for supercapacitors in half cell experiments, obtaining high capacitance values in a basic media (up to 133 F g−1 at current densities of 20 mA g−1 and 35 F g−1 at current densities of 1 A g−1). Long-cycling experiments showed excellent stability of the electrodes with no reduction of the initial capacitance values after 1000 cycles in voltammetry.

Uranium immobilization by FEBEX bentonite and steel barriers in hydrothermal conditions

Villa-Alfageme, M; Hurtado, S; El Mrabet, S; Pazos, MC; Castro, MA; Alba, MD
Chemical Engineering Journal, **269** (2015) 279-287
June, 2015 | **DOI:** 10.1016/j.cej.2015.01.134

FEBEX clay is considered a reference material in engineered barriers for safe storage of nuclear waste and uranium is a minor component of high-level radioactive waste (HLRW) and a main component of the spent nuclear fuel (SNF). Here, the kinetics of reaction of uranium with FEBEX was investigated in addition to the uranium immobilisation ability and the structural analysis of the reaction products. Hydrothermal treatments were accomplished with UO22+ and tetravalent actinide simulator ZrO2+, also present in HLRW. The quantification of the reaction was performed through gamma spectrometry of uranium. Two mechanisms for UO22+ retention by FEBEX were detected: adsorption and formation of stable and insoluble new phases. The structural analyses performed using ZrO2+, confirmed the uranium adsorption and the presence of new phases, ZrO2 and Zr(SiO4), that emphasise the existence of a chemical reaction with the bentonite. The analysis of the velocity of reaction uranium-clay minerals revealed temperature dependence. An exponential fitting suggested that the removal of uranium from solution at temperatures over 200 °C could be completed in less than a year. For lower temperatures, several years are needed. Milliequivalents of UO22+ immobilised by the clay depended on temperature and time and were over cation exchange capacity (CEC) of FEBEX even at 100 °C (reaching 600% of CEC). The reaction with steel, also temperature dependent, was finally analysed. At 200 °C 40–70% of uranium reacted with steel. But only 30–15% reacted at 300 °C and 100 °C. The reactions provide a stable immobilisation mechanism for uranium even when its sorption and swelling capacities fail. Our experiments will be of particular interest for very deep borehole disposals were higher temperatures and pressures are expected.

Self-Assembling of Tetradecylammonium Chain on Swelling High Charge Micas (Na-Mica-3 and Na-Mica-2): Effect of Alkylammonium Concentration and Mica Layer Charge

Pazos, MC; Cota, A; Osuna, FJ; Pavon, E; Alba, MD
Langmuir, **31** (2015) 4394-4401
April, 2015 | **DOI:** 10.1021/acs.langmuir.5b00224

A family of tetradecylammonium micas is synthesized using synthetic swelling micas with high layer charge (NanSi8-nAln,Mg6F4O20 center dot XH2O, where n = 2 and 3) exchanged with tetradecylammonium cations. The molecular arrangement of the surfactant is,elucidated on the basis of XRD patterns and DTA. The ordering conformation of the surfactant molecules into the interlayer space of Micas is investigated by IR/FT, C-13, Al-27, and Si-29 MAS NMR. The structural arrangement of the tetradecylammonium cation in the interlayer space of high-charge micas is more sensitive to the effect of the mica layer charge at high concentration. The surfactant arrangement is found to follow the bilayer-paraffin model for all values of layer charge and surfactant concentration. However, at initial concentration below the mica CEC, a lateral monolayer is also observed. The amount of ordered conformation all-trans is directly proportional to the layer charge and surfactant concentration.

Synthesis temperature effect on Na-Mica-4 crystallinity and heteroatom distribution

Naranjo, M; Castro, MA; Cota, A; Osuna, FJ; Pavon, E; Alba, MD
Microporous and Mesoporous Materials, **204** (2015) 282-288
March, 2015 | **DOI:** 10.1016/j.micromeso.2014.11.026

The discovery of swelling brittle mica, Na-Mica-4, has been one of the most significant advances in the pursuit for a material with high ion-exchange capacity. For technical applications, the control of the phase evolution during the synthesis is crucial. The main aim of this study was to investigate the effect of Na-Mica-4 synthesis temperature on the crystalline phase evolution, Si–

Al distribution in the tetrahedral sheet, the Al occupancy between tetrahedral and octahedral sites and their effects on the interlayer space composition. The synthesis temperature range between 600 °C and 900 °C was explored. At low temperature (600 °C), the precursors were transformed in a low-charged swelling 2:1 phyllosilicate, saponite type, which was progressively aluminum enriched with temperature. The high-charged swelling mica was completely formed at 700 °C, although a minor anhydrous contribution remained up to 850 °C. Up to 800 °C, silicates and fluorides secondary phases were detected as a minor contribution.

Pectin-Lipid Self-Assembly: Influence on the Formation of Polyhydroxy Fatty Acids Nanoparticles

Guzman-Puyol, Susana; Jesus Benitez, Jose; Dominguez, Eva; Bayer, Ilker Sefik; Cingolani, Roberto; Athanassiou, Athanassia; Heredia, Antonio; Heredia-Guerrero, Jose Alejandro
Plos One, **10** (2015) e0124639
April, 2015 | **DOI:** 10.1371/journal.pone.0124639

Nanoparticles, named cutinsomes, have been prepared from aleuritic (9,10,16-trihidroxipalmitic) acid and tomato fruit cutin monomers (a mixture of mainly 9(10), 16-dihydroxypalmitic acid (85%, w/w) and 16-hydroxyhexadecanoic acid (7.5%, w/w)) with pectin in aqueous solution. The process of formation of the nanoparticles of aleuritic acid plus pectin has been monitored by UV-Vis spectrophotometry, while their chemical and morphological characterization was analyzed by ATR-FTIR, TEM, and non-contact AFM. The structure of these nanoparticles can be described as a lipid core with a pectin shell. Pectin facilitated the formation of nanoparticles, by inducing their aggregation in branched chains and favoring the condensation between lipid monomers. Also, pectin determined the self-assembly of cutinsomes on highly ordered pyrolytic graphite (HOPG) surfaces, causing their opening and forming interconnected structures. In the case of cutin monomers, the nanoparticles are fused, and the condensation of the hydroxy fatty acids is strongly affected by the presence of the polysaccharide. The interaction of pectin with polyhydroxylated fatty acids could be related to an initial step in the formation of the plant biopolyester cutin.

Impact of hydrothermal treatment of FEBEX and MX80 bentonites in water, HNO_3 and $Lu(NO_3)(3)$ media: Implications for radioactive waste control

Osuna, FJ; Chain, P; Cota, A; Pavon, E; Alba, MD
Applied Clay Science, **118** (2015) 48-55
December, 2015 | **DOI:** 10.1016/j.clay.2015.08.036

Engineered barriers of deep geological repositories (DGR) are commonly constructed with bentonite. FEBEX and MX80 bentonites have been selected by different countries as reference materials for the sealing of repositories; however, their chemical reactivity with high-level long-lived radioactive wastes (HLRW) under subcritical conditions had not been explored before. The hydrothermal stability in neutral and acid media and chemical reactivity in contact with an actinide analogous compound were both studied. The long-range and short-range structural changes were analyzed by X-ray diffraction, nuclear magnetic resonance and scanning electron microscopy. Both bentonites have exhibited a good stability in neutral and acid media and have generated a new phase immobilizing the actinide analogous compound. The extent of the chemical reaction is higher in MX80 bentonite than in FEBEX bentonite.

Viability of adding gypsum and calcite for remediation of metal-contaminated soil: laboratory and pilot plant scales

Gonzalez-Nunez, R; Alba, MD; Vidal, M; Rigol, A
International Journal of Environmental Science and Technology, **12** (2015) 2697-2710
August, 2015 | **DOI:** 10.1007/s13762-014-0671-3

The effect of adding waste materials (gypsum and calcite) for the remediation of a soil contaminated by pyritic minerals was examined. Materials were characterised in terms of their acid neutralisation capacity (ANC), sorption capacity and structural components. Their effect on the contaminant leaching in soil + material mixtures over a wide range of pH was also evaluated. Results at laboratory and pilot plant scales were compared to account for the potential variability in the material efficiency when applied at larger scale. The use of gypsum permitted its valorisation, although calcite was a more effective amendment because its addition led to a greater increase in the pH and acid neutralisation capacity, and thus in the sorption capacity in the resulting soil + material mixture. In the same way, when the combination of gypsum + calcite was added to the soil, it led to an increase in the pH from 2.5 to 6.9 and in the ANC from −86 to 1,513 meq/kg. As a result, the concentration of extractable heavy metals and As was reduced, and they were successfully immobilised both at laboratory and at pilot plant scales. Thus, the use of these materials induced a significant reduction in the contaminant mobility and permitted the valorisation of waste materials.

Sliding wear resistance of biomorphic SiC ceramics

Vera, MC; Ramirez-Rico, J; Martinez-Fernandez, J; Singh, M
International Journal of Refractory Metals & Hard Materials, **49** (2015) 327-333
March, 2015 | **DOI:** 10.1016/j.ijrmhm.2014.07.004

Biomorphic SiC ceramics were fabricated from four different wood precursors and their Knoop hardness and sliding wear resistance when sliding against a Si_3N_4 ball in air were studied. Tribological experiments were performed using a pin on disk apparatus, under normal loads of 2 and 5 N, at a sliding speed of 100 mm/s. The effects of specimen porosity and microstructure on measured wear were evaluated. A commercial sintered silicon carbide ceramic was also tested for comparison. Small differences in friction coefficient comparable to monolithic SiC ceramics were obtained. Several concurrent wear mechanisms are taking place: microfracture, plastic deformation in the Si phase and oxidation of the Si and/or SiC phase. The presence of an oxide tribolayer was assessed using fluorescence microscopy. Wear rates were found to scale with SiC content and depend on residual porosity in the composite.

Sliding wear resistance of sintered SiC-fiber bonded ceramics

Vera, MC; Ramirez-Rico, J; Martinez-Fernandez, J; Singh, M
International Journal of Refractory Metals & Hard Materials, **49** (2015) 232-239
March, 2015 | **DOI:** 10.1016/j.ijrmhm.2014.06.020

Advanced SiC-based ceramics and fiber reinforced composites are interesting materials for a wide variety of applications involving sliding wear conditions because of their excellent thermomechanical properties. The microstructure and wear resistance of sintered SiC fiber bonded ceramics (SA Tyrannohex) were studied. The material is composed of SiC-fibers in two orientations, with polygonal cross sections and cores having higher carbon content than their surroundings, as observed with SEM. A thin layer of C exists between the fibers. This layer has been found to be a turbostratic-layered structure oriented parallel to the fiber surface. XRD shows that the material is highly crystalline and composed mostly of β-SiC. Unlubricated wear behavior of the SA-Tyrannohex material when sliding against a Si_3N_4 ball in air at room temperature was evaluated. Experiments were performed using a pin on disk apparatus, under different normal loads of 2, 5 and 10 N at sliding speeds of 25, 50, 100 mm/s. A decrease of the friction coefficient with load was found due to the presence of the turbostratic carbon layer between the fibers. Wear rates of the order of 100 mm3/MJ were obtained, independently of sliding speed. Microfracture of the fibers is the main wear mechanism.

Polyester Films Obtained by Noncatalyzed Melt-Condensation Polymerization of Aleuritic (9,10,16-Trihydroxyhexadecanoic) Acid in Air

Benitez, JJ; Heredia-Guerrero, JA; Guzman-Puyol, S; Dominguez, E; Heredia, A
Journal of Applied Polymer, **132** (2015) art. 41328
January, 2015 | **DOI:** 10.1002/app.41328

To mimic nontoxic and fully biodegradable biopolymers like the plant cutin, polyester films from a natural occurring fatty polyhydroxyacid like aleuritic (9,10,16-trihydroxyhexadecanoic) acid have been prepared by noncatalyzed melt-polycondensation at moderate temperature (150 degrees C) directly in air. The course of the reaction has been followed by infrared spectroscopy, C-13 magic angle spinning nuclear magnetic resonance spectroscopy, differential scanning calorimetry and X-ray diffraction and well differentiated stages are observed. First, a high conversion esterification reaction leads to an amorphous rubbery, infusible, and insoluble material whose structure is made out of ester linkages mostly involving primary hydroxyls and partially branched by minor esterification with secondary ones. Following the esterification stage, the cleavage of vicinal secondary hydroxyls and further oxidation to carboxylic acid is observed at the near surface region of films. New carboxylic groups created also undergo esterification and generate cross-linking points within the polymer structure. Additionally, and despite the harsh preparation conditions used, very little additional side reaction like peroxidation and dehydration are observed. Results demonstrate the feasibility of polyester films fabrication from a reference fatty polyhydroxyacid like aleuritic acid by noncatalyzed melt-polycondensation directly in air. The methodology can potentially be extended to similar natural occurring hydroxyacids to obtain films and coatings to be used, for instance, as nontoxic and biodegradable food packaging material.

Long-Chain Polyhydroxyesters from Natural Occurring Aleuritic Acid as Potential Material for Food Packaging

Benitez, JJ; Heredia-Guerrero, JA; Guzman-Puyol, S; Dominguez, E; Heredia, A
Soft Materials, **13** (2015) 5-11
January, 2015 | **DOI:** 10.1080/1539445X.2014.993476

Fatty polyhydroxyesters ($C \geq 16$) are present in nature as barrier polymers like cutin in some protective tissues of higher plants. The mimicry of these biopolymers is regarded as a strategy to design nontoxic and fully biodegradable food packaging films and coatings. To obtain cutin inspired materials we have used a natural occurring polyhydroxylated monomer like aleuritic (9,10,16-trihydroxypalmitic) acid and a direct and scalable synthesis route consisting in the noncatalyzed melt-condensation polymerization in air. To reduce the number of hydroxyl groups and to increase hydrophobicity, palmitic acid has been used as a capping agent. Aleuritic-palmitic polyhydroxyesteres films have been obtained and characterized.

Specific features of the electrical properties in partially graphitized porous bio-carbons of beech wood

Popov, VV; Orlova, TS; Gutierrez-Pardo, A; Ramirez-Rico, J

Physics of the Solid State, **57** (2015) 1746-1751

September, 2015 | **DOI:** 10.1134/S1063783415090280

The electrical and galvanomagnetic properties of partially graphitized highly porous bioC(Ni) biocarbon matrices produced by pyrolysis (carbonization) of beech wood at temperatures T (carb) = 850-1600A degrees C in the presence of a Ni-containing catalyst have been studied in comparison with their microstructural features. The temperature dependences of the resistivity, the magnetoresistance, and the Hall coefficient have been measured in the temperature range of 4.2-300 K in magnetic fields to 28 kOe. It has been shown that an additional graphite phase introduction into samples with T (carb) a parts per thousand yen 1000A degrees C results in an increase in the carrier mobility by a factor of 2-3, whereas the carrier (hole) concentration remains within similar to 10(20) cm(-3), as in biocarbons obtained without catalyst. An analysis of experimental data has demonstrated that the features of the conductivity and magnetoresistance of these samples are described by quantum corrections related to their structural features, i.e., the formation of a globular graphite phase of nano- and submicrometer sizes in the amorphous matrix. The quantum corrections to the conductivity decrease with increasing carbonization temperature, which indicates an increase in the degree of structure ordering and is in good agreement with microstructural data.

Microstructure, elastic and inelastic properties of partially graphitized biomorphic carbons

Orlova, TS; Kardashev, BK; Smirnov, BI; Gutierrez-Pardo, A; Ramirez-Rico, J; Martinez-Fernandez, J

Physics of the Solis State, **57** (2015) 586-591

March, 2015 | **DOI:** 10.1134/S106378341503018X

The microstructural characteristics and amplitude dependences of the Young's modulus E and internal friction (logarithmic decrement delta) of biocarbon matrices prepared by beech wood carbonization at temperatures T (carb) = 850-1600A degrees C in the presence of a nickel-containing catalyst have been studied. Using X-ray diffraction and electron microscopy, it has been shown that the use of a nickel catalyst during carbonization results in a partial graphitization of biocarbons at T (carb) a parts per thousand yen 1000A degrees C: the graphite phase is formed as 50- to 100-nm globules at T (carb) = 1000A degrees C and as 0.5- to 3.0-mu m globules at T (carb) = 1600A degrees C. It has been found that the measured dependences E(T (carb)) and delta(T (carb)) contain three characteristic ranges of variations in the Young's modulus and logarithmic decrement with a change in the carbonization temperature: E increases and delta decreases in the ranges T (carb) < 1000A degrees C and T (carb) > 1300A degrees C; in the range 1000 < T (carb) < 1300A degrees C, E sharply decreases and delta increases. The observed behavior of E(T (carb)) and delta(T (carb)) for biocarbons carbonized in the presence of nickel correlates with the evolution of their microstructure. The largest values of E are obtained for samples with T (carb) = 1000 and 1600A degrees C. However, the samples with T (carb) = 1600A degrees C exhibit a higher susceptibility to microplasticity due to the presence of a globular graphite phase that is significantly larger in size and total volume.

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

1st International Caparica Conference on Pollutant Toxic Ions and Molecules | PTIM2015
2 – 4 noviembre [Caparica, Portugal]

Theoretical and experimental approach of the Zr1-xHfxSiO4 study: A stable matrix for radio-active waste immobilization
A. Cota, B.P. Burton, F.J. Osuna, E. Pavon, M.D. Alba
Poster

CONGRESOS Y REUNIONES NACIONALES / NATIONAL CONGRESS AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

XXIV Reunión de la Sociedad Española de Arcillas
5 – 7 septiembre [Madrid, España]

Micas de diseño para la adsorción de metales pesados
F.J. Osuna, A. Cota, E. Pavon, M.D. Alba
Poster

Formacion / Training

Tesis Doctorales/ Doctor Degree Thesis

Título: **Estrategia de Optimización de Dispositivos Fotovoltaicos de Lámina Delgada desde la Microestructura a la Macroestructura**
Autor: José María Delgado Sánchez
Directores: María Dolores Alba Carranza
Calificación: Sobresaliente “Cum Laude” por Unanimidad
Centro: Universidad de Sevilla
Fecha: 18 de febrero de 2015

Docencia / Teaching

II International NMR Training Course: Industrial and Socioeconomic Applications of Solid State NMR
Ceramic hosts for inmmobilization of hazardous waste: study by solid state NMR
Dra. M. Dolores Alba Carranza
Lugar: Curso de Especialización del CSIC. Instituto de Cerámica y Vidrio

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

Proyectos de Investigación / Research Projects

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.

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

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 / 31-12-2017
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, modificando principalmente la microestructura y la composición química de las fases cerámicas em-

pleadas. 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.

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

Diseño por procedimientos mecanoquímicos de materiales estructurales para aplicaciones tecnológicas de alta temperatura Mechanochemical desing of structural materials for high-temperature technological applications

Código/Code:	MAT2011-22981
Periodo/Period:	01-01-2012 / 30-06-2015
Organismo Financiador/Financial source:	Ministerio de Ciencia e Innovación
Importe total/Total amount:	80.000,36 €
Investigador responsable/Research head:	Francisco José Gotor Martínez
Componentes/Research group:	M. Jesús Sayagués de Vega, Concepción Real Pérez, M. Dolores Alcalá González, Pedro José Sánchez Soto, José Manuel Córdoba Gallego, Ernesto Chicardi Augusto

RESUMEN / ABSTRACT

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

Durante el procesado de estos materiales es frecuente observar importantes gradientes de composición e interacciones entre las distintas fases constitutivas que dificultan alcanzar las propiedades deseadas. En el presente proyecto, pretendemos abordar un nuevo diseño para este tipo de materiales consistente en incorporar la mayor parte de sus componentes esenciales como soluciones sólidas complejas. Esto permitirá reducir el número final de fases en el material y obtener con mayor garantía de éxito las propiedades preestablecidas para las diversas aplicaciones tecnológicas. Para ello, planteamos una nueva ruta de síntesis basada en el proceso mecanoquímico denominado reacción de auto-propagación inducida mecánicamente (MSR), ya

que nuestro grupo ha demostrado que este método permite obtener de manera sencilla soluciones sólidas pertenecientes a los sistemas MT-B-C-N con un elevado control de la estequiometría. El objetivo principal del presente proyecto consiste en incorporar el método MSR a la metodología empleada para el desarrollo de materiales constituidos por soluciones sólidas que puedan ser utilizados en aplicaciones de alta temperatura. Se pretende caracterizar adecuadamente las propiedades de los materiales obtenidos y compararlas con aquellos fabricados con la metodología hasta ahora empleada.

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

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

Unión Europea
Fondo Europeo de Desarrollo Regional
"Una manera de hacer Europa"

JUNTA DE ANDALUCIA

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.

Unión Europea
Fondo Europeo de Desarrollo Regional
"Una manera de hacer Europa"

JUNTA DE ANDALUCIA

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

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

RESUMEN / ABSTRACT

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

cuanto a su nanoestructura y propiedades, en particular la piezoresistividad, porosidad, capacidad de inserción de litio y la resistencia a la oxidación.

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

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

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

RESUMEN / ABSTRACT

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

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

CONVENIOS Y CONTRATOS / CONTRACTS AND AGREEMENTS

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

Código/Code:	20124462
Periodo/Period:	25-11-2011 / 24-02-2015
Organismo Financiador/Financial source:	Proyectos e Implantación de Tuberias, SA
Importe total/Total amount:	319.440 €
Investigador responsable/Research head:	Luis A. Pérez Maqueda
Componentes/Research group:	Pedro E. Sánchez Jiménez, M. Jesús Diánez Millán, José Manuel Criado Luque, Enrique Jiménez Roca, M. Carmen Jiménez de Haro

PATENTES / PATENTS

Procedimiento de preparación de un mortero impermeabilizante que emplea filitas, producto obtenido y su utilización
Inventores: P.J. Sánchez Soto, F.J. Gotor Martínez, N. Fernández, E. Garzón
Ambito Geográfico: Patente Española, España
Patente Solicitada. Número de solicitud: P201530329
Fecha de solicitud: 13/marzo/2015
Entidades Titulares: CSIC - Universidad de Almería

Implemento mecánico para cuchara bivalva y su uso en la ejecución de pozos verticales de gran diámetro
Inventores: M. Cano, E. Garzón, I. Pulido, P.J. Sánchez Soto
Ambito Geográfico: Patente Española, España
Patente Solicitada. Número de solicitud: P201500466

Fecha de solicitud: 24/junio/2015
Entidades Titulares: CSIC - Universidad de Huelva - Universidad de Almería

Composición para el tratamiento químico de la verruga plantar
Inventores: J. Mª Juárez Jiménez, M. Romero Prieto, A. Córdoba, R. Rayo, P. Montaño, M.A. Avilés, P.J. Sánchez Soto
Ambito Geográfico: Patente Española, España
Patente Solicitada. Número de solicitud: P201500881
Fecha de solicitud: 1/diciembre/2015
Entidades Titulares: CSIC - Universidad de Sevilla

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

Ca-looping for postcombustion CO_2 capture: A comparative analysis on the performances of dolomite and limestone

Valverde, JM; Sanchez-Jimenez, PE; Perez-Maqueda, LA
Applied Energy, **138** (2015) 202-215
January, 2015 | **DOI:** 10.1016/j.apenergy.2014.10.087

The low cost and wide availability of natural limestone ($CaCO_3$) is at the basis of the industrial competitiveness of the Ca-looping (CaL) technology for postcombustion CO_2 capture as already demonstrated by similar to 1 Mw(t) scale pilot projects. A major focus of studies oriented towards further improving the efficiency of the CaL technology is how to prevent the gradual loss of capture capacity of limestone derived CaO as the number of carbonation/calcination cycles is increased. Natural dolomite ($MgCa(CO_3)_{(2)}$) has been proposed as an alternative sorbent precursor to limestone. Yet, carbonation of MgO is not thermodynamically favorable at CaL conditions, which may hinder the capture performance of dolomite. In the work described in this paper we carried out a thermogravimetric analysis on the multicyclic capture performance of natural dolomite under realistic regeneration conditions necessarily implying high calcination temperature, high CO_2 concentration and fast transitions between the carbonation and calcination stages. Our study demonstrates that the sorbent derived from dolomite has a greater capture capacity as compared to limestone. SEM analysis shows that MgO grains in the decomposed dolomite are resistant to sintering under severe calcination conditions and segregate from CaO acting as a thermally stable support which mitigates the multicyclic loss of CaO conversion. Moreover, full decomposition of dolomite is achieved at significantly lower calcination temperatures as compared to limestone, which would help improving further the industrial competitiveness of the technology.

Toughening of complete solid solution cermets by graphite addition

Chicardi, E; Torres, Y; Sayagues, MJ; Medri, V; Melandri, C; Cordoba, JM; Gotor, FJ
Chemical Engineering Journal, **267** (2015) 297-305
May, 2015 | **DOI:** 10.1016/j.cej.2015.01.022

$(Ti_{0.95}Ta_{0.05})(C_{0.5}N_{0.5})$-Co complete solid solution cermets (CSCs) were developed by a mechanochemical synthesis process and a pressureless sintering method. The effect of different percentages of graphite used as a sintering additive on the nature of the binder phase and the mechanical properties of the cermets was investigated. Microstructural and mechanical characterisations were carried out by X-ray diffraction, optical microscopy, scanning electron microscopy, transmission electron microscopy, energy-dispersive X-ray spectroscopy, Vickers hardness, indentation fracture toughness and nanoindentation. The addition of graphite modified the carbon activity during sintering, reducing the dissolution of carbonitride ceramic particles into the molten binder. The amount of Ti and Ta remaining in the binder after sintering gradually decreased as the amount of graphite added increased, which induced a change in the nature of the binder phase. When no graphite was added, the binder consisted of the brittle $Ti_xTa_{1-x}Co_2$ intermetallic phase. With the increase in the amount of graphite added, the formation of more ductile phases, such as $Ti_xTa_{1-x}Co_3$ and α-Co, was observed, causing a significant improvement in the toughness of the cermets.

Structural, Optical, and Electrical Characterization of Yttrium-Substituted $BiFeO_3$ Ceramics Prepared by Mechanical Activation

Perejon, A; Gil-Gonzalez, E; Sanchez-Jimenez, PE; Criado, JM; Perez-Maqueda, LA
Inorganic Chemistry, **54** (2015) 9876-9884
October, 2015 | **DOI:** 10.1021/acs.inorgchem.5b01654

Ceramics of $Bi_{1-x}Y_xFeO_3$ solid solutions (x = 0.02, 0.07, and 0.10) have been prepared by mechanical activation followed by sintering. The effect of yttrium content on the structural, electrical, and optical properties of the materials has been studied. Thus, single-phase solid solutions with rhombohedral R3c structure have been achieved for x = 0.02 and 0.07, while for x = 0.10 the main R3c phase has been detected together with a small amount of the orthorhombic Pbnm phase. Multiferroic properties of the samples, studied by differential scanning calorimetry (DSC), showed that both T-N and T-C (temperatures of the antiferromagnetic paramagnetic and ferroelectric-paraelectric transitions, respectively) decrease with increasing yttrium content. The nature of the ferroelectric paraelectric transition has been studied by temperature-dependent X-ray diffraction (XRD), which revealed rhombohedral R3c to orthorhombic Pbnm phase transitions for x = 0.07 and 0.10. On the other hand, for x = 0.02 the high-temperature phase was indexed as Pnma. Optical properties of the samples, as studied by diffuse reflectance spectroscopy, showed low optical band gap that decreases with increasing yttrium content. Prepared ceramics were highly insulating at room temperature and electrically homogeneous, as assayed by impedance spectroscopy, and the conductivity increased with x.

Limestone Calcination Nearby Equilibrium: Kinetics, CaO Crystal Structure, Sintering and Reactivity

Valverde, JM; Sanchez-Jimenez, PE; Perez-Maqueda, LA
Journal of Physical Chemistry C, **119** (2015) 1523-1541
January, 2015 | **DOI:** 10.1021/jp508745u

In this work, we analyze limestone calcination kinetics at environmental conditions involving a CO_2 partial pressure P close to the equilibrium pressure Peq by means of in situ X-ray diffraction (XRD) and thermogravimetric (TG) analyses. In contrast with previous empirical observations carried out mostly at conditions far from equilibrium ($P/Peq \ll 1$), our results show that the decarbonation rate decreases as the temperature in increased while P/Peq is kept constant, which is explained from a reaction mechanism including desorption of CO_2 and the exothermic structural transformation from metastable CaO* nanocrystals to the stable CaO form. The crystal structure and sintering of nascent CaO during calcination has been investigated from in situ XRD analysis, physisorption analysis, and scanning electron microscopy (SEM), which shows that the ratio of the size of polycrystalline CaO grains to crystallite size increases linearly with the CO_2 partial pressure in the calcination atmosphere. For high CO_2 partial pressures, the size of CaO grains reaches a maximum value of around 1 µm, which leads to a residual surface area of about 1 m2/g, whereas in the limit $P \rightarrow 0$ grain size and crystallite size (of the order of 10 nm) would coincide. Accordingly, sintering in the presence of CO_2 would be triggered by the agglomeration of CaO crystals enhanced by CO_2 adsorption, which increases the surface energy. The carbonation reactivity of CaO resulting from calcination scales proportionally to its surface area and is not determined by a growth of the CaO exposed surface along a preferred crystallographic direction wherein carbonation would be unfavorable as suggested in recent works.

Morphological changes on graphene nanoplatelets induced during dispersion into an epoxy resin by different methods

Moriche, R; Prolongo, SG; Sanchez, M; Jimenez-Suarez, A; Sayagues, MJ; Urena, A

Composites Part B-Engineering, **72** (2015) 199-205

April, 2015 | **DOI:** 10.1016/j.compositesb.2014.12.012

A structural analysis demonstrating how the manufacturing method of graphene nanoplatelets (GNPs) into a polymer matrix can strongly modify the GNPs morphology and, consequently, their properties, was carried out. Three different methods based on sonication and high shear forces were used to elucidate defects induction and possible size diminution. Manufacturing methods including high shear forces caused the extension of the GNPs while sonication induces wrinkling of the sheets. Residual stresses are induced in the nanoplatelets structure showing an increase in the Raman intensities ratios I-D/I-G and I-D/I-G when a major cycles number of calendering are applied.

A new model of the carbonator reactor in the calcium looping technology for post-combustion CO_2 capture

Ortiz, C; Chacartegui, R; Valverde, JM; Becerra, JA; Perez-Maqueda, LA

Fuel, **160** (2015) 328-338

November, 2015 | **DOI:** 10.1016/j.fuel.2015.07.095

The Ca-Looping (CaL) process is considered as a promising technology for CO_2 post-combustion capture in power generation plants yielding a minor penalty on plant performance as compared with other capture technologies such as conventional amine-based capture systems. This manuscript presents a new carbonator reactor model based on lab-scale multicyclic CaO conversion results, which take into account realistic CaO regeneration conditions that necessarily involve calcination under high CO_2 partial pressure and high temperature. Under these conditions, CaO conversion in the diffusion controlled stage is a relevant contribution to the carbonation degree during typical residence times. The main novelty of the model proposed in the present work is the consideration of the capture efficiency in the diffusion controlled phase of carbonation. It is demonstrated that increasing the residence time by a few minutes in the carbonator yields a significant improvement of the capture efficiency. Model predictions are shown to agree with experimental results retrieved from pilot-scale tests. The new model allows a more accurate evaluation and prediction of carbonator's performance over a wider range of residence times. The results obtained may be relevant for the optimization of CaL operation parameters to be used in real power plants.

Synthesis of a nanosilica supported CO_2 sorbent in a fluidized bed reactor

Soria-Hoyo, C; Valverde, JM; van Ommen, JR; Sanchez-Jimenez, PE; Perez-Maqueda, LA; Sayagues, MJ

Applied Surface Science, **328** (2015) 548-553

February, 2015 | **DOI:** 10.1016/j.apsusc.2014.12.106

CaO has been deposited on a nanosilica powder matrix by a procedure based on atomic layer deposition (ALD) in a fluidized bed reactor at atmospheric pressure following a potentially scalable process. In previous works ALD in gas fluidized bed has been mostly performed under reduced pressure, which hampers scaling-up the production technology. The material synthesized in the present work is tested as CO_2 solid sorbent at calcium looping conditions. Multicyclic thermogravimetric analysis (TGA) shows that the nanosilica support stabilizes the capture capacity of CaO. EDX-STEM analysis illustrates the presence of Ca well distributed on the surface of the SiO_2 nanoparticles.

Ceramics from the Alcazar Palace in Seville (Spain) dated between the 11th and 15th centuries: Compositions, technological features and degradation processes

Garofano, I; Robador, MD; Perez-Rodriguez, JL; Castaing, J; Pacheco, C; Duran, A

Journal of the European Ceramic Society, **35** (2015) 4307-4319

December, 2015 | **DOI:** 10.1016/j.jeurceramsoc.2015.07.033

For the purpose of chemical characterization, a selection of 29 ceramic fragments from the 11th to 15th century has been made from materials excavated in the Seville Alcazar Palace. The PIXE elemental analysis results indicated that the ceramic bodies can be divided into four groups. The XRD identification of the phases present in the bodies revealed differences in the firing temperatures. The PIXE analysis of the glazes revealed variable PbO and SnO_2 contents. The latter component was not detected in all the glazes. Cu, Co, Mn, Fe and Sb elements were associated with green, blue, black and yellow colours, respectively. Some glazes were covered by iridescent layers constituted by lead carbonate and phosphates due to alteration of the glazes. It was also possible to detail the microstructure and composition of the ultimate surface layers responsible for the lustre effect observed in two of the ceramic samples using PIXE and Rutherford backscattering spectrometry (RBS).

Preparation of phase pure, dense fine grained ceramics by conventional and spark plasma sintering of La-substituted $BiFeO_3$ nanoparticles

Perejon, Antonio; Sanchez-Jimenez, Pedro E.; Poyato, Rosalia; Maso, Nahum; West, Anthony R.; Criado, Jose M.; Perez-Maqueda, Luis A.

Journal of the European Ceramic Society, **35** (2015) 2283-2293
August, 2015 | **DOI:** 10.1016/j.jeurceramsoc.2015.01.030

High density ceramics of the system $Bi_{1-x}La_xFeO_3$, 0 <= x <= 0.15, have been prepared starting from nanoparticles obtained by mechanosynthesis. The ceramics have been sintered conventionally at 850 degrees C and by spark plasma sintering (SPS). Sintering conditions have been optimized to obtain single phase ceramics, and the microstructure of the ceramics has been compared. Ceramics prepared conventionally present grain sizes from 5 mu m to less than 1 mu m, whereas grain sizes by SPS are in the range from 50 to 100 nm, which demonstrates that it is possible to obtain nanostructured ceramics of La-substituted $BiFeO_3$ using mechanosynthesis followed by SPS at low temperature (625-650 degrees C). The as-prepared SPS ceramics show low resistivity, indicating some reduction in the samples. However, after an oxidative anneal in air, ceramics are highly insulating at room temperature and electrically homogeneous. The high quality of the ceramics has also been demonstrated by XRD, EDX, Raman and DSC.

New Insights on the Kinetic Analysis of Isothermal Data: The Independence of the Activation Energy from the Assumed Kinetic Model

Sanchez-Jimenez, PE; Perejon, A; Perez-Maqueda, LA; Criado, JM

Energy & Fuels, **29** (2015) 392-397
January, 2015 | **DOI:** 10.1021/ef502269r

Isothermal experiments are widely employed to study the kinetics of solid-state reactions or processes to extract essential kinetic information needed for modeling the processes at an industrial scale. The kinetic analysis of isothermal data requires finding or assuming a kinetic function that can properly fit the evolution of the reaction rate with time, so that the resulting parameters, i.e., the activation energy and pre-exponential factor, can be considered reliable. In the present work, we demonstrate using both simulated and experimental data that the kinetic analysis of a set of isothermal plots obtained at different temperatures, considering a single-step solid-state reaction, necessarily leads to the real activation energy, regardless the mathematical function selected for performing the kinetic analysis. This makes irrelevant the election of the kinetic function used to fit the experimental data and greatly facilitates the estimation of the activation energy for any single process.

Mechanically induced self-propagating reaction of vanadium carbonitride

Roldan, MA; Alcala, MD; Real, C

Ceramics Internationall, **41** (2015) 4688-4695
April, 2015 | **DOI:** 10.1016/j.ceramint.2014.12.016

Vanadium carbonitrides (VCxN1-x) were prepared via mechanosynthesis from mixtures of elemental vanadium and carbon with different V/C atomic ratios under a nitrogen atmosphere using a high-energy ball mill. We obtained the full composition range of carbonitrides at room temperature. The products were characterized using X-ray diffraction, scanning electron microscopy and electron energy loss spectroscopy. The results showed particle-sized products with high sinterability and very high microhardness.

An improved method for determining the external specific surface area and the plasticity index of clayey samples based on a simplified method for non-swelling fine-grained soils

Garzon, E; Sanchez-Soto, PJ

Applied Clay Science, **115** (2015) 97-107

October, 2015 | **DOI:** 10.1016/j.clay.2015.07.015

Previous studies have used the clay content of soils for estimating the specific surface areas and different correlations have been found, including plasticity-value correlations. Based on several assumptions, Dolinar (2012) proposed a simplified method for determining the external specific surface area of non-swelling fine-grained soils. An equation relates the external specific surface area (BET-nitrogen) with percentage of clay fraction (< 2 μm), determined by hydrometer method, and plasticity index (Atterberg).

In this work, based on that simplified method, the authors have developed an improved method for determining the external specific surface area of fine-grained clay samples. Instead of percentage of clay fraction, it was proposed to use the clay mineral content estimated by XRD methods. From an analysis of previous Dolinars results, the calculated and measured values of external specific surface area were studied for a group of non-swelling and fine-grained soil samples (Dolinar's samples), five non-swelling clayey samples and data samples from the literature. Additionally, an estimation of the plasticity index (Atterberg) has been also considered in this improved method. Both these methods, simplified and improved, were tested and compared using all these samples. It demonstrated the practical application of both these methods for an estimation of external specific surface area and plasticity index. However, in the present research two models were considered to determine the specific surface areas (BET and Langmuir) and the influence of several sources of errors in these predictions was discussed. The predictions were found more accurate when specific surface area from Langmuir's model is considered. It is concluded that the present research will be useful for the prediction of external specific surface area and plasticity index of non-swelling clayey materials and to dispose of theoretical practical relationships between clay mineralogy and geotechnical properties of valuable interest.

Green pigments of Roman mural paintings from Seville Alcazar

Perez-Rodriguez, JL; de Haro, MDJ; Siguenza, B; Martinez-Blanes, JM

Applied Clay Science, **116** (2015) 211-219

November, 2015 | **DOI:** 10.1016/j.clay.2015.03.016

We report here a study of 30 fragments of green wall paintings from Roman times found in the Patio de Banderas excavation in Seville Alcazar. The sample characterisation was realised using optical microscopy, colourimetry, infrared and micro-Raman spectroscopy, X-ray diffraction, and scanning electron microscopy equipped with energy dispersive X-ray spectroscopy. The study of these pigments is important because it can help determine the source or the pictorial technique used. The samples studied in this work have been divided into two groups, according to the composition of their green pigments. In the first group, celadonite has been characterised as the primary component of the green colour; chlorite was also detected. Particles constituted by chromium accompanied by aluminium, iron and zinc were found in all studied samples of this group. Chlorite and chromium oxide could also be responsible for the green colour. The presence of chromium suggested the presence of green colour pigment from Verona. In the second group, a mixture of celadonite and glauconite was detected and could be responsible for the green colour observed. The addition of refracting material such as Egyptian blue was also used. A mixture of Egyptian green and Egyptian blue together with celadonite and glauconite was also found. Four classes of intonaco were recognised and classified based upon the composition of the aggregates.

Phyllite clay-cement composites having improved engineering properties and material applications

Garzon, E; Cano, M; O'Kelly, BC; Sanchez-Soto, PJ

Applied Clay Science, **144** (2015) 229-233

September, 2015 | **DOI:** 10.1016/j.clay.2015.06.006

Phyllite clays contain clay minerals (chlorite, illite and mixed-layer illite smectite), quartz and feldspars. In this experimental laboratory study, new composites of phyllite clay and cement (5, 7 and 9 wt.%) were prepared and tested to determine their Atterberg limits, dry density and optimum water content for modified Proctor (MP) compaction, California Bearing ratio, swelling potential after soakage in water, unconfined compressive strength (UCS) and water-permeability coefficient. From the mixes investigated, the composite with 5 wt.% cement was deemed most suitable for certain construction material applications, having a plasticity index of 10.5%, maximum dry density of 2.17 Mg/m3 and optimum water content of 8% for MP compaction (undergoing no swelling under soakage), a UCS of 0.74 MPa, and a very low permeability coefficient value of 7.4 × 10− 11 m/s. Potential material applications for these new composites include for building construction, roofs, and flexible pavements.

Valorization and inertization of galvanic sludge waste in clay bricks

Perez-Villarejo, L; Martinez-Martinez, S; Carrasco-Hurtado, B; Eliche-Quesada, D; Urena-Nieto, C; Sanchez-Soto, PJ

Applied Clay Science, **105-106** (2015) 89-99

March, 2015 | **DOI:** 10.1016/j.clay.2014.12.022

Galvanic sludge wastes (GSW) are produced by the physico-chemical treatments of wastewater generated by electroplating plants. These materials have a significant potential for the production of clay ceramic bricks. This paper focuses on the viability of the inertization of heavy metals from GSW mixed with clays. The original materials were obtained by mixing three types of raw clay (red, yellow and black) in equal parts with GSW. These mixtures were characterized by XRD, XRF, and chemical elemental analysis CHNS. The dosage of GSW in the clay–GSW bricks was up to 5 wt.%. The bricks were then manufactured using conventional processes. The influence of the amount of GSW was evaluated after firing the clay–GSW composites at 950 °C for 1 h. The engineering properties of the fired samples, such as density, water absorption, open porosity, water suction and compressive strength, with and without the GSW, were determined. The incorporation of GSW into the clay mix clearly decreased the linear shrinkage and bulk density of the bricks in comparison with the fired clay used as a control. These GSW–clay composites also showed lower open porosity. According to the results obtained for the bulk density of the bricks, samples with GSW addition showed slightly lower values of open porosity than clay bodies, indicating that the GSW–clay samples had slightly higher closed porosity values. This was also shown by SEM. The open porosity, SEM and pore size distribution tests indicated that the porosity generated by the addition of GSW was mainly closed and, therefore, GSW bricks had excellent mechanical properties. The environmental risks of the incorporation of GSW, rich in heavy metals (Cr, Zn, Ni and others), to a clay matrix were evaluated by leaching tests of the fired products. The results indicated a successful inertization of the pollutants.

Nanoindentation of (Ti,Ta)(C,N)-Co cermets prepared by methods of mechanochemistry

Hvizdos, Pavol; Balko, Jan; Manuel Cordoba, Jose; Chicardi, Ernesto
International Journal of Refractory Metals & Hard Materials, **49** (2015) 219-224
March, 2015 | **DOI:** 10.1016/j.ijrmhm.2014.07.038

Four materials TixTa(1 - x)C(0.5)N(0.5)-20%Co of two chemical compositions (x = 0.9 and 0.95) and two high energy milling methods (one stage and two stage milling) have been prepared. Nano-hardness and elastic modulus for microstructure as a whole and for individual constituent phases (matrix and carbide grains) were obtained by instrumented indentation. Individual targeted indentations as well as grid nanoindentation technique were used to discern the individual constituents' properties. Maximum loads of 20 mN for individual phases and 300 mN for composite microstructure were applied. Materials with higher amount of Ti had larger grains but the milling procedure had stronger influence on the grain size. The two step milling resulted in finer microstructures but with a much wider grain size distribution. Final composite mechanical properties, however, were very similar. Hardness and indentation elasticity modulus of all materials were comparable within the errors of measurement.

Applications of sample-controlled thermal analysis (SCTA) to kinetic analysis and synthesis of materials

Perez-Maqueda, L. A.; Criado, J. M.; Sanchez-Jimenez, P. E.; Dianez, M. J.
Journal of Thermal Analysis and Calorimetry, **120** (2015) 45–51
April, 2015 | **DOI:** 10.1007/s10973-014-4176-6

The advantages of the sample-controlled thermal analysis (SCTA) for both the kinetic analysis of solid-state reactions and the synthesis of materials are reviewed. This method implies an intelligent control of the temperature by the solid-state reaction under study in such a way that the reaction rate as a function of the time fits a profile previously defined by the user. It has been shown that SCTA has important advantages for discriminating the kinetic model of solid-state reactions as compared with conventional rising temperature methods. Moreover, the advantages of SCTA methods for synthesising materials with controlled texture and structure are analysed.

Study of coatings by thermal analysis in a monument built with calcarenite

Luisa Franquelo, Maria; Dolores Robador, Maria; Luis Perez-Rodriguez, Jose
Journal of Thermal Analysis and Calorimetry, **121** (2015) 195-201
July, 2015 | **DOI:** 10.1007/s10973-015-4432-4

In this research, characterization of materials either added during restoration or formed by environmental contamination in the Seville City Hall, built with calcarenite stone, was investigated by thermal methods. Three different mortars for restoration have been characterized: (a) lime micro-mortar for internal consolidation of mortar itself, (b) mortar for reconstruction of deteriorated areas and (c) mortar with Portland cement. Acrylate polymer as consolidant and protection used was characterized. Addition of gypsum or “white cement” has been also applied in the restoration. Altered materials as black crusts constituted by gypsum, calcite and organic compound were determined by thermal analysis. Patina with high concentration of hydrated calcium oxalate, and the transformation mechanism of calcium oxalate into calcium carbonate and formation of calcium oxide produced by decomposition of the calcite were also characterized in the studied monument by thermal analysis. The patina with hydrated calcium oxalate produced by high biological activity was also studied.

In Situ Synthesis of a ZrB_2-Based Composite Powder Using a Mechanochemical Reaction for the Zircon/Magnesium/Boron Oxide/Graphite System

Jalaly, M; Bafghi, MSS; Tamizifar, M; Gotor, FJ
International Journal of Applied Ceramic Technology, **12** (2015) 551-559
May, 2015 | DOI: 10.1111/ijac.12202

A $ZrSiO_4/B_2O_3/Mg/C$ system was used to synthesize a ZrB_2-based composite through a high-energy ball milling process. As a result of the milling process, a mechanically induced self-sustaining reaction (MSR) was achieved in this system. A composite powder of ZrB_2-SiC-ZrC was prepared in situ by a magnesiothermic reduction with an ignition time of approximately 6min. The mechanism for the formation of the product was investigated by studying the relevant subreactions, the stoichiometric amount of B_2O_3, and thermal analysis.

Photocatalytic Properties of TiO_2 Thin Films Modified with Ag and Pt Nanoparticles Deposited by Gas Flow Sputtering

Maicu, M; Gloss, D; Frach, P; Hecker, D; Gerlach, G; Cordoba, JM
Journal of Nanoscience and Nanotechnology, **15** (2015) 6478-6486
September, 2015 | **DOI:** 10.1166/jnn.2015.10873

In this work, a gas flow sputtering (GFS) process which allows the production and deposition of metal nanoparticles (NPs) in a vacuum environment is described. Aim of the study is to prove the potential of this technology for the fabrication of new TiO_2 films with enhanced photocatalytic properties. For this purpose, Ag and Pt NPs have been produced and deposited on photocatalytic float glass coated with TiO_2 thin films by magnetron sputtering. The influence of the process parameters and of the metal amount on the final properties of the particles (quantity, size, size distribution, oxidation state etc.,) was widely investigated. Moreover, the effect of the NPs on the photocatalytic activity of the resulting materials was evaluated for the case of the decomposition of stearic acid (SA) during UV-A irradiation. The reduction of the water contact angle (WCA) during the irradiation period was measured in order to test the photo-induced super-hydrophilicity (PSH).

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

Decenio Internacional 2005-2015 para la acción "El agua, fuente de vida"
A.Macho, P.J. Sánchez-Soto
Acta Científica y Tecnológica 25 (2014) 13-14

El Consejo Superior de Investigaciones Científicas (CSIC) celebra su 75 aniversario
P.J. Sánchez-Soto
Químicos del Sur (Revista de los Químicos de Andalucía y Extremadura), 100 (2015) 11-12

LIBROS Y OTRAS PUBLICACIONES / BOOKS AND OTHER PUBLICATIONS

S. Martínez-Martínez, L. Pérez-Villarejo, R. Moreno, P.J. Sánchez-Soto
"Desarrollo y evolución de las fases cristalinas con la temperatura en una sienita alterada", en: Más allá de la Geología, Teófilo Sanfeliu Montolio, Libro Homenaje, M.M. Jordán Vidal, F. Pardo Fabregat y Ana Belén Forteza (Eds.), págs. 131-134 (2015)
ISBN: 978-84-16356-15-7

G. Durán, L. Pérez-Villarejo, P.J. Sánchez-Soto
"Milenaria ciudad de arcilla: Guadix y su cerámica artística", en: Más allá de la Geología, Teófilo Sanfeliu Montolio, Libro Homenaje, M.M. Jordán Vidal, F. Pardo Fabregat y Ana Belén Forteza (Eds.), págs. 145-147 (2015)

E. Domínguez-Fernández, L. Pérez-Villarejo, J. Mª Martínez-Blanes, P.J. Sánchez-Soto
"Caracterización mineralógica de materiales procedentes de intervenciones arqueológicas de los restos del Hospital de la orden militar de San Juan de Jerusalén (Iglesia de Cedillo del Condado, Toledo)", en: Más allá de la Geología, Teófilo Sanfeliu Montolio, Libro Homenaje, M.M. Jordán Vidal, F. Pardo Fabregat y Ana Belén Forteza (Eds.), págs. 187-192 (2015)

L. Pérez-Villarejo, S. Martínez-Martínez, E. Garzón, A. Macho, P.J. Sánchez-Soto
"Caracterización mineralógica de pizarras carboníferas de Sierra Morena Occidental", en: Más allá de Geología, Teófilo Sanfeliu Montolio, Libro Homenaje, M.M. Jordán Vidal, F. Pardo Fabregat y Ana Belén Forteza (Eds.), págs. 193-196 (2015)

A.Kriznar, P.J. Sánchez-Soto. "Caracterización mineralógica de morteros usados como soporte en pinturas murales de Johannes Aquila (siglo XIV)", en: Más allá de la Geología, Teófilo Sanfeliu Montolio, Libro Homenaje, M.M. Jordán Vidal, F. Pardo Fabregat y Ana Belén Forteza (Eds.), págs. 197-201 (2015)

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

19th International Congress on Project Management and Engineering

15 - 17 julio [Granada, España]

The Guadua Angustifolia as a structural applied in greenhouses design
E. Garzón, M. Cano, L. Pérez-Villarejo, S. Martínez, P.J. Sánchez-Soto
Comunicación oral

Determination of sustainability indicators in the assessment of the conservation status of popular Architecture
M. Cano, E. Garzón, P.J. Sánchez-Soto
Comunicación oral

14th International Conference of the European Ceramic

21 - 25 junio [Toledo, España]

Characterization of ash from biomass combustion. Viability of use as raw material for cement chemically modified and activated
S. Martínez-Martínez, D. Eliche-Quesada, L. Pérez-Villarejo, P.J. Sánchez-Soto
Comunicación oral

Advances in Materials & Processing Technologies Conference

14 - 17 diciembre [Madrid, España]

A new family of cermets: chemically complex but simple microstructurally
F.J. Gotor, M.A. Avilés, A. García de la Obra, M.J. Sayagués, Y. Torres, L. Llanes, E. Chicardi, J.M. Córdoba
Conferencia Invitada

Fabrication and characterization of CeO_2 radial graded porosity pellets: Influence of Power Metallurgy parameters
C. García-Ostos, C. Arévalo, J.A. Rodríguez-Ortiz, J.J. Pavón, F.J. Gotor, J. Cobos, Y. Torres
Comunicación oral

Ti(C,N)-Co/WC-Co laminates: A thermomechanical compromise
F.J. Gotor, L. González, R. Bermejo, Y. Torres
Poster

CONGRESOS Y REUNIONES NACIONALES / NATIONAL CONGRESS AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

XXXII Encuentro del Grupo Español de Fractura
27 - 29 abril [Zamora, España]

Aumento de la tenacidad de fractura en cermets basados en carbonitruro de titanio mediante adición de grafito
E. Chicardi, Y. Torres, M.J. Sayagués, V. Medri, C. Melandri, J.M. Córdoba, F.J. Gotor
Comunicación oral

Diseño, procesado y caracterización de materiales con porosidad radial controlada para aplicaciones nucleares
C. García Ostos, J.A. Rodríguez Ortiz, C. Arévalo, J. Cobos, F.J. Gotor, J.J. Pavón, Y. Torres
Comunicación oral

Diseño y fabricación de laminados tipo Ti(C,N)-Co/WC-Co con un potencial equilibrio de propiedades en servicio
L.M. González, F.J. Gotor, R. Bermejo, E. Chicardi, J.M. Córdoba, V. Medri, D. Dalle Fabbriche, Y. Torres
Comunicación oral

Resistencia mecánica de carburos cementados, influencia de la temperatura y la microestructura
I. Moyano, R. Bermejo, N. Salan, E. Chicardi, F.J. Gotor, L. Llanes, Y. Torres
Comunicación oral

Materials 2015
21 - 23 junio [Oporto, Portugal]

Precipitation hardening mechanism of Cu-Ni-Zn-Al Alloys
E. Donoso, M.J. Diánez, J.M. Criado, M.J. Sayagués, G. Díaz, L. Olivares
Comunicación oral

V Congreso Español de Pulvimetalurgia
1 - 3 julio [Girona, España]

Influencia de la temperatura, en la resistencia mecánica de cermets basados en carbonitruro de titanio
F.J. Gotor, E. Chicardi, R. Bermejo, L. Llanes, J.M. Córdoba, Y. Torres
Comunicación oral

Influencia de la configuración del ensayo, la temperatura y la microestructura en el comportamiento mecánico de WC-Co
F.J. Gotor, I. Moyano, R. Bermejo, E. Chicardi, N. Salan, L. Llanes, Y. Torres
Poster

XXIV Reunión de la Sociedad Española de Arcillas

5 - 7 noviembre [Sevilla, España]

Estudio de la evolución térmica de una filita mediante análisis térmico y difracción de rayos X de alta temperatura
P.J. Sánchez-Soto, S. Martínez-Martínez, L. Pérez Villarejo, L. Eliche-Quesada, E. Garzón
Póster

Desarrollo de un nuevo tipo de clínker de cemento que incorpora subproductos procedentes de la industria ladrillera
S. Martínez-Martínez, L. Pérez Villarejo, L. Eliche-Quesada, E. Garzón, J.M. Galdón-Requena, P.J. Sánchez Soto
Póster

Estudio y Conservación del Patrimonio Cultural | ECPC 2015

16 - 19 noviembre [Málaga, España]

Caracterización de muestras aplicadas al estudio comparativo de dos obras de Murillo sobre soporte de tabla y lienzo, de la Iglesia de la Santa Caridad de Sevilla, Niño Jesús y La Anunciacón
A. Justo Erbez, M. Arjonilla Alvarez, A. Ruiz Conde, B. Sigüenza Carballo
Poster

CONAMET / SAM 2015

17 - 20 noviembre [Concepción, Chile]

Caracterización no isotérmica del proceso de precipitación en una aleación de Cu-11Ni-19Zn-15n
E. Donoso, M.J. Diánez, J.M. Criado, R. Espinoza, E. Mosquera
Comunicación oral

Formacion / Training

Formación de Graduados / Master Degree Thesis

Título: **Materiales compuestos multicapas ultra-resistentes para mecanizado a alta velocidad**
Autor: Sebastián Barroso de la Rosa
Directores: Francisco José Gotor Martínez
Grado: Trabajo Fin de Grado
Año Académico: 2014-2015 (15 enero 2015)

Título: **Síntesis y caracterización de composites de SiC/Si_3N_4 a partir de cáscara de arroz**
Autor: Javier López de la Osa García
Directores: Concepción Real Pérez, María Dolores Alcalá González
Grado: Trabajo Fin de Grado
Año Académico: 2014-2015 (15 enero 2015)

Título: **Comportamiento termo-mecánico de WC-Co. Influencia de la microestructura y la temperatura**
Autor: Ismael Juan Moyano Rodríguez
Directores: Francisco José Gotor Martínez
Grado: Trabajo Fin de Grado
Año Académico: 2014-2015 (9 julio 2015)

Título: **Comportamiento termo-mecánico de carbonitruros de titanio y tántalo (Ti Ta)(CN)**
Autor: José Pavón Pérez
Directores: Francisco José Gotor Martínez
Grado: Trabajo Fin de Grado
Año Académico: 2014-2015 (15 octubre 2015)

Título: **Desarrollo de cermets con aleaciones de CoCrFeNiV como fase ligante**
Autor: Jesús Castillejo García
Directores: Francisco José Gotor Martínez
Grado: Trabajo Fin de Grado
Año Académico: 2014-2015 (15 octubre 2015)

Título: **Síntesis de silicio tecnológico a partir de cascarilla de arroz**
Autor: Carlos González Alanís

Directores: Concepción Real Pérez, María Dolores Alcalá González
Grado: Trabajo Fin de Grado
Año Académico: 2014-2015 (16 julio 2015)

Título: **Desarrollo de cermets empleando aleaciones de alta entropía como fase cementante**
Autor: Alma García de la Obra
Directores: Francisco José Gotor Martínez
Grado: Trabajo Fin de Master
Año Académico: 2014-2015 (11 septiembre 2015)

Premios y Reconocimientos / Prizes and Acknowledgements

Premio "Jaume Blasco" a la innovación 2015, otorgado por AEIPRO (Asociación Española de Dirección e Ingeniería de Proyectos), y unanimidad del jurado (Granada, 16 de julio de 2015), realizado dentro del 19th International Congress on Project Management and Engineering (Granada, 15-17 julio 2015) por la Comunicación presentada
P.J. Sánchez-Soto, co-autor junto a E. Garzón, M. Cano, L. Pérez-Villarejo y S. Martínez

Equipamiento Científico Available Equipment

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

MATERIALES FUNCIONALES NANOESTRUCTURADOS
NANOSTRUCTURED FUNCTIONAL MATERIALS

PERSONAL / PERSONNEL

Profesor de Investigación

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

Catedráticos

Dr. José Cotrino Bautista

Investigadores Científicos

Dr. Francisco Yubero Valencia

Científicos Titulares

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

Profesor Visitante

Dr. Richard M. Lambert

Investigadores Contratados

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

Becarios Predoctorales

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

Personal Contratado

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

PROYECTOS DE INVESTIGACIÓN / RESEARCH PROJECTS

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

Código/Code:	307081
Periodo/Period:	01-01-2012 / 30-11-2017
Organismo Financiador/Financial source:	Unión Europea. 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.

Arquitecturas SOFC innovadoras basadas en operación "Triodo"

Innovative SOFC Architecture based on Triode Operation

Código/Code:	FCH-JU-2011-1
Periodo/Period:	01-09-2012 / 31-08-2015
Organismo Financiador/Financial source:	Unión Europea
Importe total/Total amount:	256.363 €
Investigador responsable/Research head:	Agustín R. González-Elipe
Componentes/Research group:	Francisco Yubero Valencia, Juan Pedro Espinós Manzorro, Angel Barranco Quero, Richard Lambert, Victor J. Rico, Ana Borrás Martos, José Cotrino, Jorge Gil, Pedro Castillero, Fran J. García, Alberto Palmero

RESUMEN / ABSTRACT

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

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

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

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

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

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

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

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

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

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

RESUMEN / ABSTRACT

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

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

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

It is the main goal of this project to bring to the host institution and the European Research Area the knowledge and technology to prepare current record flexible dye sensitized photovoltaic devices, previously developed by the candidate in South Korea and then the USA, in order to be able to further improve them, while endowing them with semi-transparency, using stretchable and bendable optical materials. The candidate has demonstrated that several key materials and processes provide better performance of bendable dye solar cells, i.e., enhanced efficiency and flexibility, by allowing the preparation of electrodes in which the electron diffusion length is longer and charge collection efficiency is consequently enhanced. However, highly efficient dye solar cells are opaque as a consequence of the particular diffuse scattering design employed to improve light absorption, which limits their application in building or auto-

motive integrated photovoltaics. This proposal seeks to solve such drawback by introducing photonic nanostructures in different configurations, yielding both light harvesting enhancement and preserving transparency, hence placing Europe at the forefront of research in this specific area within the field of renewable energy. This final goal will be attempted through different approaches, each one challenging from the materials science perspective. Preparation of such highly efficient and transparent devices will combine the flexible solar cell processing tools previously developed by the candidate with the versatile optical material preparation techniques pioneered by the host institution. More specifically, integration of novel porous flexible photonic structures into the light harvesting layer, use of flexible mirrors attached to the back of the counter-electrode, and designed distribution of scatterers will be employed to reach the target.

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

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

RESUMEN / ABSTRACT

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

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

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

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

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

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

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

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

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

Purificación del aire en invernaderos y centros de tratamiento de alimentos

Purification of air in greenhouses and food processing centers

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

RESUMEN / ABSTRACT

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

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

Unión Europea
Fondo Europeo de Desarrollo Regional
"Una manera de hacer Europa"
Ministerio de Economía y Competitividad

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

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

RESUMEN / ABSTRACT

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

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

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

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

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

RESUMEN / ABSTRACT

HYBR(1)D es un proyecto de carácter multidisciplinar en el que se persigue el desarrollo de nuevos materiales nanoestructurados multifuncionales que encuentren aplicación final en campos como las energías renovables, fotónica y la miniaturización de dispositivos. En el proyecto se plantea como principal objetivo el desarrollo de métodos de fabricación de nuevos materiales unidimensionales nanoestructurados como nanocables orgánicos e inorgánicos y sistemas unidimensionales heteroestructurados e híbridos. Haciendo especial hincapié en sistemas compuestos del tipo coaxial "core@shell/multi-shell" que integren componentes orgánicas, metales y óxidos. Estos materiales serán fabricados mediante una innovativa metodología que permiten su formación sobre sustratos procesables de distinta naturaleza involucrando tecnologías escalables industrialmente. Además se propone de forma exploratoria la fabricación de membranas "compuestas" que permitan el uso de estas nanoestructuras embebidas de forma autosoportada. El segundo objetivo de este proyecto es probar la funcionalidad de estas nuevas nanoestructuras en distintas aplicaciones atendiendo al concepto de “nanosistema autoalimentado”: como sistemas de generación de energía (celdas solares y piezoelectricidad) y nanosensores. Cabe resaltar que aunque los materiales a fabricar son muy diversos, desde nanotubos de semiconductores inorgánicos (TiO2 y ZnO) a nanohilos orgánicos ("small-molecule single crystal nanowires) híbridos y heteroestruturados, los métodos de fabricación que se pretenden utilizar se basan en técnicas de vacío muy similares y facilmente acoplables. Así, las distintas nanoestructuras y heteroestructuras se fabricarán mediante cuatro técnicas principales y combinaciones de las mismas: deposición física desde fase vapor aplicada a moléculas orgánicas, deposición química desde fase vapor asistida por plasma de moléculas orgánicas y óxidos semiconductores, sputtering-dc de metales y "etching" por plasma de oxígeno. El IP y el grupo Nanotechnology on Surfaces del ICMS tienen un amplio background en la aplicación de estas técnicas para el desarrollo de sistemas del tipo láminas delgadas y recubrimientos funcionales, experiencia que se ha extendido en los últimos años al estudio de nanoestructuras 1D soportadas. El proyecto aborda toda la cadena de valor que lleva desde la síntesis de nuevos materiales a sus aplicaciones, incluyendo su caracterización avanzada e integración en dispositivos y prototipos a escala de laboratorio.

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

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

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

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.

Unión Europea
Fondo Europeo de Desarrollo Regional
"Una manera de hacer Europa"
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:	Manuel Ocaña Jurado, Ana Isabel Becerro Nieto, Nuria Núñez Alvarez, Mauricio E. Calvo

Roggiani, Gabriel Lozano Barbero, Juan Francisco Galisteo López, Miguel Anaya Martin

RESUMEN / ABSTRACT

El proyecto MODO se centrará en el desarrollo de materiales ópticos que optimicen el funcionamiento de dispositivos optoelectrónicos tales como celdas fotovoltaicas o dispositivos emisores de luz, mejorando así su eficiencia de conversión enérgetica. 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.

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

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 / 31-12-2016
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.

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

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

RESUMEN / ABSTRACT

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

This project aims to elaborate ERC Starting Grant 2016 proposal entitled: Photonic nanostructures to more efficient and versatile light-emitting devices (NanoLED), which will become granted. NanoLED project seeks to largely surpass the optical performance of state-of-the-art light emitters devised for illumination applications. This project aims to develop novel optical

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

Unión Europea
Fondo Europeo de Desarrollo Regional
"Una manera de hacer Europa"

JUNTA DE ANDALUCIA

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

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

RESUMEN / ABSTRACT

Este Proyecto pretende contribuir al avance en el desarrollo de nuevos biomateriales con bioactividad mejorada para su aplicación en la reparación y regeneración ósea. Se propone la preparación de nuevos recubrimientos y andamios cerámicos mediante técnicas de procesado láser a partir de materiales cerámicos particulados, nanoestructurados de composición bioactiva en el sistema SiO_2-CaO-P_2O_5 sintetizados en el ICMS por la Dra. Díaz (IP de esta solicitud). La hipótesis de partida es que las características nanoestructurales y texturales del material cerámico particulado en combinación con la fuente láser tiene un gran potencial para resultar en deposiciones con microestructura y nanoestructura controlada. Se plantea procesar dos tipos de pieza prototipo: i) piezas de sustrato metálico (de titanio) con recubrimiento cerámico de composición bioactiva y ii) piezas monolito (andamio) solo cerámica bioactiva con geometría controlada. Hay que distinguir por tanto la persecución de dos hitos. Un primer Hito es la obtención de materiales prototipo (recubrimientos y andamios) con unos requerimientos de micro y nanoestructura, superficie, propiedades mecánicas, homogeneidad y reproducibilidad mínimas. Y un segundo Hito consistente en la verificación de sus propiedades biológicas in vitro e in vivo.

El éxito en ambos hitos es lo que finalmente denominaremos biomaterial prototipo. La obtención de los recubrimientos tendría una translación muy directa para implementarse en distintas partes de los componentes de implantes ortopédicos. En este sentido el equipo de investigación integra a biólogos de regeneración esquelética y clínicos de cirugía ortopédica y traumatología que van a estudiar la bioactividad y biocompatibilidad de estos recubrimientos sobre sustratos de titanio suministrados por Synthes, empresa líder en fabricación y comercialización de implantes para ortopedia, que participa en esta propuesta como EPO. Por otra parte y desde el punto de vista del procesado de materiales, la aplicación del procesado láser a los materiales cerámicos nanoestructurados SiO_2-CaO-P_2O_5 es totalmente novedoso y creemos que puede optimizarse para obtener recubrimientos y también andamios tridimensionales, conformados con macroporosidad interconectada pero a su vez con microestructura fina y nanoestructura, que culminen en la obtención de piezas prototipo con alta reproducibilidad y de calidad e innovación tecnológica elevada. El Proyecto se encuadra en áreas de investigación fuertemente innovadoras y pujantes como son la nanotecnología y la medicina regenerativa ambas con un carácter multidisciplinar intrínseco donde la frontera entre las distintas disciplinas científicas tradicionales aparece difuminada. El Proyecto aúna esfuerzos de investigadores con formación básica en las disciplinas de química (ICMS), física e ingeniería (Empresa Subcontratada AIMEN), biología (UMA-CIBER-BBN) y medicina clínica y traslacional (HCS). Creemos que la integración de estos tres pilares i) síntesis, procesado y caracterización de materiales, ii) biología de la regeneración e ingeniería tisular y iii) práctica clínica ofrece una propuesta con capacidad para aportar resultados de impacto trasferibles a la industria y que puede por tanto ayudar al desarrollo de productos para aplicaciones en la reparación y regeneración esquelética en Andalucía.

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

Unión Europea
Fondo Europeo de Desarrollo Regional
"Una manera de hacer Europa"
JUNTA DE ANDALUCIA

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

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

RESUMEN / ABSTRACT

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

The objective of the LUMEN project is the development of luminescent devices incorporating as active layers rare earth containing thin films deposited by plasma CVD. The thin films will be deposited by novel synthetic approaches that combined classic approaches as magnetron sputtering and plasma CVD with the sublimation of functional molecules. This methodology is

very effective to introduce a controlled amount of functional elements (i.e., rare earth cations of functional organic groups) in the growing film. Due to the full compatibility of the proposed methodology with optoelectronics processes the active layers will be directly incorporated in photonic structures as Bragg reflectors and photonic crystals to fabricate prototype devices. Thus, the LUMEN projects start with the development of new materials but also intend to study the functionality of devices that integrates these novel materials in real life conditions. These devices are intelligent label structures, up-converters and ion detectors.

Unión Europea
Fondo Europeo de Desarrollo Regional
"Una manera de hacer Europa"

JUNTA DE ANDALUCIA

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.

Unión Europea
Fondo Europeo de Desarrollo Regional
"Una manera de hacer Europa"

JUNTA DE ANDALUCIA

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

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

RESUMEN / ABSTRACT

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

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

cells of different nature, and includes cytotoxicity studies, with special attention to the role played by the morphology and chemistry of the particles.

Otros Proyectos / Other Projects

Nanopartículas basadas en matrices inorgánicas de tierras raras para aplicaciones biotecnólogicas

Código/Code:	201560E056
Periodo/Period:	01-06-2015 / 31-05-2018
Organismo Financiador/Financial source:	CSIC (Intramural)
Importe total/Total amount:	75.789 €
Investigador responsable/Research head:	Ana Isabel Becerro Nieto
Componentes/Research group:	Daniel González Mancebo, Manuel Ocaña Jurado

Convenios y Contratos / Contracts and Agreements

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

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

Investigación y desarrollo sobre recubrimientos ópticos para sistemas fotovoltáicos de baja concentración

Periodo/Period:	15-05-2014 / 31-03-2015
Organismo Financiador/Financial source:	ABENGOA Solar New Technologies, S.A.
Importe total/Total amount:	150.000€
Investigador responsable/Research head:	Hernán Míguez García
Componentes/Research group:	Mauricio Calvo Roggiani, Juan F. Galisteo López, Alberto Jiménez Solano, Miguel Anaya Martín

Acuerdos de colaboración para producir espejos de alta reflectavidad a los fines de reducir costes en la tecnología cilindro parabólica

Periodo/Period:	9-10-2014 / 8-07-2015
Organismo Financiador/Financial source:	ABENGOA SOLAR LLC
Importe total/Total amount:	30.000 €
Investigador responsable/Research head:	Hernán Míguez García

Componentes/Research group: Mauricio Calvo Roggiani, Juan F. Galisteo López, Gabriel Lozano Barbero, Alberto Jiménez Solano, Miguel Anaya Martín

MULTILAYER MIRROR

Periodo/Period: 15-03-2015 / 15-02-2016
Organismo Financiador/Financial source: ABENGOA SOLAR NEW TECHNOLOGIES, S.A.
Importe total/Total amount: 110.000€
Investigador responsable/Research head: Hernán Míguez García
Componentes/Research group: Mauricio Calvo Roggiani, Juan F. Galisteo López, Alberto Jiménez Solano, Miguel Anaya Martín

Caracterización de la respuesta químico-resistiva de composites plásticos conductores

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

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

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

Mechanically resistant superhydrophobic micro/nanostructures of stainless steel

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

EXPERIMENTOS EN GRANDES INSTALACIONES / LARGE FACILITY EXPERIMENTS

Sincrotrón ALBA / ALBA Synchrotron

Unraveling the wetting properties of TiO_2 under UV Irradiation
Following the Wetting of the one-dimensional photoactive surfaces

Código/Code:	2012010293
Periodo/Period:	2 periodos de 1 semana
Organismo Financiador/Financial source:	Sincrotrón ALBA
Instalación Científica:	ALBA línea BL24. Cerdanyola del Vallés, Barcelona (España)
Investigadores/Researchers:	Maria del Carmen López Santos, Juan Pedro Espinós Manzorro, Victor Rico Gavira, Ana Borrás Martos, Agustín R. González- Elipe

PATENTES / PATENTS

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

Inventores: Manuel Oliva Ramírez, Francisco Yubero Valencia, Agustín R. González-Elipe, Angel Barranco Quero
Tipo de Patente: Nacional
Número de Solicitud: 201531540
Fecha Solicitud: 27 de octubre de 2015
Entidad Titular: Consejo Superior de Investigaciones Científicas

Resorbable membrane for guided bone regeneration

Inventores: Agustín R. González-Elipe, Gabriel Castillo Dali, Francisco Yubero Valencia, María Aránzazu Díaz Cuenca, Angel Barranco Quero, Antonia Terriza Fernández
Tipo de Patente: Nacional
Número de Solicitud: 14/655279
Fecha Solicitud: 25 junio 2015
Entidad/es Titular/es: Universidad de Sevilla, Servicio Andaluz de Salud Junta de Andalucia, Universidad de Cádiz, Consejo Superior de Investigaciones Científicas

Recubrimientos de metal negro plasmónico fabricado mediante deposición de pulverización catódica a incidencia rasante

Inventores: José Miguel García Martín (IMM-CNM), Rafael Alvarez Molina, Alberto Palmero Acebedo

País de inscripción: España
Número de Solicitud: PCT/ES/15/070516
Fecha Solicitud: 2 julio 2015
Entidad/es Titular/es: Consejo Superior de Investigaciones Científicas

Procedure for making, encryption labelling and optical coding

Inventores: José Miguel García Martín (IMM-CNM), Rafael Alvarez Molina, Alberto Palmero Acebedo
País de inscripción: España
Número de Solicitud: PCT/ES/15/070516
Fecha Solicitud: 2 julio 2015
Entidad/es Titular/es: Consejo Superior de Investigaciones Científicas

Nanostructured perovskite

Inventores: Juan Francisco Galisteo López, Miguel Anaya Martín, Hernán R. Míguez García, Mauricio Calvo Roggiani
País de inscripción: España
Número de Solicitud: EP15382285
Fecha Solicitud: 29 mayo 2015
Entidad/es Titular/es: Consejo Superior de Investigaciones Científicas

Heterojunction device

Inventores: Hernán R. Míguez García, Gabriel S. Lozano Barbero, Miguel Anaya Martín, Mauricio E. Calvo Roggiani
País de inscripción: España
Número de Solicitud: 15382026
Fecha Solicitud: 30 enero 2015
Entidad/es Titular/es: Consejo Superior de Investigaciones Científicas, Isis Innovation Lim.

Detector de partículas ionizantes

Inventores: Jorge Gil-Rostra, Agustín R. González-Elipe, Juan Pedro Espinós Manzorro, Francisco Yubero Valencia, Angel Barranco Quero, Javier Ferrer (CNA), José Cotrino Bautista
País de inscripción: España
Número de Solicitud: 300151813. PCT/ES2015/070149
Fecha Solicitud: marzo 2015
Entidad Titular: Consejo Superior de Investigaciones Científicas

Sístema y método de análisis del gas presente en el espacio interanular de receptores solares de tubo

Inventores: Guillermo Espinosa Rueda, Pedrto Castillero Durán, Francisco Yubero Valencia, Angel Barranco Quero, José Cotrino Bautista, Juan P. Espinós Manzorro, Agustín Rodríguez González-Elipe

País de inscripción: España
Número de Solicitud: P201530521
Fecha Solicitud: abril 2015
Entidad Titular: Abengoa Solar New Tecnologies

Implantes biocompatibles de titanio nanoestructurado con propiedades antibacterianas

Inventores: José Miguel García-Martín, Alberto Palmero Acebedo, Rafael Álvarez Molina, María Vallet Regi, Daniel Arcos Navarrete, Isabel Izquierdo Barba
País de inscripción: España
Número de Solicitud: PCT/ES2015/070345
Fecha Solicitud: abril 2015
Entidad Titular: Consejo Superior de Investigaciones Científicas, Universidad Complutense de Madrid

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

Highly Efficient Perovskite Solar Cells with Tunable Structural Color

W. Zhang, M. Anaya, G. Lozano, M.E. Calvo, M.B. Johnston, H. Míguez, H.J. Snaith
Nano Letters, **15** (2015) 1698-1702
March, 2015 | **DOI:** 10.1021/nl504349z

The performance of perovskite solar cells has been progressing over the past few years and efficiency is likely to continue to increase. However, a negative aspect for the integration of perovskite solar cells in the built environment is that the color gamut available in these materials is very limited and does not cover the green-to-blue region of the visible spectrum, which has been a big selling point for organic photovoltaics. Here, we integrate a porous photonic crystal (PC) scaffold within the photoactive layer of an opaque perovskite solar cell following a bottom-up approach employing inexpensive and scalable liquid processing techniques. The photovoltaic devices presented herein show high efficiency with tunable color across the visible spectrum. This now imbues the perovskite solar cells with highly desirable properties for cladding in the built environment and encourages design of sustainable colorful buildings and iridescent electric vehicles as future power generation sources.

Sonogashira Cross-Coupling and Homocoupling on a Silver Surface: Chlorobenzene and Phenylacetylene on Ag(100)

Sanchez-Sanchez, C; Orozco, N; Holgado, JP; Beaumont, SK; Kyriakou, G; Watson, DJ; Gonzalez-Elipe, AR; Feria, L; Sanz, JF; Lambert, RM
Journal of the American Chemical Society, **137** (2015) 940-947
January, 2015 | **DOI:** 10.1021/ja5115584

Scanning tunneling microscopy, temperature-programmed reaction, near-edge X-ray absorption fine structure spectroscopy, and density functional theory calculations were used to study the adsorption and reactions of phenylacetylene and chlorobenzene on Ag(100). In the absence of solvent molecules and additives, these molecules underwent homocoupling and Sonogashira cross-coupling in an unambiguously heterogeneous mode. Of particular interest is the use of silver, previously unexplored, and chlorobenzene—normally regarded as relatively inert in such reactions. Both molecules adopt an essentially flat-lying conformation for which the observed and calculated adsorption energies are in reasonable agreement. Their magnitudes indicate that in both cases adsorption is predominantly due to dispersion forces for which interaction nevertheless leads to chemical activation and reaction. Both adsorbates exhibited pronounced island formation, thought to limit chemical activity under the conditions used and posited to occur at island boundaries, as was indeed observed in the case of phenylacetylene. The implications of these findings for the development of practical catalytic systems are considered.

Optical Description of Mesostructured Organic-Inorganic Halide Perovskite Solar Cells

Anaya, M; Lozano, G; Calvo, ME; Zhang, W; Johnston, MB; Snaith, HJ; Miguez, H
Journal of Physical Chemistry Letters, **6** (2015) 48-53
January, 2015 | **DOI:** 10.1021/jz502351s

Herein we describe both theoretically and experimentally the optical response of solution-processed organic–inorganic halide perovskite solar cells based on mesostructured scaffolds. We develop a rigorous theoretical model using a method based on the propagation of waves in layered media, which allows visualizing the way in which light is spatially distributed across the device and serves to quantify the fraction of light absorbed by each medium comprising the cell. The discrimination between productive and parasitic absorption yields an accurate determination of the internal quantum efficiency. State-of-the-art devices integrating mesoporous scaffolds infiltrated with perovskite are manufactured and characterized to support the calculations. This combined experimental and theoretical analysis provides a rational understanding of the optical behavior of perovskite cells and can be beneficial for the judicious design of devices with improved performance. Notably, our model justifies the presence of a solid perovskite capping layer in all of the highest efficiency perovskite solar cells based on thinner mesoporous scaffolds.

Environmental Effects on the Photophysics of Organic-Inorganic Halide Perovskites

Galisteo-Lopez, JF; Anaya, M; Calvo, ME; Miguez, H
Journal of Physical Chemistry Letters, **6** (2015) 2200-2205
June, 2015 | **DOI:** 10.1021/acs.jpclett.5b00785

The photophysical properties of films of organic-inorganic lead halide perovskites under different ambient conditions are herein reported. We demonstrate that their luminescent properties are determined by the interplay between photoinduced activation and darkening processes, which strongly depend on the atmosphere surrounding the samples. We have isolated oxygen and moisture as the key elements in each process, activation and darkening, both of which involve the interaction with photogenerated carriers. These findings show that environmental factors play a key role in the performance of lead halide perovskites as efficient luminescent materials.

Fine Tuning the Emission Properties of Nanoemitters in Multilayered Structures by Deterministic Control of their Local Photonic Environment

Alberto Jiménez-Solano, Juan Francisco Galisteo-López and Hernán Míguez
Small, **11** (2015) 2727-2732
June, 2015 | **DOI:** 10.1002/smll.201402898

Deterministic control on the dynamics of organic nanoemitters is achieved through precise control of its photonic environment. Resonators are fabricated by a combination of spin- and dip-coating techniques, which allows placement of the emitters at different positions within the sample, thus acting as a probe of the local density of states.

Nanocolumnar 1-dimensional TiO_2 photoanodes deposited by PVD-OAD for perovskite solar cell fabrication

Javier Ramos, F.; Oliva-Ramirez, Manuel; Nazeeruddin, Mohammad Khaja; Graetzel, Michael; Gonzalez-Elipe, Agustin R.; Ahmad, Shahzada
Journal of Materials Chemistry A, **3** (2015) 13291-13298
July, 2015 | **DOI:** 10.1039/c5ta02238j

Perovskite solar cells have attracted increasing interest among the photovoltaic community in the last few years owing to their unique properties and high efficiency. In the present work, we report the fabrication of perovskite solar cells based on highly ordered 1-dimensional porous TiO_2 photoanodes, which are uniform on a large area. These nanocolumnar porous TiO_2 photoanodes were deposited by physical vapor deposition in an oblique angle configuration (PVD-OAD) by varying the zenithal angle between the target and the substrate normal. Perovskite infiltration into these 1-dimensional nanocolumnar structures was homogeneous through the entire thickness of the porous layer as revealed by secondary ion mass spectroscopy studies. The fabricated solar cells, with an optimized thickness of the photoanode and with industrially accepted methods, will pave the way for easy implementation on a large scale.

Full solution processed mesostructured optical resonators integrating colloidal semiconductor quantum dots

Calvo, ME; Hidalgo, N; Schierholz, R; Kovacs, A; Fernandez, A; Bellino, MG; Soler-Illia, GJAA; Miguez, H
Nanoscale, **7** (2015) 16583-16589
October, 2015 | **DOI:** 10.1039/C5NR03977K

Herein we show a solution based synthetic pathway to obtain a resonant optical cavity with embedded colloidal semiconductor quantum dots (CSQDs). The optical cavity pore network, surrounded by two dense Bragg mirrors, was designed ad hoc to selectively host the quantum dots, while uncontrolled infiltration of those in the rest of the layered structure was prevented. Coupling between the optical resonant modes of the host and the natural emission of the embedded nanoparticles gives rise to the fine tuning of the luminescence spectrum extracted from the ensemble. Our approach overcomes, without the need for an encapsulating agent and exclusively by solution processing, the difficulties that arise from the low thermal and chemical stability of the CSQDs. It opens the route to achieving precise control over their location and hence over the spectral properties of light emitted by these widely employed nanomaterials. Furthermore, as the porosity of the cavity is preserved after infiltration, the system remains responsive to environmental changes, which provides an added value to the proposed structure.

Sunlight Absorption Engineering for Thermophotovoltaics: Contributions from the Optical Design

Miguez, H
ChemSusChem, **8** (2015) 786-788
March, 2015 | **DOI:** 10.1002/cssc.201403361

Nowadays, solar thermophotovoltaic systems constitute a platform in which sophisticated optical material designs are put into practice with the aim of achieving the long sought after dream of developing an efficient energy conversion device based on this concept. Recent advances demonstrate that higher efficiencies are at reach using photonic nanostructures amenable to mass production and scale-up.

Design and realization of transparent solar modules based on luminescent solar concentrators integrating nanostructured photonic crystals

Jimenez-Solano, A; Delgado-Sanchez, JM; Calvo, ME; Miranda-Munoz, JM; Lozano, G; Sancho, D; Sanchez-Cortezon, E; Miguez, H
Progress in Photovoltaics, **23** (2015) 1785-1792
December, 2015 | **DOI:** 10.1002/pip.2621

Herein, we present a prototype of a photovoltaic module that combines a luminescent solar concentrator integrating one-dimensional photonic crystals and in-plane $CuInGaSe_2$ (CIGS) solar cells. Highly uniform and wide-area nanostructured multilayers with photonic crystal properties were deposited by a cost-efficient and scalable liquid processing amenable to large-scale fabrication. Their role is to both maximize light absorption in the targeted spectral range, determined by the fluorophore employed, and minimize losses caused by emission at angles within the escape cone of the planar concentrator. From a structural perspective, the porous nature of the layers facilitates the integration with the thermoplastic polymers typically used to encapsulate and seal these modules. Judicious design of the module geometry, as well as of the optical properties of the dielectric mirrors employed, allows optimizing light guiding and hence photovoltaic performance while preserving a great deal of transparency. Optimized in-plane designs like the one herein proposed are of relevance for building integrated photovoltaics, as ease of fabrication, long-term stability and improved performance are simultaneously achieved.

Anisotropic In-Plane Conductivity and Dichroic Gold Plasmon Resonance in Plasma-Assisted ITO Thin Films e-Beam-Evaporated at Oblique Angles

Parra-Barranco, Julian; Garcia-Garcia, Francisco J.; Rico, Victor; Borras, Ana; Lopez-Santos, Carmen; Frutos, Fabian; Barranco, Angel; Gonzalez-Elipe, Agustin R.
ACS Applied Materials & Interfaces, **7** (2015) 10993-11001
May, 2015 | **DOI:** 10.1021/acsami.5b02197

ITO thin films have been prepared by electron beam evaporation at oblique angles (OA), directly and while assisting their growth with a downstream plasma. The films microstructure, characterized by scanning electron microscopy, atomic force microscopy, and glancing incidence small-angle X-ray scattering, consisted of tilted and separated nanostructures. In the plasma assisted films, the tilting angle decreased and the nanocolumns became associated in the form of bundles along the direction perpendicular to the flux of evaporated material. The annealed films presented different in-depth and sheet resistivity as confirmed by scanning conductivity measurements taken for the individual nanocolumns. In addition, for the plasma-assisted thin films, two different sheet resistance values were determined by measuring along the nanocolumn bundles or the perpendicular to it. This in-plane anisotropy induces the electrochemical deposition of elongated gold nanostructures. The obtained Au-ITO composite thin films were characterized by anisotropic plasmon resonance absorption and a dichroic behavior when examined with linearly polarized light.

Laser Treatment of Ag@ZnO Nanorods as Long-Life-Span SERS Surfaces

Macias-Montero, M; Pelaez, RJ; Rico, VJ; Saghi, Z; Midgley, P; Afonso, CN; Gonzalez-Elipe, AR; Borras, A
ACS Applied Materials & Interfaces, **7** (2015) 2331-2339
February, 2015 | **DOI:** 10.1021/am506622x

UV nanosecond laser pulses have been used to produce a unique surface nanostructuration of Ag@ZnO supported nanorods (NRs). The NRs were fabricated by plasma enhanced chemical vapor deposition (PECVD) at low temperature applying a silver layer as promoter. The irradiation of these structures with single nanosecond pulses of an ArF laser produces the melting and reshaping of the end of the NRs that aggregate in the form of bundles terminated by melted ZnO spherical particles. Well-defined silver nanoparticles (NPs), formed by phase separation at the surface of these melted ZnO particles, give rise to a broad plasmonic response consistent with their anisotropic shape. Surface enhanced Raman scattering (SERS) in the as-prepared Ag@ZnO NRs arrays was proved by using a Rhodamine 6G (Rh6G) chromophore as standard analyte. The surface modifications induced by laser treatment improve the stability of this system as SERS substrate while preserving its activity.

Plasma reforming of methane in a tunable ferroelectric packed-bed dielectric barrier discharge reactor

Montoro-Damas, AM; Brey, JJ; Rodriguez, MA; Gonzalez-Elipe, AR; Cotrino, J
Journal of Power Sources, **296** (2015) 268-275
November, 2015 | **DOI:** 10.1016/j.jpowsour.2015.07.038

In a tunable circular parallel plate dielectric barrier discharge reactor with pellets of a ferroelectric material separating the electrodes we investigate the plasma reforming of methane trying to maximize both the reaction yield and the energetic efficiency of the process. The geometrical configuration of the reactor (gap between electrodes, active electrode area) and the ferroelectric pellet size have been systematically varied to determine their influence on the process efficiency. The comparison between wet (with H_2O as reactant), oxidative (with O_2), and dry (with CO_2) reforming reactions reveals a higher efficiency for the former with CO + H_2 as main reaction products. The maximum energetic efficiency EE, defined as the produced number of litres of H_2 per kWh, found for optimized working conditions at low-level applied power is higher than the up to date best-known results. A comprehensive discussion of the influence of the different parameters affecting the reaction yield is carried out.

Nanocolumnar coatings with selective behavior towards osteoblast and Staphylococcus aureus proliferation

Izquierdo-Barba, Isabel; Miguel Garcia-Martin, Jose; Alvarez, Rafael; Palmero, Alberto; Esteban, Jaime; Perez-Jorge, Concepcion; Arcos, Daniel; Vallet-Regi, Maria

Acta Biomaterialia, **15** (2015) 20-28
March, 2015 | **DOI:** 10.1016/j.actbio.2014.12.023

Bacterial colonization and biofilm formation on orthopedic implants is one of the worst scenarios in orthopedic surgery, in terms of both patient prognosis and healthcare costs. Tailoring the surfaces of implants at the nanoscale to actively promote bone bonding while avoiding bacterial colonization represents an interesting challenge to achieving better clinical outcomes. Herein, a Ti6Al4V alloy of medical grade has been coated with Ti nanostructures employing the glancing angle deposition technique by magnetron sputtering. The resulting surfaces have a high density of nanocolumnar structures, which exhibit strongly impaired bacterial adhesion that inhibits biofilm formation, while osteoblasts exhibit good cell response with similar behavior to the initial substrates. These results are discussed on the basis of a "lotus leaf effect" induced by the surface nanostructures and the different sizes and biological characteristics of osteoblasts and Staphylococcus aureus.

Biocompatible Films with Tailored Spectral Response for Prevention of DNA Damage in Skin Cells

Nunez-Lozano, R; Pimentel, B; Castro-Smirnov, JR; Calvo, ME; Miguez, H; de la Cueva-Mendez, G

Advanced Healthcare Materials, **4** (2015) 1944-1948
September, 2015 | **DOI:** 10.1002/adhm.201500223

A hybrid nanostructured organic–in-organic biocompatible film capable of efficiently blocking a preselected range of ultraviolet light is designed to match the genotoxic action spectrum of human epithelial cells. This stack protects cultured human skin cells from UV-induced DNA lesions. As the shielding mechanism relies exclusively on reflection, the secondary effects due to absorption harmful radiation are prevented.

An Optically Controlled Microscale Elevator Using Plasmonic Janus Particles

Nedev, S; Carretero-Palacios, S; Kuhler, P; Lohmuller, T; Urban, AS; Anderson, LJE; Feldmann, J
ACS Photonics, **2** (2015) 491-496
April, 2015 | **DOI:** 10.1021/ph500371z

In this article, we report how Janus particles, composed of a silica sphere with a gold half-shell, can be not only stably trapped by optical tweezers but also displaced controllably along the axis of the laser beam through a complex interplay between optical and thermal forces. Scattering forces orient the asymmetric particle, while strong absorption on the metal side induces a thermal gradient, resulting in particle motion. An increase in the laser power leads to an upward motion of the particle, while a decrease leads to a downward motion. We study this reversible axial displacement, including a hysteretic jump in the particle position that is a result of the complex pattern of a tightly focused laser beam structure above the focal plane. As a first application we simultaneously trap a spherical gold nanoparticle and show that we can control the distance between the two particles inside the trap. This photonic micron-scale "elevator" is a promising tool for thermal force studies, remote sensing, and optical and thermal micromanipulation experiments.

Electrochemical activation of an oblique angle deposited Cu catalyst film for H-2 production

Gonzalez-Cobos, J; Rico, VJ; Gonzalez-Elipe, AR; Valverde, JL; de Lucas-Consuegra, A
Catalysis Science & Technology, **5** (2015) 2203-2214
May, 2015 | **DOI:** 10.1039/c4cy01524j

A novel Cu catalyst film was prepared by oblique angle physical vapour deposition (OAD) on a K-βAl_2O_3 solid electrolyte (alkaline ionic conductor) for catalytic/electrocatalytic purposes. This technique allowed us to obtain a highly porous and electrically conductive Cu catalyst electrode which was tested in the partial oxidation of methanol (POM) reaction for H_2 production and its catalytic activity was in situ enhanced via electrochemical promotion of catalysis (EPOC). The electropromotional effect was reversible and reproducible, and allowed us to increase both hydrogen and methyl formate production rates by almost three times under optimal promotion conditions (320 °C, 2.2 × 10−7 mol of K+ transferred). The observed promotional effect was attributed to a decrease in the Cu catalyst work function as a consequence of the controlled migration of electropositive K+ ions which favoured the chemisorption of electron acceptor molecules (O_2) at the expense of the electron donor ones (CH_3OH). Under the reaction conditions these ions formed some kinds of potassium surface compounds as demonstrated by SEM, EDX and XPS post-reaction characterization analyses. The obtained results demonstrate the interest of the used catalyst-electrode preparation technique for the electrochemical activation of non-noble metal catalyst films.

Flexible Distributed Bragg Reflectors from Nanocolumnar Templates

Calvo, ME; Gonzalez-Garcia, L; Parra-Barranco, J; Barranco, A; Jimenez-Solano, A; Gonzalez-Elipe, AR; Miguez, H
Advanced Optical Materials, **3** (2015) 171-175
February, 2015 | **DOI:** 10.1002/adom.201400338

A flexible distributed Bragg reflector is made by the infiltration of a nanocolumnar array with polydimethyl siloxane oligomers. The high optical reflectance displayed by the final material is a direct consequence of the high refractive index contrast of the columnar layers whereas the structural stability is due to the polymer properties.

Adaptable Ultraviolet Reflecting Polymeric Multilayer Coatings of High Refractive Index Contrast

Smirnov, JRC; Ito, M; Calvo, ME; Lopez-Lopez, C; Jimenez-Solano, A; Galisteo-Lopez, JF; Zavala-Rivera, P; Tanaka, K; Sivaniah, E; Miguez, H
Advanced Optical Materials, **3** (2015) 1633-1639
November, 2015 | **DOI:** 10.1002/adom.201500209

A synthetic route is demonstrated to build purely polymeric nanostructured multilayer coatings, adaptable to arbitrary surfaces, and capable of efficiently blocking by reflection a targeted and tunable ultraviolet (UV) range. Reflection properties are determined by optical interference between UV light beams reflected at the interfaces between polystyrene layers of different porosity and hence refractive index. As no dopant absorber intervenes in the shielding effect, polymer degradation effects are prevented. Alternated porosity results from the modulation of photochemical effects at the few tens of nanometers length scale, combined with the collective osmotic shock induced during the processing of the precursor diblock copolymer film. Experimental evidence of the application of this method to coat rough surfaces with smooth and conformal UV protecting films is provided.

New Copper wide range nanosensor electrode prepared by physical vapor deposition at oblique angles for the non-enzimatic determination of glucose

Salazar, P; Rico, V; Rodriguez-Amaro, R; Espinos, JP; Gonzalez-Elipe, AR
Electrochimica Acta, **169** (2015) 195-201
July, 2015 | **DOI:** 10.1016/j.electacta.2015.04.092

In this work a novel Cu nanostructured electrode is presented. Cu tilted nanocolumnar and porous thin films have been prepared by physical vapor deposition (PVD) in an oblique angle configuration and characterized by different techniques. Cyclic voltammetry and amperometry were used to study the sensing ability of the copper films deposited on ITO to quantitatively determine glucose and to optimize the experimental conditions of detection. Scanning electron microscopy data revealed that the film microstructure consists of tilted nanocolumns of around 70 nm of diameter and an inclination of 65° with respect to the surface normal that extend through the total thickness of the layer of ca. 300 nm. X ray photoelectron spectroscopy and Raman, used to determine the oxidation state of Cu, revealed that an oxy/hydroxide external layer formed around the nanocolumns is the active phase responsible for the electrocatalytic detection of glucose. Under optimized conditions, the CuO/Cu nanoporous/ITO electrode presented a sensitivity of 1.41 A mol dm−3 cm−2 (R2:0.999) with a limit of detection of 0.36 μmol dm−3 and a reproducibility of 3.42%.The selectivity of the proposed sensor was checked against various interferences, including physiological compounds, different sugars and ethanol, thereby showing excellent anti-interference properties. The CuO/Cu nanoporous/ITO electrode was also used successfully to determine glucose in blood samples showing a performance comparable to that of a commercial glucometer. An extended working range covering from 1 to 5 × 10−3 mol dm−3 was determined for these sensor films which, in this way, could be applied for different analytical purposes including agro industrial liquids.

A novel and improved surfactant-modified Prussian Blue electrode for amperometric detection of free chlorine in water

Salazar, Pedro; Martin, Miriam; Garcia-Garcia, Francisco J.; Luis Gonzalez-Mora, Jose; Gonzalez-Elipe, Agustin R.
Sensors and Actuators B: Chemical, **213** (2015) 116-123
July, 2015 | **DOI:** 10.1016/j.snb.2015.02.092

A surfactant-modified Prussian Blue (PB) electrochemical sensor has been developed. Benzethonium was used to assist the electrodeposition of PB onto a glassy carbon electrode (GCE). The surface coverage ([View the MathML source]) was 7.75 × 10−8 mol cm−2, five times higher than the value obtained in the absence of surfactant, and the film thickness of ca. 123 nm. SEM, EDX, Raman were used to characterize the electrodes while their electrochemical analysis proved a superior performance for the surfactant modified PB film. Cyclic voltammetry and amperometry were used to study the sensor ability to detect chlorine, and the main experimental variables were optimized. Under optimized conditions, the sensor presented a sensitivity of 12 µA ppm−1 cm−2, a linear range from 9 ppb to 10 ppm and a reproducibility of 4.2%. For the first time, we proved the sensor performance for real applications. Thus, chlorine was determined in tap water and the obtained concentrations validated with a standard colorimetric method. The obtained results showed that our sensor is highly performant and reliable for applications involving determinations of environmental residual chlorine.

Rapid Legionella pneumophila determination based on a disposable core–shell Fe3O4@poly(dopamine) magnetic nanoparticles immunoplatform

Martin, M; Salazar, P; Jimenez, C; Lecuona, M; Ramos, MJ; Ode, J; Alcoba, J; Roche, R; Villalonga, R; Campuzano, S; Pingarron, JM; Gonzalez-Mora, JL
Analytica Chimica Acta, **887** (2015) 51-58
August, 2015 | **DOI:** 10.1016/j.aca.2015.05.048

A novel amperometric magnetoimmunoassay, based on the use of core–shell magnetic nanoparticles and screen-printed carbon electrodes, was developed for the selective determination of Legionella pneumophila SG1. A specific capture antibody (Ab) was linked to the poly(dopamine)–modified magnetic nanoparticles (MNPs@pDA-Ab) and incubated with bacteria. The captured bacteria were sandwiched using the antibody labeled with horseradish peroxidase (Ab-HRP), and the resulting MNPs@pDA-Ab-Legionella neumophila-Ab-HRP were captured by a magnetic field on the electrode surface. The amperometric response measured at −0.15 V vs. Ag pseudo-reference electrode of the SPCE after the addition of H2O2 in the presence of hydroquinone (HQ) was used as transduction signal. The achieved limit of detection, without pre-concentration or pre-enrichment steps, was 104 Colony Forming Units (CFUs) mL−1. The method showed a good selectivity and the MNPs@pDA-Ab exhibited a good stability during 30 days. The possibility of detecting L. pneumophila at 10 CFU mL−1 level in less than 3 h, after performing a membrane-based preconcentration step, was also demonstrated.

Nanolevitation Phenomena in Real Plane-Parallel Systems Due to the Balance between Casimir and Gravity Forces

Esteso, V; Carretero-Palacios, S; Miguez, H
Journal of Physical Chemistry C, **119** (2015) 5663-5670
March, 2015 | **DOI:** 10.1021/jp511851z

We report on the theoretical analysis of equilibrium distances in real plane-parallel systems under the influence of Casimir and gravity forces at thermal equilibrium. Due to the balance between these forces, thin films of Teflon, silica, or polystyrene in a single-layer configuration and immersed in glycerol stand over a silicon substrate at certain stable or unstable positions depending on the material and the slab thickness. Hybrid systems containing silica and polystyrene, materials which display Casimir forces and equilibrium distances of opposite nature when considered individually, are analyzed in either bilayer arrangements or as composite systems made of a homogeneous matrix with small inclusions inside. For each configuration, equilibrium distances and their stability can be adjusted by fine-tuning of the volume occupied by each material. We find the specific conditions under which nanolevitation of realistic films should be observed. Our results indicate that thin films of real materials in plane-parallel configurations can be used to control suspension or stiction phenomena at the nanoscale.

Absorption Enhancement in Organic-Inorganic Halide Perovskite Films with Embedded Plasmonic Gold Nanoparticles

Carretero-Palacios, S; Calvo, ME; Miguez, H
Journal of Physical Chemistry C, **119** (2015) 18635-18640
August, 2015 | **DOI:** 10.1021/acs.jpcc.5b06473

We report on the numerical analysis of solar absorption enhancement in organic-inorganic halide perovskite films embedding plasmonic gold nanoparticles. The effect of particle size and concentration is analyzed in realistic systems in which random particle location within the perovskite film and the eventual formation of dimers are also taken into account. We find a maximum integrated solar absorption enhancement of similar to 10% in perovskite films of 200 nm thickness and similar to 6% in 300 nm films, with spheres of radii 60 and 90 nm, respectively, in volume concentrations of around 10% in both cases. We show that the presence of dimers boosts the absorption enhancement up to,similar to 12% in the thinnest films considered. Absorption reinforcement arises from a double contribution of plasmonic near-field and scattering effects, whose respective weight can be discriminated and evaluated from the simulations.

Physiological Degradation Mechanisms of PLGA Membrane Films under Oxygen Plasma Treatment

Lopez-Santos, C; Terriza, A; Portoles, J; Yubero, F; Gonzalez-Elipe, AR
Journal of Physical Chemistry C, **119** (2015) 20446-20452

September, 2015 | **DOI:** 10.1021/acs.jpcc.5b05011

Degradation under simulated physiological conditions of poly(lactic-co-glycolic) (PLGA) copolymer membrane films subjected to an oxygen plasma treatment compared to its “as prepared” state has been studied by gas cluster ion beam assisted X-ray photoelectron spectroscopy for chemical depth profiling analysis. This investigation is complemented with atomic force microscopy, weight loss measurements, and visual inspection of the films at the different stages of the degradation process. The obtained results show that the carbon functional groups of the PLGA membrane films undergo a heterogeneous hydrolytic degradation to different rates depending on the plasma pretreatment. The content of glycolic groups (GA) in untreated PLGA samples immersed for 3 weeks in a phosphate-buffered saline solution decreased at the surface, whereas the ratio between glycolic and lactic units (LA) did not vary in the inner regions (~400 nm depth) of the degraded membrane films. By contrast, oxygen plasma pretreatment enhances the degradation efficiency and causes that both lactic and glycolic functional components decreased at the surface and in the interior of the film, although with less prevalence for the lactic units that present a comparatively higher resistance to degradation.

Ultraviolet Pretreatment of Titanium Dioxide and Tin-Doped Indium Oxide Surfaces as a Promoter of the Adsorption of Organic Molecules in Dry Deposition Processes: Light Patterning of Organic Nanowires

Oulad-Zian, Y; Sanchez-Valencia, JR; Parra-Barranco, J; Hamad, S; Espinos, JP; Barranco, A; Ferrer, J; Coll, M; Borras, A
Langmuir, **31** (2015) 8294-8302
August, 2015 | **DOI:** 10.1021/acs.langmuir.5b01572

In this article we present the preactivation of TiO_2 and ITO by UV irradiation under ambient conditions as a tool to enhance the incorporation of organic molecules on these oxides by evaporation at low pressures. The deposition of p-stacked molecules on TiO_2 and ITO at controlled substrate temperature and in the presence of Ar is thoroughly followed by SEM, UV-vis, XRD, RBS, and photoluminescence spectroscopy, and the effect is exploited for the patterning formation of small-molecule organic nanowires (ONWs). X-ray photoelectron spectroscopy (XPS) in situ experiments and molecular dynamics simulations add critical information to fully elucidate the mechanism behind the increase in the number of adsorption centers for the organic molecules. Finally, the formation of hybrid organic/inorganic semiconductors is also explored as a result of the controlled vacuum sublimation of organic molecules on the open thin film microstructure of mesoporous TiO_2.

Core-shell polydopamine magnetic nanoparticles as sorbent in micro-dispersive solid-phase extraction for the determination of estrogenic compounds in water samples prior to high-performance liquid chromatography-mass spectrometry analysis

Socas-Rodriguez, B; Hernandez-Borges, J; Salazar, P; Martin, M; Rodriguez-Delgado, MA
Journal of Chromatography A, **1397** (2015) 1-10
June, 2015 | **DOI:** 10.1016/j.chroma.2015.04.010

In this work, core-shell Fe_3O_4@poly(dopamine) magnetic nanoparticles (m-NPs) were prepared and characterized in our laboratory and applied as sorbents for the magnetic-micro solid phase extraction (m-mu SPE) of twelve estrogenic compounds of interest (i.e. 17 alpha-estradiol, 17 beta-estradiol, estrone, hexestrol, 17 alpha-ethynylestradiol, diethylstibestrol, dienestrol, zearalenone, alpha-zearalanol,beta-zearalanol, alpha-zearalenol and beta-zearalenol) from different water samples. Separation, determination and quantification were achieved by high-performance liquid chromatography coupled to ion trap mass spectrometry with electrospray ionization. NPs@poly(dopamine) were synthesized by a chemical coprecipitation procedure and characterized by different surface characterization techniques (X-ray diffraction, X-ray photoelectron spectroscopy, thermogravimetric analysis, transmission and scanning electron microscopy, infrared and Raman spectroscopy, vibrating sample magnetometry, microelectrophoresis and adsorption/desorption isotherms). Parameters affecting the extraction efficiency of m-mu SPE (i.e. polymerization time, pH of the sample, extraction and elution conditions) were studied and optimized. The methodology was validated for Milli-Q, mineral, tap and wastewater using 2-methoxyestradiol as internal standard, obtaining recoveries ranging from 70 to 119% with relative standard deviation values lower than 20% and limits of quantification in the range 0.02-1.1 mu g/L.

$BaGa_4O_7$, a new $A_3BC_{10}O_{20}$ crystalline phase: synthesis, structural determination and luminescence properties

Boyer, Marina; Veron, Emmanuel; Becerro, Ana Isabel; Porcher, Florence; Suchomel, Matthew R.; Matzen, Guy; Allix, Mathieu
CrystEngComm, **17** (2015) 6127-6135
August, 2015 | **DOI:** 10.1039/C5CE01101A

The synthesis, structural determination and luminescence properties of a new barium gallate, $BaGa_4O_7$, are reported. This crystalline material can uniquely be obtained by direct cooling from the molten state. The crystallographic structure was determined using a combination of electron, synchrotron and neutron powder diffraction data. $BaGa_4O_7$ crystallizes in the monoclinic I2/mspace group with a = 15.0688(1) Å, b = 11.7091(1) Å, c = 5.1429(2) Å and β = 91.0452(2)° and can be described as an original member of the A3BC10O20 family. Atypical for this A3BC10O20 structural framework, $BaGa_4O_7$ is found to contain exclusively divalent and trivalent cations. In order to maintain overall electroneutrality, disordered defect-type partial substitution of gallium and oxygen ions on barium sites occurs within the pentagonal channels of $BaGa_4O_7$. Thanks to the flexibility of this structural framework, $BaGa_4O_7$ can be heavily doped with europium and thus is shown to exhibit strong orange-red luminescence emission at 618 nm under 393 nm excitation.

Single-step fabrication process of 1-D photonic crystals coupled to nanocolumnar TiO_2 layers to improve DSC efficiency

Gonzalez-Garcia, L; Colodrero, S; Miguez, H; Gonzalez-Elipe, AR
Optics Express, **23** (2015) A1642-A1650
November, 2015 | **DOI:** 10.1364/OE.23.0A1642

The present work proposes the use of a TiO_2 electrode coupled to a one-dimensional photonic crystal (1DPC), all formed by the sequential deposition of nanocolumnar thin films by physical vapor oblique angle deposition (PV-OAD), to enhance the optical and electrical performance of DSCs while transparency is preserved. We demonstrate that this approach allows building an architecture combining a non-dispersive 3 µm of TiO_2 electrode and 1 µm TiO_2-SiO_2 1DPC, both columnar, in a single-step process. The incorporation of the photonic structure is responsible for a rise of 30% in photovoltaic efficiency, as compared with a transparent cell with a single TiO_2 electrode. Detailed analysis of the spectral dependence of the photocurrent demonstrates that the 1DPC improves light harvesting efficiency by both back reflection and optical cavity modes confinement within the TiO_2 films, thus increasing the overall performance of the cell.

Active vacuum brazing of CNT films to metal substrates for superior electron field emission performance

Longtin, R; Sanchez-Valencia, JR; Shorubalko, I; Furrer, R; Hack, E; Elsener, H; Groning, O; Greenwood, P; Rupesinghe, N; Teo, K; Leinenbach, C; Groning, P

Science and Technology of Advanced Materials, **16** (2015) 015005 (11 pp)

February, 2015 | **DOI:** 10.1088/1468-6996/16/1/015005

The joining of macroscopic films of vertically aligned multiwalled carbon nanotubes (CNTs) to titanium substrates is demonstrated by active vacuum brazing at 820 degrees C with a Ag-Cu-Ti alloy and at 880 degrees C with a Cu-Sn-Ti-Zr alloy. The brazing methodology was elaborated in order to enable the production of highly electrically and thermally conductive CNT/metal substrate contacts. The interfacial electrical resistances of the joints were measured to be as low as 0.35 Omega. The improved interfacial transport properties in the brazed films lead to superior electron fieldemission properties when compared to the as-grown films. An emission current of 150 mu A was drawn from the brazed nanotubes at an applied electric field of 0.6 V mu m(-1). The improvement in electron field-emission is mainly attributed to the reduction of the contact resistance between the nanotubes and the substrate. The joints have high re-melting temperatures up to the solidus temperatures of the alloys; far greater than what is achievable with standard solders, thus expanding the application potential of CNT films to high-current and high-power applications where substantial frictional or resistive heating is expected.

Quick synthesis, functionalization and properties of uniform, luminescent $LuPO_4$-based nanoparticles

Becerro, AI; Ocana, M

RSC Advances, **44** (2015) 34517-34524

January, 2015 | **DOI:** 10.1039/C5RA05305F

The aim of this study was to find a surfactant-free method for the synthesis of uniform Eu:$LuPO_4$ nanophosphors which are able to form stable colloidal suspensions in aqueous media. Uniform, ovoid Eu-doped $LuPO_4$ fluorescent nanoparticles were obtained after aging for 30 minutes at 180 °C a butylene glycol solution containing, exclusively, lutetium acetate, europium acetate and H_3PO_4. XRD and digital diffraction patterns of HRTEM images suggested that the particles were single crystalline in nature with the c-axis of the unit cell parallel to the long particle axis. The luminescence study revealed that the optimum doping level was 5 molar%. The latter particles

(85 nm × 40 nm dimensions) were functionalized with polyacrylic acid and their colloidal stability in two different biological buffers was demonstrated to persist for at least 15 days.

Synergistic strategies for the preparation of highly efficient dye-sensitized solar cells on plastic substrates: combination of chemical and physical sintering

Li, Y; Yoo, K; Lee, DK; Kim, JY; Son, HJ; Kim, JH; Lee, CH; Miguez, H; Ko, MJ
RSC Advances, **5** (2015) 76795-76803
October, 2015 | **DOI:** 10.1039/C5RA10290A

Preparation of well-interconnected TiO_2 electrodes at low temperature is critical for the fabrication of highly efficient dye-sensitized solar cells (DSCs) on plastic substrates. Herein we explore a synergistic approach using a combination of chemical and physical sintering. We formulate a binder-free TiO_2 paste based on "nanoglue" as the chemical sintering agent, and use it to construct a photoelectrode on plastic by low-temperature physical compression to further improve the connectivity of TiO_2 films. We systematically investigated the factors affecting the photovoltaic performance and found the conditions to achieve electron diffusion lengths as long as 25 µm and charge collection efficiencies as high as 95%, as electrochemical impedance spectroscopy measurements indicate. We apply this approach to obtain a DSC deposited on plastic displaying 6.4% power conversion efficiency based on commercial P25 titania particles.

Porous, robust highly conducting Ni-YSZ thin film anodes prepared by magnetron sputtering at oblique angles for application as anodes and buffer layers in solid oxide fuel cells

Garcia-Garcia, Francisco J.; Yubero, Francisco; Gonzalez-Elipe, Agustin R.; Balomenou, Stella P.; Tsiplakides, Dimitris; Petrakopoulou, Ioanna; Lambert, Richard M.
International Journal of Hydrogen Energy, **40** (2015) 7382-7387
June, 2015 | **DOI:** 10.1016/j.ijhydene.2015.04.001

Uniform, highly porous, columnar thin films incorporating YSZ and NiO prepared by magnetron sputtering with deposition at glancing incidence exhibited stoichiometries close to that of the Y-Zr-Ni sputter target. Characterization by means of SEM, XRD, XPS and RBS revealed that the uniformly distributed nickel component in the as-deposited films consisted of NiO, and that the YSZ component was essentially amorphous. Annealing such films at 850 degrees C in hydrogen resulted in crystallization of the YSZ phase with preservation of the columnar morphology, while the NiO underwent reduction to metallic Ni, which partially segregated to the film surface. The hydrogen-annealed thin film anodes exhibited high conductivity, comparable to that of conventionally-prepared anodes, in both hydrogen and hydrogen/water mixtures at temperatures relevant to SOFC operation. They were also robust against strain-induced separation from the substrate under limited thermal cycling in both oxidizing and reducing atmospheres and are promising candidates for use as anodes in their own right and as strain-accommodating buffer layers between conventional anodes and the electrolyte for use in SOFC applications.

Optical properties of zirconium oxynitride films: The effect of composition, electronic and crystalline structures

Carvalho, P; Borges, J; Rodrigues, MS; Barradas, NP; Alves, E; Espinos, JP; Gonzalez-Elipe, AR; Cunha, L; Marques, L; Vasilevskiy, MI; Vaz, F

Applied Surface Science, **358** (2015) 660-669

December, 2015 | **DOI:** 10.1016/j.apsusc.2015.09.129

This work is devoted to the investigation of zirconium oxynitride (ZrOxNy) films with varied optical responses prompted by the variations in their compositional and structural properties. The films were prepared by dc reactive magnetron sputtering of Zr, using Ar and a reactive gas mixture of N-2 + O-2 (17:3). The colour of the films changed from metallic-like, very bright yellow-pale and golden yellow, for low gas flows to red-brownish for intermediate gas flows. Associated to this colour change there was a significant decrease of brightness. With further increase of the reactive gas flow, the colour of the samples changed from red-brownish to dark blue or even to interference colourations. The variations in composition disclosed the existence of four different zones, which were found to be closely related with the variations in the crystalline structure. XRD analysis revealed the change from a B1 NaCl face-centred cubic zirconium nitride-type phase for films prepared with low reactive gas flows, towards a poorly crystallized over-stoichiometric nitride phase, which may be similar to that of Zr_3N_4 with some probable oxygen inclusions within nitrogen positions, for films prepared with intermediate reactive gas flows. For high reactive gas flows, the films developed an oxynitride-type phase, similar to that of gamma-Zr_2ON_2 with some oxygen atoms occupying some of the nitrogen positions, evolving to a ZrO_2 monoclinic type structure within the zone where films were prepared with relatively high reactive gas flows. The analysis carried out by reflected electron energy loss spectroscopy (REELS) revealed a continuous depopulation of the d-band and an opening of an energy gap between the valence band (2p) and the Fermi level close to 5 eV. The ZrN-based coatings (zone land II) presented intrinsic colourations, with a decrease in brightness and a colour change from bright yellow to golden yellow, red brownish and dark blue. Associated to these changes, there was also a shift of the reflectivity minimum to lower energies, with the increase of the non-metallic content. The samples lying in the two last zones (zone III, oxynitride and zone IV, oxide films) revealed a typical semi-transparent-optical behaviour showing interference-like colourations only due to the complete depopulation of the d band at the Fermi level. The samples lying in these zones presented also an increase of the optical bandgap from 2 to 3.6 eV.

Template-free synthesis and luminescent properties of hollow Ln:YOF (Ln = Eu or Er plus Yb) microspheres

Martinez-Castro, E; Garcia-Sevillano, J; Cusso, F; Ocana, M

Journal of Alloys and Compounds, **619** (2015) 44-51

January, 2015 | **DOI:** 10.1016/j.jallcom.2014.09.023

A method for the synthesis of hollow lanthanide doped yttrium oxyfluoride (YOF) spheres in the micrometer size range with cubic structure based on the pyrolysis at 600 degrees C of liquid aerosols generated from aqueous solutions containing the corresponding rare earth chlorides and trifluoroacetic acid has been developed. This procedure, which has been used for the first time for the synthesis of YFO based materials, is simpler and advantageous when compared with other methods usually employed for the production of hollow spheres since it does not require the use of sacrificial templates. In addition, it is continuous, which is desirable because of practical reasons. The procedure is also suitable for doping the YOF spheres with europium cations resulting in down converting red phosphors when activated with UV light, or for co-doping with both Er^{3+} and Yb^{3+} giving rise to up-converting phosphors, which emit intense red light under near infrared (NIR) irradiation. Because of their optical properties and hollow architecture, the developed materials may find applications in optoelectronic devices and biotechnology.

Efficient synthesis of ammonia from N-2 and H-2 alone in a ferroelectric packed-bed DBD reactor

Gomez-Ramirez, A; Cotrino, J; Lambert, RM; Gonzalez-Elipe, AR

Plasma Sources Science and Technology, **24** (2015) 065011

Decembe, 2015 | **DOI:** 10.1088/0963-0252/24/6/065011

A detailed study of ammonia synthesis from hydrogen and nitrogen in a planar dielectric barrier discharge (DBD) reactor was carried out. Electrical parameters were systematically varied, including applied voltage and frequency, electrode gap, and type of ferroelectric material ($BaTiO_3$ versus PZT). For selected operating conditions, power consumption and plasma electron density were estimated from Lissajous diagrams and by application of the Bolsig + model, respectively. Optical emission spectroscopy was used to follow the evolution of plasma species (NH*, N*, N-2(+) and N-2*) as a function of applied voltage with both types of ferroelectric material. PZT gave both greater energy efficiency and higher ammonia yield than $BaTiO_3$: 0.9 g NH3 kWh(-1) and 2.7% single pass N-2 conversion, respectively. This performance is substantially superior to previously published findings on DBD synthesis of NH_3 from N-2 and H-2 alone. The influence of electrical working parameters, the beneficial effect of PZT and the importance of controlling reactant residence time are rationalized in a reaction model that takes account of the principal process variables.

Modulating Low Energy Ion Plasma Fluxes for the Growth of Nanoporous Thin Films

Alvarez, Rafael; Lopez-Santos, Carmen; Ferrer, Francisco J.; Rico, Victor; Cotrino, Jose; Gonzalez-Elipe, Agustin R.; Palmero, Alberto

Plasma Process and Polymers, **12** (2015) 719-724

August, 2015 | **DOI:** 10.1002/ppap.201400209

The growth of nanoporous layers by plasma-assisted deposition techniques is strongly mediated by the ion fluxes in the reactor. To analyze their influence we have deposited different nanostructured thin films by the magnetron sputtering technique at oblique angles, modulating the ion fluxes in the plasma by tuning the frequency of the electromagnetic signal from pure DC to 160 kHz DC pulsed mode. In the DC case, ions possess energies below 5 eV and do not induce noticeable changes in the film structure. However, when the signal is pulsed, ions with energies up to 40 eV impinge on the film, decreasing the porosity of the layers and tilting down the porous/nanocolumnar structures. As a result, we demonstrate that the overall porosity of the layers and the tilt angle of the columns can be tailored as two independent morphological quantities.

Uniform Poly(acrylic acid)-Functionalized Lanthanide-Doped $LaVO_4$ Nanophosphors with High Colloidal Stability and Biocompatibility

Nunez, NO; Zambrano, P; Garcia-Sevillano, J; Cantelar, E; Rivera-Fernandez, S; de la Fuente, JM; Ocana, M

European Journal of Inorganic Chemistry, **27** (2015) 4546-4554

September, 2015 | **DOI:** 10.1002/ejic.201500265

Ln-doped (Ln = Eu or Nd) $LaVO_4$ nanoparticles functionalized with poly(acrylic acid) (PAA) were prepared from lanthanide and vanadate precursors in the presence of PAA by a simple one-pot method that consists of a homogeneous precipitation reaction in ethylene glycol/water at a moderate temperature (120 degrees C). The size of the nanoparticles could be modified in the 40-70 nm range by adjusting the amount of PAA added. The effects of the Eu and Nd contents of these nanomaterials on theirs optical properties (emission intensity and lifetime) were also analyzed to find the optimum nanophosphors. Finally, the nanoparticles showed negligible cytotoxicity for Vero cells at concentrations up to 0.05 mgmL(-1) and a high colloidal stability in physiological buffer solutions; therefore, they satisfy the most important requirements for in vitro biotechnological applications.

Biosynthesis of silver fine particles and particles decorated with nanoparticles using the extract of Illicium verum (star anise) seeds

Luna, Carlos; Chavez, V. H. G.; Diaz Barriga-Castro, Enrique; Nunez, Nuria O.; Mendoza-Resendez, Raquel

Spectrochimica Acta Part A-Molecular and Biomolecular Spectroscopy, **141** (2015) 43-50

April, 2015 | **DOI:** 10.1016/j.saa.2014.12.076

Given the upsurge of new technologies based on nanomaterials, the development of sustainable methods to obtain functional nanostructures has become an imperative task. In this matter, several recent researches have shown that the biodegradable natural antioxidants of several plant extracts can be used simultaneously as reducing and stabilizing agents in the wet chemical synthesis of metallic nanoparticles, opening new opportunities to design greener synthesis. However, the challenge of these new techniques is to produce stable colloidal nanoparticles with controlled particle uniformity, size, shape and aggregation state, in similar manner than the well-established synthetic methods. In the present work, colloidal metallic silver nanoparticles have been synthesized using silver nitrate and extracts of Illicium verum (star anise) seeds at room temperature in a facile one-step procedure. The resulting products were colloidal suspensions of two populations of silver nanoparticles, one of them with particle sizes of few nanometers and the other with particles of tens of nm. Strikingly, the variation of the $AgNO_3$/extract weight ratio in the reaction medium yielded to the variation of the spatial distribution of the nanoparticles: high $AgNO_3$/extract concentration ratios yielded to randomly dispersed particles, whereas for lower $AgNO_3$/extract ratios, the biggest particles appeared coated with the finest nanoparticles.-This biosynthesized colloidal system, with controlled particle aggregation states, presents plasmonic and SERS properties with potential applications in molecular sensors and nanophotonic devices.

Up-conversion in Er^{3+}/Yb^{3+} co-doped $LaPO_4$ submicron-sized spheres

Garcia-Sevillano, J.; Cantelar, E.; Cusso, F.; Ocana, M.
Optical Materials, **41** (2015) 104-107
March, 2015 | **DOI:** 10.1016/j.optmat.2014.10.022

Er^{3+}/Yb^{3+} co-doped materials have been extensively used for imaging in biomedical applications using either visible up-converted (UC) or near-infrared (NIR) emissions. The UC spectrum is composed mainly by two Erbium emissions in the green (2H11/2:4S3/2 → 4I15/2) and red (4F9/2 → 4I15/2) spectral range, while the NIR spectrum includes Er3+ (λ ~ 1.5 µm, 4I13/2 → 4I15/2) and Er^{3+}/Yb^{3+} (λ ~ 980 nm, 2F5/2 → 2F7/2(Yb^{3+}):4I11/2 → 4I15/2 (Er^{3+})) transitions; which relative intensities are dependent on several physical parameters. In the present work, we present the preparation and optical characterization of Er^{3+}/Yb^{3+} co-doped LaPO4 submicron-sized spheres. The luminescence (CW and pulsed) characteristics, after different post-annealing treatments, are studied. It is found that such treatments strongly increment the emission efficiency, possibly due to the suppression of residual impurities. After calcination at 1100 °C the material behaves as an excellent UC and NIR–NIR wavelength converter.

Uniform, luminescent Eu: LuF_3 nanoparticles

Becerro, AI; Gonzalez-Mancebo, D; Ocana, M
Journal of Nanoparticle Research, **17** (2015) 58
January, 2015 | **DOI:** 10.1007/s11051-015-2874-z

A simple procedure for the synthesis of orthorhombic, uniform, LuF_3 particles with two different morphologies (rhombus- and cocoon-like) and nanometer and sub-micrometer size, respectively, is reported. The method consists in the aging, at 120 °C for 2 h, a solution containing [BMIM]BF_4 ionic liquid (0.5 mL) and lutetium acetate (in the case of the rhombi) or lutetium nitrate (in the case of the cocoons) (0.02 M) in ethylene glycol (total volume 10 mL). This synthesis method was also adequate for the synthesis of Eu^{3+}-doped LuF_3 particles of both morphologies, whose luminescence properties were investigated in detail. The experimental observations reported herein suggest that these materials are suitable phosphors for optoelectronic as well as in vitro biotechnological applications

Free-Base Carboxyphenyl Porphyrin Films Using a TiO_2 Columnar Matrix: Characterization and Application as NO_2 Sensors

Roales, Javier; Pedrosa, Jose M.; Guillen, Maria G.; Lopes-Costa, Tania; Castillero, Pedro; Barranco, Angel; Gonzalez-Elipe, Agustin R.
Sensors, **15** (2015) 11118-11132
May, 2015 | **DOI:** 10.3390/s150511118

The anchoring effect on free-base carboxyphenyl porphyrin films using TiO_2 microstructured columns as a host matrix and its influence on NO_2 sensing have been studied in this work. Three porphyrins have been used: 5-(4-carboxyphenyl)10,15,20-triphenyl-21H,23H-porphyrin (MCTPP); 5,10,15,20-tetrakis(4-carboxyphenyl)-21H,23H-porphyrin (p-TCPP); and 5,10,15,20-tetrakis(3-carboxyphenyl)-21H,23H-porphyrin (m-TCPP). The analysis of UV-Vis spectra of MCTPP/TiO_2, p-TCPP/TiO_2 and m-TCPP/TiO_2 composite films has revealed that m-TCPP/TiO_2

films are the most stable, showing less aggregation than the other porphyrins. IR spectroscopy has shown that m-TCPP is bound to TiO_2 through its four carboxylic acid groups, while p-TCPP is anchored by only one or two of these groups. MCTPP can only be bound by one carboxylic acid. Consequently, the binding of p-TCPP and MCTPP to the substrate allows them to form aggregates, whereas the more fixed anchoring of m-TCPP reduces this effect. The exposure of MCTPP/TiO_2, p-TCPP/TiO_2 and m-TCPP/TiO_2 films to NO_2 has resulted in important changes in their UV-Vis spectra, revealing good sensing capabilities in all cases. The improved stability of films made with m-TCPP suggests this molecule as the best candidate among our set of porphyrins for the fabrication of NO_2 sensors. Moreover, their concentration-dependent responses upon exposure to low concentrations of NO_2 confirm the potential of m-TCPP as a NO2 sensor.

Amperometric magnetobiosensors using poly(dopamine)-modified Fe_3O_4 magnetic nanoparticles for the detection of phenolic compounds

Martin, M; Salazar, P; Campuzano, S; Villalonga, R; Pingarron, JM; Gonzalez-Mora, JL

Analytical Methods, **7** (2015) 8801-8808
October, 2015 | **DOI:** 10.1039/C5AY01996F

The synthesis of poly(dopamine)-modified magnetic nanoparticles (MNPs) and their application in preparing electrochemical enzyme biosensors that are useful to detect phenolic compounds is reported in this work. MNPs of about 16 nm were synthesized by a co-precipitation method and conveniently modified with poly(dopamine). Non-modified and modified MNPs were characterized using X-ray photoelectron spectroscopy (XPS), Raman and infrared spectroscopy, X-ray diffraction (XRD) and atomic force microscopy (AFM). Horseradish peroxidase (HRP) was covalently immobilized onto the surface of the poly(dopamine)-modified MNPs via Michael addition and/or Schiff base formation and used to construct a biosensor for phenolic compounds by capturing the HRP-modified-nanoparticles onto the surface of a magnetic-modified glassy carbon electrode (GCE). Cyclic voltammetry and amperometry were used to study the electrochemical and analytical properties of the biosensor using hydroquinone (HQ) as a redox probe. Among the different phenolic compounds studied, the biosensor exhibited higher sensitivity for HQ, 1.38 A M−1 cm−2, with limits of detection and quantification of 0.3 and 1.86 µM, respectively. The analytical biosensor performance for HQ and 2-aminophenol compared advantageously with those of previous phenolic biosensors reported in the literature.

Microstructure of mixed oxide thin films prepared by magnetron sputtering at oblique angles

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

Thin Solid Films, **591** (2015) 330-335
September, 2015 | **DOI:** 10.1016/j.tsf.2015.01.058

Several mixed oxide thin film series of samples (Si–Co–O, Si–Ni–O, Si–W–O) have been prepared by reactive magnetron sputtering at oblique angle geometries. The paper focuses on the description of microstructure of the films as a function of their stoichiometry. It is found that for identical process parameters (gas mixture, pressure, magnetron-substrate distance, incidence angle of the vapour flux, etc.) the tilt angle of the developed columnar microstructure and the

film porosity is strongly dependent on the stoichiometry of the films. The results are discussed in the framework of several theoretical models on this topic.

"In situ" XPS studies of laser-induced surface nitridation and oxidation of tantalum

Lahoz, R; Espinos, JP; Yubero, F; Gonzalez-Elipe, AR; de la Fuente, GF
Journal of Materials Research, **30** (2015) 2967-2976
October, 2015 | **DOI:** 10.1557/jmr.2015.190

This work studies the nitridation of Ta by laser irradiation by means of x-ray photoelectron spectroscopy. The study has been carried out under "in situ" conditions by controlling the nitrogen partial pressure, the presence of traces of oxygen, and the irradiance of the laser. It is found that a thin layer of Ta_2O_5 is directly obtained when irradiating in the presence of oxygen, while a Ta_3N_5 surface compound and some minor contributions of nonstoichiometric phases are formed in the presence of nitrogen. For O-2:N-2 mixtures at 0.1 Pa, preferential nitride formation occurs up to a ratio of 1:4, while Ta_2O_5 starts to be predominant for ratios above this value. The air stability of the tantalum nitride layer formed by laser irradiation and the surface topography of the irradiated metal are also studied. The possible factors determining this behavior are discussed.

Effect of magnesium and titanium on the cathodic behaviour of aluminium in nitric acid

Garcia-Garcia, FJ, Chiu, TY, Skeldon, P, Thompson, GE
Surface and Interface Analysis, **47** (2015) 30-36
January, 2015 | **DOI:** 10.1002/sia.5640

Cathodic polarization of aluminium and Al-0.18wt.%Mg and Al-0.08wt.% Ti alloys in 0.24moldm(-3) nitric acid solution at 38 degrees C has been employed to assist understanding of the roles of alloying elements in electrograining. The findings indicate that additions of magnesium and titanium to aluminium accelerate the corrosion of the substrate under the alkalization caused by the cathodic reactions. The accelerated dissolution and the consequent formation of hydrated alumina result in a decreased net cathodic current density in potentiostatic and potentiodynamic polarization conditions relative to the behaviour of aluminium.

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

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

16th European Conference on Applications of Surface and Interface Analysis | ECASIA' 15

28 Septiembre – 1 octubre [Granada, España]

Francisco Yubero Valencia [Miembro del Comité Científico)

COMUNICACIONES / COMMUNICATIONS

MAT2VL days on Materials Optics and Electronic

18 - 19 febrero [Orleans, Francia]

New Single-Phase, White-Light-Emitting Proshor Based on δ-$Gd_2Si_2O_7$ for Solid-State Lighting
Alberto J. Fernández Carrión, Ana Isabel Becerro Nieto, Manuel Ocaña Jurado
Comunicación oral

Hybrid and Organic Photovoltaics Conference | HOPV 2015

10 - 13 mayo [Roma, Italia]

Colorful Perovskite Solar Cells Using Photonic Scaffolds
Hernán Míguez
Conferencia invitada

Nanolevitation phenomena in real plane-parallel sys-tems mediated by gravity and Casimir forces at thermal equilibrium
Sol Carretero Palacios; Victoria Esteso Carrizo; Hernán Míguez
Conferencia invitada

Next Generation Solar

24 mayo [Canadá]

Relevance of Optical Design in Third Generation Solar Cells
Hernán Míguez
Conferencia invitada

Progress In Electromagnetics Research Symposium | PIERS 2015

6 - 9 julio [Praga, República Checa]

Random Optical Media for Third Generation Photovoltaics
Hernán Míguez
Conferencia invitada

Nanolevitation Phenomena in Real Plane-parallel Systems Mediated by Gravity and Casimir Forces at Thermal Equilibrium
Sol Carretero-Palacios; Victoria Esteso; Hernán Míguez
Comunicación oral

Temperature Dependence of the Equilibrium Distance in Plane-parallel Thin Films at Thermal Equilibrium
Victoria Esteso; Sol Carretero-Palacios; Hernán Míguez
Póster

Progress In Electromagnetics Research Symposium | PIERS 2015

6 - 9 julio [Praga, República Checa]

Random Optical Media for Third Generation Photovoltaics
Hernán Míguez
Conferencia invitada

10th International Conference on Composite Science and Technology | ICCST/10

2 - 4 septiembre [Lisboa, Portugal]

Development of high emitting Rosamine-TiO_2/SiO_2 composite thin films
Belén Suárez, María G. Guillén, Tânia Lopes-Costa, José Mª Pedrosa, Juan R. Sánchez-Valencia, Ángel Barranco, Agustín R. González-Elipe, Ana M. G. Silva
Poster

Tetracarboxyphenylporphyrin/TiO_2 composite thin films as selective optical sensors for the detection of volatile organic compounds (VOCs)
María G. Guillén, Javier Roales, Belén Suárez, Tânia Lopes-Costa, José M. Pedrosa, Pedro Castillero, Ángel Barranco, Agustín R. González-Elipe
Poster

16th European Conference on Applications of Surface and Interface Analysis | ECASIA’ 15

28 Septiembre – 1 octubre [Granada, España]

Structure and composition of undoped and Au-modified Ni/YSZ thin films for solid oxide fuel cells

Francisco J. García-García, Francisco Yubero Valencia, Juan Pedro Espinós Manzorro, D. Gamarra, Agustín R. González-Elipe, Richard M. Lambert
Comunicación oral

Quantitative description of Ni2p photoemission from NiO and Ni:SiO_2
N. Pauly, Francisco Yubero Valencia, Francisco J. García-García, S. Tougaard
Comunicación oral

Tunable cathode-luminescent electron monitor devices based on multilayer thin film technology
Jorge Gil-Rostra, Agustín R. González-Elipe, Francisco Yubero Valencia
Comunicación oral

Depth profile study of the in-vitro degradation for PLGA surfaces under biomedical treatments
Carmen López Santos, Antonia Terriza, J. Portoles, Francisco Yubero Valencia, P. Cumpson, Agustín R. González-Elipe
Comunicación oral

"In operando" XAS study during electrochemical cycling of $W_xSi_yO_z$ amorphous electrochromic thin film cathodes
Francisco J. García-García, Jorge Gil-Rostra, Francisco Yubero Valencia, Juan Pedro Espinós Manzorro, Agustín R. González-Elipe, J. Chaboy
Poster

He-eRBS MC code for elemental multilayer profile analysis
Rafael Alvarez Molina, Francisco Yubero Valencia
Poster

CONGRESOS Y REUNIONES NACIONALES / NATIONAL CONGRESS AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

Aportando Valor al CO_2

17-18 febrero [Madrid, España]

Tecnologías de Plasma Catálisis para la conversión del CO_2
Agustín R. González-Elipe
Conferencia invitada

ImagineNano 2015

10 marzo [Bilbao, España]

Fine tuning of the emission properties of nano-emitters in multilayered structures by deterministic control of their local photonic environment
Alberto Jiménez Solano, Juan F. Galisteo López, Míguez Hernán
Comunicación oral

7th European Meeting on Chemical Industry and Environment | EMChIE 2015

10 – 12 junio [Madrid, España]

Microwave-based plasma reactors containing water for the oxidative treatment and modificacion of carbonaceous wastes from refinery processes
Victor J. Rico, José Luis Hueso, José Cotrino Bautista, Juan Pedro Espinós Manzorro, J.M. Jiménez-Mateos, Agustín R. González-Elipe
Comunicación oral

9th International workshop on Microwave Discharges: Fundamentals and Applications

7-11 septiembre [Córdoba, España]

Functionalization of polymeric materials by surface wave plasmas with biomedical applications
Carmen López Santos, Antonia Terriza, Francisco Yubero Valencia, José Cotrino Bautista, Agustín R. González-Elipe
Conferencia invitada

Workshop POLIGHT

5 – 7 octubre [Sevilla, España]

Flexible photonic structures from porous multilayers films
Mauricio Calvo
Comunicación oral

Photophysics of hypbrid organic-inorganic perovskite films: environmental effects
Juan Galisteo-López
Comunicación oral

Optical modelling of optoelectronic devices
Gabriel Lozano
Comunicación oral

Absorption Enhancement in Perovskite Films with Embedded Gold Nanoparticles
Sol Carretero-Palacios

Comunicación oral

Enhancement of Light Absorption in Photonic Crystals Integrated Flexible Dye Solar Cells
Yuelong Li
Comunicación oral

Fine tuning of the emission properties of nano-emitters in ultilayered strucutres by deterministic control of their local photonic enviroment
Alberto Jimenez-Solano
Comunicación oral

Nanostructured scaffolds for perovskite solar cells
Miguel Anaya
Comunicación oral

Levitation of self-standing thin films due to the Casimir force
Victoria Esteso; Sol Carretero-Palacios; Hernán Míguez
Comunicación oral

Efficient bifacial dye-sensitized solar cells through disorder by design
Jose M. Miranda
Comunicación oral

FORMACION / TRAINING

TESIS DOCTORALES/ DOCTOR DEGREE THESIS

Título: **Nanoestructuras Ópticas Híbridas Flexibles para Aplicaciones como Filtros de Radiación Ultravioleta**
Autor: José Raúl Castro Smirnov
Directores: Hernán Míguez García, Mauricio Calvo Roggiani
Calificación: Apto "Cum Laude" por Unanimidad
Centro: Universidad de Sevilla
Fecha: 13 de febrero de 2015

Título: **Development of Photonic Gas Sensors Base on Porphyrin/Metal Oxide Nanostructured Thin Films Obtained by Glancing Ange Deposition**
Autor: Pedro Castillero Durán
Directores: Ángel Barranco Quero, José María Pedrosa Poyato
Calificación: Sobresaliente "Cum Laude"
Centro: Universidad Pablo de Olavide
Fecha: 16 de febrero de 2015

Título: **Remote Plasma Assited Fabrication of Functional Organic and Hybrid Thin Films and Supported Nanostructures**
Autor: María Alcaire Martín
Directores: Ana Isabel Borrás Martos, Ángel Barranco Quero
Calificación: Sobresaliente "Cum Laude" por Unanimidad
Centro: Universidad de Sevilla
Fecha: 26 de febrero de 2015

Título: **Preparación de Nanoestructuras Orgánicas e Híbridas Orgánico-Inorgánicas mediante Condensación desde Fase Vapor**
Autor: Youssef Oulad Zian
Directores: Ana Isabel Borrás Martos, Juan Pedro Espinós Manzorro
Calificación: Sobresaliente "Cum Laude" por Unanimidad
Centro: Universidad de Sevilla
Fecha: 25 de junio de 2015

Título: **In-Plane Anisotropic Nanocolumnar Films for Advanced Functional Applications**
Autor: Julián Parra Barranco
Directores: Agustín R. González-Elipe, Ángel Barranco Quero
Calificación: Sobresaliente "Cum Laude" por Unanimidad
Centro: Universidad de Sevilla
Fecha: 10 de julio de 2015

Título: **Development of Supported 1D Nanomaterials by Vacuum and Plasma Technologies: From Sensors to Nanogenerators**
Autor: Alejandro Nicolás Filippin
Directores: Ana Isabel Borrás Martos, Ángel Barranco Quero, Juan Ramón Sánchez Valencia
Calificación: Sobresaliente "Cum Laude" por Unanimidad
Centro: Universidad de Sevilla
Fecha: 9 de noviembre de 2015

Formación de Graduados / Master Degree Thesis

Título: **Sensores de humedad basados en estructuras fotónicas**
Autor: Olga Blanco Jaime
Directores: Dra. Carmen López-Santos, Manuel Oliva Ramírez
Tutor: Dr. Agustin R. González-Elipe

Grado: Trabajo Fin de Grado
Año Académico: 2014-2015 (septiembre 2015)

Título: **Capas delgadas para el desarrollo de sensores eléctricos de deformación mecánica**
Autor: Francisco Javier Alvarez Ceballos
Directores: Dra. Carmen López-Santos
Tutor: Dr. Agustin R. González-Elipe
Grado: Trabajo Fin de Grado
Año Académico: 2014-2015 (julio 2015). PREMIO SOCIEMAT – CAJA INGENIEROS 2015 al "Mejor Proyecto Fin de Carrera Ingeniería de Materiales"

Título: **Nanopartículas fluorescentes para aplicaciones biomédicas y biotecnológicas**
Autor: Borja Nemesio Rivas Camacho
Tutoras: Dra. Ana Isabel Becerro Nieto, Dra. Carmen Gallardo Cruz
Grado: Trabajo Fin de Grado
Año Académico: 2014-2015 (diciembre 2015)

DOCENCIA / TEACHING

Máster "Láser, Plasma y Tecnología de Superficies"

Tecnología de lámina delgada

Alberto Palmero Acebedo, Agustín R. González-Elipe, Ana Isabel Borrás Martos, Angel Barranco Quero, Francisco Yubero Valencia, José Cotrino Bautista, Ana Isabel Becerro Nieto

Lugar: Universidad de Córdoba

5th Intensive School on Conservation Science: Ceramic Materials

Layers' microsctructure: influence and utility control for specific applications

Agustín R. González-Elipe

Lugar: Toledo, España

PREMIOS Y RECONOCIMIENTOS / PRIZES AND ACKNOWLEDGEMENTS

Beca "la Caixa" 2015 para realizar doctorado en universidades españolas, obtenida por la estudiante Victoria Esteso Carrizo para comenzar sus estudios de doctorado en el programa de Ciencia y Tecnología de los Nuevos Materiales. Su trabajo se centrará en el estudio teórico y experimental de la fuerza de Casimir entre materiales reales analizando cómo las propiedades ópticas de los materiales involucrados influyen en la intensidad y dirección de esta fuerza.

Francisco Yubero Valencia. Representante español en la Thin Film Division de la organización IUVSTA (trienio 2013-2016).

Miguel Anaya, primer accésit, Premio CicCartuja Ebro Foods 2015 Celdas solares de colores. El segundo trabajo galardonado está firmado por el investigador madrileño Miguel Anaya, perteneciente al Grupo de Materiales Ópticos Multifuncionales del ICMS, que lidera el profesor Hernán Míguez. Su artículo, aparecido en la revista Nano Letters, ha merecido el primer accésit del Premio "cicCartuja – Ebro Foods" por profundizar en las posibilidades de la perovskita como compuesto aplicado en celdas solares. A sus 27 años, Miguel Anaya, en colaboración con investigadores del ICMS y la Universidad de Oxford, ha diseñado celdas solares de perovskita con colores brillantes, que van desde el azul hasta el rojo.
Con este artículo, que ha generado una patente y que ha cosechado una sobresaliente repercusión internacional en publicaciones como Science, Miguel Anaya ha ampliado aún más las alternativas que ofrecen las celdas solares de perovskita; un material que ha revolucionado desde 2012 el campo de la tecnología fotovoltaica, gracias a sus propiedades ópticas y eléctricas únicas, que permiten unos costes de producción muy bajos. Tanto es así, que los esfuerzos científicos de los últimos años han permitido alcanzar valores de eficiencia del 21% en este nuevo tipo de dispositivos, cercanos a los de tecnologías muy asentadas en el mercado como son las celdas solares de silicio.

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

Photovoltaic and Optoelectronic Devices Group dirigido por Juan Bisquert. Universidad Jaume I

Castellón, España	**Miguel Anaya Martín**	2 semanas

Photovoltaic and Optoelectronic Devices Group dirigido por Henry Snaith. Universidad de Oxford

Oxford, Reino Unido	**Miguel Anaya Martín**	1 mes

EQUIPAMIENTO CIENTÍFICO AVAILABLE EQUIPMENT

- Sistema de Magnetron Sputttering para la preparación de láminas delgadas
- Potenciostato y sistema electroquímico
- Sistema de medida de ángulos de contacto (advancing and receding)
- Equipo de crecimiento en vacío de nanofibras y otros materiales nanoestructurados
- Microscopio de efecto túnel (STM)

- Sistema de desorción térmica programada
- Espectrómetro de Fotoemisión (XPS)
- Analizador de potencial Z, tamaño de partícula y pesos moleculares (Malvern, ZS90)
- Bomba híbrida de vacío resistente a químicos VACUUBRAND RC-6
- Software y sondas para monitorización para equipo de liofilización CHRIST EPSILON 2-4 LSC
- Liofilizador de altas prestaciones Epsilon 2-4 (CHRIST)
- Estufa de desecación de 90 litros (RAYPA)
- Cámara Incubadora Opaq + Orbital Maxi (OVAN)
- pH & Ion-metro GLP 22+ (CRISON A)
- Malvern, Modelo Zetasizer Nano: para determinar mediante "Dynamic light scattering" la distribución de tamaños de partícula de sistemas coloidales en suspensión (disolvente acuoso u orgánico) en los rangos 1-3000 nanómetros.
  Este equipo también es adecuado para la evaluación del potencial "Z" de sistemas coloidales en suspensión (disolvente acuoso u orgánico) mediante medidas de Movilidad Electroforética.
- Colorímetro Dr. Lange: para la medida de parámetros de color (x, y, Y/L*a*b*, etc.) de superficies y polvos
- Ellipsómetro espectroscópico Woolan VB-400 con rango de frecuencias entre 300 y 1700 nm. Medida de índices de refracción y coeficiente de extención de capas delgadas y superficies.
- Espectrómetro visible-UV CARY-100. Medidas de coeficiente de absorción con luz normal y polarizada.
- Fluorímetro espectroscópico (HORYBA Jobin Yvon Fluorolog) con accesorio para la determinación de tiempos de vida. Microscopio de fluorescencia (HORYBA Jobin Yvon sigle photon controller: FluoroHub).
- Medidor de ángulos de contacto líquidos. Medidas ángulos de avance y retroceso, así como de energías de adhesión de líquidos sobre superficies (Dataphysics Contact Angle System SCA 20).
- Medida de cuatro puntos de conductividad eléctrica en superficies y láminas delgadas (Fuente de corriente Keithley 617 y voltímetro Keithley 2400).
- Medidas eléctricas en capas delgadas en función de la temperatura y la atmósfera
- Microscopio de Fuerzas atómicas (AFM) para la caracterización de superficies (Cervantes de Nanotec).
- Microscopio de efecto túnel (STM) con posibilidad de trabajar desde nitrógeno líquido hasta 600 ºC (VT-STM de Omicrom).
- Técnicas de caracterización de plasmas: sonda de Langmuir (Plasma Consult single and double sound), espectroscopía de emisión óptica (Avantes 200-900 nm resolución 1 nm) y espectrometría de masas (Hyden=
- Espectrómetro FT-IR con celda DRIFT (Pelkin elmer Spectrum One)
- Sistema de medida de porosidades en capas delgadas.
- Sistema de desorción térmica programada dotado con espectroscopía Auger (VG-8047).
- Espectrómetro de XPS (espectrómetro VSW) con sistema REELS de alta resolución (Kimball Physics EGPS-1022B) y fuente de átomos incorporada (Oxford Scientific Osprey plasma Source).

Diseño de Nanomateriales y Microestructuras

Tailored Nanomaterials and Microstructure

PERSONAL / PERSONNEL

Profesores de Investigación

Dra. Asunción Fernández Camacho

Catedráticos

Dr. Luis M. Esquivias Fedriani
Dr. Diego Gómez García

Investigadores Científicos

Dr. Juan Carlos Sánchez López

Científicos Titulares

Dra. Rosalía Poyato Galán
Dra. T. Cristina Rojas Ruiz

Investigadores Contratados

Dra. Gisela M. Arzac Di Tomaso
Dra. Ana María Beltrán Custodio
Dra. Vanda C. Fortio Godinho

Becarios Predoctorales

Lda. Mariana Paladini San Martin

Personal Contratado

Ing. Tec. M. Rocío García Gil
Ldo. Dirk Hufschmidt
D. Miguel Nieto Redondo

Proyectos de Investigación / Research Projects

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

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

RESUMEN / ABSTRACT

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

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

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

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

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

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

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

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

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

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

Código/Code:	CTQ2012-32519
Periodo/Period:	01-01-2013 / 31-12-2015
Organismo Financiador/Financial source:	Ministerio de Ciencia e Innovación
Importe total/Total amount:	190.710 €
Investigador responsable/Research head:	Fernández Camacho, Asunción
Componentes/Research group:	Gisela Arzac, Jaime Caballero, Lionel Cervera, Vanda Fortio, Carlos Negrete, Dirk Hufschmidt, Cristina Rojas Ruiz, Roland Schierholz

RESUMEN / ABSTRACT

El hidrógeno como vector de transporte y almacenamiento de energía es un candidato muy atractivo en el contexto de un mayor uso de las energías renovables y limpias. En el presente proyecto se abordará el estudio de los distintos procesos que conducen a la configuración final integrada de sistemas de generación y uso del hidrógeno principalmente en aplicaciones portátiles (y potencialmente escalables para aplicaciones estacionarias). En particular se trabajará en este proyecto en las siguientes líneas de actuación:

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

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

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

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

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

f) Caracterización microestructural y química de los nuevos materiales y catalizadores desarrollados en el proyecto. Se trata típicamente de materiales con una nanoestructura controlada en donde las modernas técnicas nanoscopicas van a jugar un papel fundamental en la fabricación a medida de estos materiales.

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

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

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

c) Studies of coupling a hydrogen generator system with a low cost fuel cell. Typically a continuous reactor for the hydrolysis of $NaBH_4$ with Co-B catalyst for providing H_2 at constant flow rate conditions to directly feed a PEM fuel cell of 60 W.

d) Fundamental studies for the development of catalysts and supports for the controlled combustion of hydrogen. It's a new line in the research group based on wet chemical preparation of noble metal nanoparticle catalysts on commercial porous ceramic supports (i.e. SiC). The line also includes the design of a reactor for laboratory-scale study of heat production by controlled combustion of hydrogen.

e) Development of sputtering technology ("magnetron sputtering") for the preparation of catalysts and nano-structures on various substrates for use in the processes developed in the previous sections. The group has extensive experience in this technology to be applied in novel ways in this project leading to a great versatility regarding nanostructure, composition and addition of additives to improve catalytic activity, durability and selectivity of catalysts.

f) Microstructural and chemical characterization of new materials and catalysts developed in the project. We are dealing typically with materials of controlled nanostructure where modern nanoscopic techniques will play a key role in the custom manufacturing of these materials.

Unión Europea
Fondo Europeo de Desarrollo Regional
"Una manera de hacer Europa"

JUNTA DE ANDALUCIA

Desarrollo de procesos de combustión catalítica de hidrógeno y estudio de su integración en dispositivos para aplicaciones portátiles Development of processes for the catalytic combustion of hydrogen and study of the integration in devices for portable applications

Código/Code:	P12-TEP-862 (Proyecto de Excelencia)
Periodo/Period:	16-05-2014 / 15-05-2016
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	153.025 €
Investigador responsable/Research head:	Asunción Fernández Camacho

Componentes/Research group: Julián Martínez, Gisela Arzac, Dirk Hufschmidt, Joaquín Ramírez, M.Carmen Vera, Vanda Godinho, Lionel Cervera, T.Cristina Rojas, Olga Montes, Mariana Paladini, Jaime Caballero-Hernández

RESUMEN / ABSTRACT

El hidrógeno como vector de transporte y almacenamiento de energía es un candidato muy atractivo en el contexto de un mayor uso de las energías renovables y limpias. La producción y el uso de la energía basada en la tecnología del hidrógeno es de especial relevancia en pequeña escala para aplicaciones portátiles (y potencialmente escalable para aplicaciones estacionarias). En el presente proyecto se abordará el estudio del proceso de combustión catalítica o controlada de hidrógeno en los distintos aspectos que puedan conducir a una configuración final integrada con un sistema de generación de H2 en aplicaciones portátiles. Para ello se aprovecharán las sinergias integrando investigadores de dos grupos del PAI: i) Del grupo TEP217, especialistas en almacenamiento y generación de hidrógeno en sistemas basados en hidruros metálicos, hidruros complejos y composites de hidruros reactivos; así como en el uso de catalizadores y aditivos para controlar y mejorar las cinéticas de estos procesos. ii) Del grupo FQM342, especialistas en la obtención de cerámicos porosos de alto interés como soportes de catalizadores en entornos agresivos de combustión. Además la colaboración se completa con la participación de la empresa Abengoa Hidrógeno S.A. que participa en calidad de subcontratada como especialistas en sistemas de producción y almacenamiento de hidrógeno.

En particular se trabajará en este proyecto en las siguientes líneas de actuación:

1.- Desarrollo de catalizadores y soportes para la combustión controlada. Típicamente cerámicas porosas biomórficas de carburo de silicio y catalizadores clásicos tipo metal noble y nuevos catalizadores de bajo coste a desarrollar en el proyecto.

2.- Desarrollo de los reactores necesarios para el estudio de la combustión controlada. Típicamente para flujos de hidrógeno de unos pocos ml/min y para la escala de un generador de H2 ya disponible de 0.5 a 1.5 L/min.

3.- Acoplamiento al sistema de combustión controlada de los sistemas portátiles de generación de hidrógeno que hemos desarrollado en proyectos anteriores.

4.- Aplicación de la tecnología de pulverización catódica de una manera exploratoria en este proyecto para depositar los catalizadores de combustión catalítica en sustratos porosos.

5.- Caracterización microestructural y química de los soportes y catalizadores en la nanoescala para seguir los procedimientos de síntesis y evolución en operación.

Hydrogen is an attractive candidate as a vector for storage and transport of energy in the context of an increased use of renewable and clean energies. The production and use of energy based on hydrogen technology is particularly important for small-scale portable (and potentially scalable for stationary) applications. In this project the process of catalytic (controlled) combustion of hydrogen will be investigated in the various aspects that could lead to a final integrated configuration with a H2 generation system for portable applications. For that the project will take advantage of the synergy of integrating two researcher groups from the PAI: i) The TEP217 group, specialists in storage and generation of hydrogen based on metal hydrides, complex hydrides and hydride composites reactive systems; and in the use of catalysts and additives to control and improve the kinetics of these processes. ii) The FQM342, specialist group

for the fabrication of porous ceramics of high interest as catalyst supports for harsh combustion environments. Further collaboration is completed with the participation of the company Abengoa Hidrógeno SA that will be involved as sub-contractor as specialist in systems for the production and storage of hydrogen.

In particular we will work on this project in the following lines:

1.- Development of catalysts and supports for catalytic combustion. Typically porous biomorphic silicon carbide ceramics and classic noble metal catalysts, as well as new low cost catalysts to be developed in the project.

2.- Development of reactors needed for the study of the catalytic combustion. Typically hydrogen flows from a few ml/min to the scale of a H2 generator already available in the range 0.5 to 1.5 L/min.

3.- Coupling the catalytic combustion system with a portable hydrogen generation systems that we have developed in previous projects.

4.- Application of the sputtering technology in an exploratory manner in this project to deposit the catalyst materials for the H2 catalytic combustion on porous substrates.

5.- Microstructural and chemical characterization of the supports and catalysts in the nanoscale to follow the procedures of synthesis and evolution in operation.

Unión Europea
Fondo Europeo de Desarrollo Regional
"Una manera de hacer Europa"
JUNTA DE ANDALUCIA

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

Tribological study of functional coatings working under extreme conditions

Código/Code: 201560E013
Periodo/Period: 01-01-2015 / 31-12-2016
Organismo Financiador/Financial source: CSIC (Intramural)
Importe total/Total amount: 17.780 €
Investigador responsable/Research head: Juan Carlos Sánchez López
Componentes/Research group: T.Cristina Rojas, Santiago Domínguez Meister

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 para avisadores sonoros

Periodo/Period: 18-01-2010 / 31-12-2015
Organismo Financiador/Financial source: Clarton Horn
Importe total/Total amount: 14.916,75 €
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 por microscopía electrónica

Periodo/Period: 14-02-2014 / 15-02-2016
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

COOPERACIÓN INTERNACIONAL Y OTROS INTERNATIONAL COOPERATION AND OTHERS

Nano-Phenomena and Functionality of Modern Carbon-Based Tribo-Coatings

Periodo/Period:	1-06-2015 / 30-06-2018
Código/Code:	CARBITRIB
Entidad Financiadora/Financial source:	Leverhulme Trust (UK)
Investigador responsable/Research head:	Feodor Borodich
Participantes/Participants:	Juan Carlos Sánchez López

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

Self-lubricity of WSe_x nanocomposite coatings

S. Dominguez-Meister; M. Conte; A. Igartua; T.C. Rojas; J.C. Sánchez-López
ACS Applied Materials & Interfaces, **7** (2015) 7979-7986
April, 2015 | **DOI:** 10.1021/am508939s

Transition metal chalcogenides with lamellar structure are known for their use in tribological applications although limited to vacuum due to their easy degradation in the presence of oxygen and/or moisture. Here we present a tailored WSe_x coating with low friction (0.07) and low wear rates (3×10^{-7} mm^3 Nm^{-1}) even in ambient air. To understand the low friction behavior and lower chemical reactivity a tribological study is carried out in a high-vacuum tribometer under variable pressure (atmospheric pressure to 1×10^{-8} mbar). A detailed investigation of the film nanostructure and composition by advanced transmission electron microscopy techniques with nanoscale resolution determined that the topmost layer is formed by nanocrystals of WSe_2 embedded in an amorphous matrix richer in W, a-W(Se). After the friction test, an increased crystalline order and orientation of WSe_2 lamellas along the sliding direction were observed in the interfacial region. On the basis of high angle annular dark field, scanning transmission electron microscopy, and energy dispersive X-ray analysis, the release of W atoms from the interstitial basal planes of the a-W(Se) phase is proposed. These W atoms reaching the surface, play a sacrificial role preventing the lubricant WSe_2 phase from oxidation. The increase of the WSe_2 crystalline order and the buffer effect of W capturing oxygen atoms would explain the enhanced chemical and tribological response of this designed nanocomposite material.

Fabrication of Optical Multi layer Devices from Porous Silicon Coatings with Closed Porosity by Magnetron Sputtering

Caballero-Hernandez, Jaime; Godinho, Vanda; Lacroix, Bertrand; Jimenez de Haro, Maria C.; Jamon, Damien; Fernandez, Asuncion
ACS Applied Materials & Intefaces, **7** (2015) 13880-13897
July, 2015 | **DOI:** 10.1021/acsami.5b02356

The fabrication of single-material photonic-multilayer devices is explored using a new methodology to produce porous silicon layers by magnetron sputtering. Our bottom-up methodology produces highly stable amorphous porous silicon films with a controlled refractive index using magnetron sputtering and incorporating a large amount of deposition gas inside the closed pores. The influence of the substrate bias on the formation of the closed porosity was explored here for the first time when He was used as the deposition gas. We successfully simulated, designed, and characterized Bragg reflectors and an optical microcavity that integrates these porous layers. The sharp interfaces between the dense and porous layers combined with the adequate control of the refractive index and thickness allowed for excellent agreement between the simulation and the experiments. The versatility of the magnetron sputtering technique allowed for the preparation of these structures for a wide range of substrates such as polymers while also taking advantage of the oblique angle deposition to prepare Bragg reflectors with a controlled lateral gradient in the stop band wavelengths.

Hydration and carbolenation reactions of calcium oxide by weathering: Kinetics and changes in the nanostructure

Morales-Florez, V; Santos, A; Romero-Hermida, I; Esquivias, L
Chemical Engineering Journal, **265** (2015) 194-200
April, 2015 | **DOI:** 10.1016/j.cej.2014.12.062

The weathering reactions of hydration and carbonation of nanostructured calcium oxide with atmospheric moisture and carbon dioxide have been characterized. This work is the first-to-date combined kinetic and nanostructural research on CaO oriented to two key processes for different systems, i.e. hardening of construction materials and carbon mineral sequestration. The evolution of the precipitated crystalline phases was monitored by X-ray diffraction and thermogravimetry, along with structural characterization by nitrogen physisorption, electron microscopy and small-angle scattering. Complete hydration of the samples was always found prior to the onset of carbon sequestration, which depended on the nanostructure of the samples. Hence, carbonation started after 300 h of weathering for samples with a specific surface area of 40 m(2)/g, whereas carbonation of the samples with 20 m(2)/g occurred after 550 h. Full carbonation from atmospheric CO2 (100% efficiency) was obtained in all cases. This combined research was completed by developing an empirical description of the weathering reactions in terms of a two-process Random Pore Model. Finally, this work aimed to determine the role of the nanostructure of samples based on industrial wastes as one of the most important factors for developing efficient carbon sequestration technologies.

Ultra-fast and energy-efficient sintering of ceramics by electric current concentration

Zapata-Solvas, E; Gomez-Garcia, D; Dominguez-Rodriguez, A; Todd, RI

Scientific Reports, **5** (2015) art. 8513

February, 2015 | **DOI:** 10.1038/srep08513

Electric current activated/assisted sintering (ECAS) techniques, such as electrical discharge sintering (EDS) or resistive sintering (RS), have been intensively investigated for longer than 50 years. In this work, a novel system including an electrically insulated graphite die for Spark Plasma Sintering (SPS) is described, which allows the sintering of any refractory ceramic material in less than 1 minute starting from room temperature with heating rates higher than 2000°C/min and an energy consumption up to 100 times lower than with SPS. The system alternates or combines direct resistive sintering (DRS) and indirect resistive sintering (IRS). Electrical insulation of the die has been achieved through the insertion of a film made of alumina fibers between the graphite die and the graphite punches, which are protected from the alumina fiber film by a graphite foil. This system localized the electric current directly through the sample (conductive materials) as in DRS and EDS, or through the thin graphite foil (non-conductive materials) as in IRS, and is the first system capable of being used under EDS or RS conditions independently combining current concentration/localization phenomena.

STEM-in-SEM high resolution imaging of gold nanoparticles and bivalve tissues in bioaccumulation experiments

C.A. García-Negrete; M.C. Jiménez de Haro; J. Blasco; M. Soto; A. Fernández

Analyst, **140** (2015) 3082-3089

May, 2015 | **DOI:** 10.1039/C4AN01643B

The methodology termed scanning transmission electron microscopy in scanning electron microscopy (STEM-in-SEM) has been used in this work to study the uptake of citrate stabilized gold nanoparticles (AuNPs) (average particle sizes of 23.5 ± 4.0 nm) into tissue samples uponin vitro exposure of the dissected gills of the Ruditapes philippinarum marine bivalve to the nanoparticle suspensions. The STEM-in-SEM methodology has been optimized for achieving optimum resolution under SEM low voltage operating conditions (20–30 kV). Based on scanning microscope assessments and resolution testing (SMART), resolutions well below 10 nm were appropriately achieved by working at magnifications over 100k×, with experimental sample thickness between 300 and 200 nm. These relatively thick slices appear to be stable under the beam and help avoid NP displacement during cutting. We herein show that both localizing of the internalized nanoparticles and imaging of ultrastructural disturbances in gill tissues are strongly accessible due to the improved resolution, even at sample thicknesses higher than those normally employed in standard TEM techniques at higher voltages. Ultrastructural imaging of bio-nano features in bioaccumulation experiments have been demonstrated in this study.

High-temperature deformation of fully-dense fine-grained boron carbide ceramics: Experimental facts and modeling

Moshtaghioun, BM; Garcia, DG; Rodriguez, AD

Materials & Design, **88** (2015) 287-293
December, 2015 | **DOI:** 10.1016/j.matdes.2015.08.134

Boron carbide ceramics are the hardest material in Nature after diamond and the cubic phase of boron nitride. Due to this fact, their room-temperature fracture properties are the object of intense research. Paradoxically, high-temperature deformation is essentially unknown, because very high temperatures and stresses are necessarily required and high-quality specimens have not been available until recently. In this paper, the high-temperature compressive creep of fine-grained boron carbide polycrystals is reported. The breakdown of the classical power-law for high-temperature plasticity in ceramics is found. An analytical model is proposed. The model assumes that deformation is produced by dislocation glide. However, since the formation of twins is energetically favorable in this material and they act as strong barriers for dislocation glide, their motion turns to become progressively more difficult as elongation proceeds. The combination of increasing twin barriers and dislocations in mutual interaction is proposed to be the mechanism for high-temperature plasticity in this material. The model is validated with the experimental results. Final elongation of boron carbide specimens is reported to be over 100%, although this material cannot be described as a superplastic ceramic.

Fractionation and fluxes of metals and radionuclides during the recycling process of phosphogypsum wastes applied to mineral CO_2 sequestation

Contreras, M., Pérez-López, R., Gazquez, M.J., Moralez-Florez, V., Santos, A., Esquivias, L., Bolivar, J.P.
Waste Management, **45** (2015) 41-419
November, 2015 | **DOI:** 10.1016/j.wasman.2015.06.046

The industry of phosphoric acid produces a calcium-rich by-product known as phosphogypsum, which is usually stored in large stacks of millions of toms. Up to now, no commercial application has been widely implemented for its reuse because of the significant presence of potentially toxic contaminants. This work confirmed that up to 96% of the calcium of phosphogypsum could be recycled for CO_2 mineral sequestration by a simple two-steps process: alkaline dissolution and aqueous carbonation, under ambient pressure and temperature. This CO_2 sequestration process based on recycling phosphogypsum wastes would help to mitigate greenhouse gasses emissions. Yet this work goes beyond the validation of the sequestration procedure; it tracks the contaminants, such as trace metals or radionuclides, during the recycling process in the phosphogypsum. Thus, most of the contaminants were transferred form raw phosphogypsum to portlandite, obtained by dissoulution of the phosphogypsum in soda, and from portlandite to calcite during aqueous carbonation. These findings provide valuable information for managing phosphogypsum wastes and designing potential technological applications of the by-products of this environmentally-friendly proposal.

STEM-EELS analysis reveals stable highdensity He in nanopores of amorphous silicon coatings deposited by magnetron sputtering

Schierholz, Roland; Lacroix, Bertrand; Godinho, Vanda; Caballero-Hernandez, Jaime; Duchamp, Martial; Fernandez, Asuncion

Nanotechnology, **26** (2015) 075703
February, 2015 | **DOI:** 10.1088/0957-4484/26/7/075703

A broad interest has been showed recently on the study of nanostructuring of thin films and surfaces obtained by low-energy He plasma treatments and He incorporation via magnetron sputtering. In this paper spatially resolved electron energy-loss spectroscopy in a scanning transmission electron microscope is used to locate and characterize the He state in nanoporous amorphous silicon coatings deposited by magnetron sputtering. A dedicated MATLAB program was developed to quantify the helium density inside individual pores based on the energy position shift or peak intensity of the He K-edge. A good agreement was observed between the high density (~35–60 at nm−3) and pressure (0.3–1.0 GPa) values obtained in nanoscale analysis and the values derived from macroscopic measurements (the composition obtained by proton backscattering spectroscopy coupled to the macroscopic porosity estimated from ellipsometry). This work provides new insights into these novel porous coatings, providing evidence of high-density He located inside the pores and validating the methodology applied here to characterize the formation of pores filled with the helium process gas during deposition. A similar stabilization of condensed He bubbles has been previously demonstrated by high-energy He ion implantation in metals and is newly demonstrated here using a widely employed methodology, magnetron sputtering, for achieving coatings with a high density of homogeneously distributed pores and He storage capacities as high as 21 at%.

Island-type growth of Au–Pt heterodimers: direct visualization of misfit dislocations and strain-relief mechanisms

Garcia-Negrete, CA; Knappett, BR; Schmidt, FP; Rojas, TC; Wheatley, AEH; Hofer, F; Fernandez, A
RSC Advances, **5** (2015) 55262-55268
June, 2015 | **DOI:** 10.1039/C5RA09808D

Structural and analytical characterization related to the formation mechanism of Au–Pt heterodimers from polyhedral Pt nanocrystals is reported. The observation of specific lattice strain effects and the emergence of misfit dislocations point to the relevance of the Stranski–Krastanov growth mode as a means of explaining the previously reported dimerisation reaction between Au and Pt. Two size-dependent strain relief mechanisms were identified. For dimers grown from 4.7 nm seeds, the mechanism is related to bulk lattice strain accumulation at {111} planes along with lattice relaxation effects on other crystalline planes. However, for dimers grown from 11.2 nm seed sizes, the formation of misfit dislocations proved to be a highly efficient mechanism by which to release interface mismatch strain. Nanoscale chemical mapping at Au–Pt interfaces also revealed Au–Pt alloying to be unlikely under the mild temperature conditions employed in this work for Au–Pt heterodimer synthesis.

Hydrogen production through sodium borohydride ethanolysis

Arzac, GM; Fernandez, A
International Journal of Hydrogen Energy, **40** (2015) 5326-5332
April, 2015 | **DOI:** 10.1016/j.ijhydene.2015.01.115

In this work, sodium borohydride (SB) ethanolysis was explored for the first time as a method to generate hydrogen for Polymer Exchange Membrane Fuel Cells. Ethanolysis by-product was characterized by Fourier Transform Infrared Spectroscopy, X-Ray Diffraction, and Nuclear Magnetic Resonance. Metal and acid catalysts were tested. RuCl3 center dot 3H(2)O was the best metal catalyst. Acetic acid was selected for the study because of its effectiveness, low cost and relative greenness. The maximum gravimetric hydrogen density obtained was 2.1% wt. The addition of water produces an increase in hydrogen generation rate and a decrease in conversion. The use of ethanol-methanol mixtures produces an increase in reaction rates in absence of catalyst. As a proof of concept the reaction was performed in a small reactor which operates by the addition of ethanolic acetic acid solutions to solid SB (in the form of granules). The reactor produces stable and constant hydrogen generation in the range of 20-80 ml min(-1) during 1 h at constant temperature (around 27-35 degrees.

High N-content a-C:N films elaborated by femtosecond PLD with plasma assistance

Maddi, C; Donnet, C; Loir, AS; Tite, T; Barnier, V; Rojas, TC; Sanchez-Lopez, JC; Wolski, K; Garrelie, F
Applied Surface Science, **332** (2015) 346-353
March, 2015 | **DOI:** 10.1016/j.apsusc.2015.01.123

Amorphous carbon nitride (a-C:N) thin films are a interesting class of carbon-based electrode materials. Therefore, synthesis and characterization of these materials have found lot of interest in environmental analytical microsystems. Herein, we report the nitrogen-doped amorphous carbon thin film elaboration by femtosecond pulsed laser deposition (fs-PLD) both with and without a plasma assistance. The chemical composition and atomic bonding configuration of the films were investigated by multi-wavelength (MW) Raman spectroscopy, X-ray photoelectron spectroscopy (XPS) and electron energy-loss spectroscopy (EELS). The highest nitrogen content, 28 at.%, was obtained with plasma assistance. The I(D)/I(G) ratio and the G peak position increased as a function of nitrogen concentration, whereas the dispersion and full width at half maximum (FWHM) of G peak decreased. This indicates more ordered graphitic like structures in the films both in terms of topological and structural, depending on the nitrogen content. EELS investigations were correlated with MW Raman results. The interpretation of XPS spectra of carbon nitride films remains a challenge. Plasma assisted PLD in the femtosecond regime led to a significant high nitrogen concentration, which is highlighted on the basis of collisional processes in the carbon plasma plume interacting with the nitrogen plasma

Role of Y in the oxidation resistance of CrAlYN coatings

Dominguez-Meister, S; El Mrabet, S; Escobar-Galindo, R; Mariscal, A; de Haro, CJ; Justo, A; Brizuela, M; Rojas, TC; Sanchez-Lopez, JC
Applied Surface Science, **363** (2015) 504-511
October, 2015 | **DOI:** 10.1016/j.apsusc.2015.06.099

CrAlYN coatings with different aluminum (4–12 at.%) and yttrium (2–5 at.%) contents are deposited by d.c. reactive magnetron sputtering on silicon and M2 steel substrates using metallic targets and Ar/N2 mixtures. The influence of the nanostructure and chemical elemental distribution on the oxidation resistance after heating in air at 1000 °C is studied by means of cross-sectional scanning electron microscopy (X-SEM), energy dispersive X-ray analysis (EDX), X-ray diffraction (XRD) and glow discharge optical emission spectroscopy (GD-OES). The sequential exposure to the metallic targets during the synthesis leads to a multilayer structure where concentration of metallic elements (Cr, Al and Y) is changing periodically. A good oxidation resistance is observed when Al- and Y-rich regions are separated by well-defined CrN layers, maintaining crystalline coherence along the columnar structure. This protective behavior is independent of the type of substrate and corresponds to the formation of a thin mixed (Al, Cr)-oxide scale that protects the film underneath. The GD-OES and XRD analysis have demonstrated that Y acts as a reactive element, blocking the Fe and C atoms diffusion from the steel and favoring higher Al/Cr ratio in the passivation layer after heating. The coating with Y content around 4 at.% exhibited the best performance with a thinner oxide scale, a delay in the CrN decomposition and transformation to Cr2N, and a more effective Fe and C blocking.

Mechanical and electrical properties of low SWNT content 3YTZP composites

Poyato, R; Macias-Delgado, J; Garcia-Valenzuela, A; Gallardo-Lopez, A; Morales-Rodriguez, A; Munoz, A; Dominguez-Rodriguez, A

Journal of the European Ceramic Society, **35** (2015) 2351-2359
August, 2015 | **DOI:** 10.1016/j.jeurceramsoc.2015.02.022

Fully dense 3 mol% Y_2O_3-ZrO_2 (3YTZP) composites with low single wall carbon nanotube content (0.5, 1 and 1.5 vol% SWNT) were prepared by colloidal processing and spark plasma sintering (SPS). SWNT were distributed at ceramic grain boundaries and also into agglomerates. Characterization of SWNT agglomerates indicated that increase in SWNT vol% does not imply an increase in agglomeration. SWNT agglomerate density was related to the evolution of hardness and fracture toughness with SWNT vol%. Electrical properties of the composites were characterized in a wide temperature range, and percolation threshold was estimated. A model allowing separation of the individual SWNT bundles contribution to resistance from the resistance due to junctions between bundles was proposed for composites with a percolating SWNT network.

Biotribological behavior of Ag–ZrC_xN_{1-x} coatings against UHMWPE for joint prostheses devices

Calderon, SV; Sanchez-Lopez, JC; Cavaleiro, A; Carvalho, S

Journal of the Mechanical Behavior of Biomedical Materials, **41** (2015) 83-91
January, 2015 | **DOI:** 10.1016/j.jmbbm.2014.09.028

This study aims to evaluate the structural, mechanical and tribological properties of zirconium carbonitrides ($ZrC_xN_{1-x)}$ coatings with embedded silver nanoparticles, produced with the intention of achieving a material with enhanced multi-functional properties, including mechanical strength, corrosion resistance, tribological performance and antibacterial behavior suitable for their use in joint prostheses. The coatings were deposited by direct current (DC) reactive magnetron sputtering onto 316 L stainless steel, changing the silver content from 0 to 20 at% by modifying the current density applied to the targets. Different nitrogen and acetylene gas fluxes were used as reactive gases. The coatings revealed different mixtures of crystalline

ZrC_xN_{1-x}, silver nanoparticles and amorphous carbon phases. The hardness of the films was found to be mainly controlled by the ratio between the hard (ZrC_xN_{1-x}) and soft (Ag and amorphous carbon) phases in the films, fluctuating between 7.4 and 20.4 GPa. The coefficient of friction, measured against ultra-high molecular weight polyethylene (UHMWPE) in Hank's balanced salt solution with 10 g L−1albumin, is governed by the surface roughness and hardness. The UHMWPE wear rates were in the same order of magnitude (between 1.4 and 2.0×10−6 mm3 N−1 m−1), justified by the effect of the protective layer of albumin formed during the tests. The small differences were due to the hydrophobic/hydrophilic character of the surface, as well as to the silver content.

Removing the effects of the "dark matter" in tomography

Gontard, Lionel C.
Ultramicroscopy, **154** (2015) 64-72
July, 2015 | **DOI:** 10.1016/j.ultramic.2015.03.017

Electron tomography (ET) using different imaging modes has been progressively consolidating its position as a key tool in materials science. The fidelity of a tomographic reconstruction, or tomogram, is affected by several experimental factors. Most often, an unrealistic cloud of intensity that does not correspond to a real material phase of the specimen ("dark matter") blurs the tomograms and enhances artefacts arising from the missing wedge (MW). Here we show that by simple preprocessing of the background level of any tomographic tilt series, it is possible to minimise the negative effects of that "dark matter". Iterative reconstruction algorithms converge better, leading to tomograms with fewer streaking artefacts from the MW, more contrast, and increased accuracy. The conclusions are valid irrespective of the imaging mode used, and the methodology improves the segmentation and visualisation of tomograms of both crystalline and amorphous materials. We show examples of HAADF STEM and BF TEM tomography.

Influence of the Processing Route on the Carbon Nanotubes Dispersion and Creep Resistance of 3YTZP/SWCNTs Nanocomposites

Castillo-Rodriguez, M; Munoz, A; Morales-Rodriguez, A; Poyato, R; Gallardo-Lopez, A; Dominguez-Rodriguez, A
Journal of the American Ceramic Society, **98** (2015) 645-653
February, 2015 | **DOI:** 10.1111/jace.13348

3YTZP matrix composites containing 2.5 vol% of single-walled carbon nanotubes (SWCNT) were fabricated by Spark Plasma Sintering (SPS) at 1250°C, following different processing routines with the aim of optimizing the SWCNTs dispersion throughout the ceramic matrix. Microstructural characterization of the as-fabricated samples has been performed by means of scanning electron microscopy (SEM). The specimens have been crept at 1200°C to correlate creep resistance and SWCNTs distribution. There are no creep experimental results on these nanocomposites reported in literature. Mechanical results show that the incorporation of SWCNTs into a 3YTZP matrix produces an increase in the strain rate at high temperature with respect to monolithic zirconia. The creep resistance of these nanocomposites decreases with the improvement of the SWCNTs dispersion, where a smaller SWCNTs agglomerate size and consequently a higher concentration of carbon nanotubes surrounding the 3YTZP grain boundaries is found. This fact indicates that SWCNTs act as a lubricant making grain-boundary sliding easier during deformation of these composites.

Microstructure and impedance spectroscopy of 3YTZP/SWNT ceramic nanocomposites

Poyato, R; Macias-Delgado, J; Gallardo-Lopez, A; Munoz, A; Dominguez-Rodriguez, A
Ceramics International, **41** (2015) 12861-12868
December, 2015 | **DOI:** 10.1016/j.ceramint.2015.06.123

This work provides new insights on microstructure and electrical properties of 3 mol% Y2O3-ZrO2 (3YTZP) composites with 0.5, 1, and 1.5 vol% single walled carbon nanotubes (SWNTs). The composites were spark plasma sintered (SPS) in identical conditions at 1250 degrees C from powder prepared by two different processing routines, with the aim of optimizing the SWNTs dispersion throughout the ceramic matrix. High densification and submicrometric grain size were achieved in all the composites. Electrical properties of the composites were characterized in a wide temperature range, and modeling of the impedance properties was approached by means of an equivalent circuit that allows separation of the individual SWNT bundles contribution to resistance from the resistance due to junctions between bundles. Effects of the homogeneous distribution of SWNTs at the ceramic grain boundaries on the crystalline phases, percolation threshold, total conductivity and evolution of junctions' resistivity with temperature were analyzed and discussed.

Tribocorrosion behavior of TiBxCy/a-C nanocomposite coating in strong oxidant disinfectant solutions

Gracia-Escosa, E; Garcia, I; Sanchez-Lopez, JC; Abad, MD; Mariscal, A; Arenas, MA; de Damborenea, J; Conde, A
Surface & Coatings Technlology, **263** (2015) 78-85
February, 2015 | **DOI:** 10.1016/j.surfcoat.2014.12.047

Corrosion and tribocorrosion studies of a TiBxCy/a-C coating deposited on AISI 316L steel have been performed in an aqueous solution of 026 vol.% acetic, 0.16 vol.% peracetic and 0.18 vol.% hydrogen peroxide (commercial product Oxonia I vol.%). The corrosion current density of the TiBxCy/a-C coating ranges on the same order as bare steel but with a significantly decreasing friction (0.1 vs. 0.6) and wear rate (similar to 10 times lower). The compact microstructure of the coating hinders the access of the aggressive electrolyte to the substrate, preventing the onset of the corrosion attack, while maintaining an excellent tribological behavior in strong oxidant solutions.

Direct observation of doping incorporation pathways in self-catalytic GaMnAs

Kasama, T.; Thuvander, M.; Siusys, A.; Gontard, L. C.; Kovacs, A.; Yazdi, S.; Duchamp, M.; Gustafsson, A.; Dunin-Borkowski, R. E.; Sadowski, J.
Journal of Applied Physics, **118** (2015) 054302
August, 2015 | **DOI:** 10.1063/1.4927623

Doping mechanisms of Mn in GaAs nanowires (NWs) that have been grown self-catalytically at 600 °C by molecular beam epitaxy (MBE) are investigated using advanced electron microscopy techniques and atom probe tomography. Mn is found to be incorporated primarily in the form of non-magnetic tetragonal Ga0.82Mn0.18 nanocrystals in Ga catalyst droplets at the ends of the NWs, while trace amounts of Mn (22 ± 4 at. ppm) are also distributed randomly in the NW bodies without forming clusters or precipitates. The nanocrystals are likely to form after switching off the reaction in the MBE chamber, since they are partially embedded in neck regions of the NWs. The Ga0.82Mn0.18 nanocrystals and the low Mn concentration in the NW bodies are insufficient to induce a ferromagnetic phase transition, suggesting that it is difficult to have high Mn contents in GaAs even in 1-D NW growth via the vapor-liquid-solid process.

Transmission electron microscopy of thiol-capped Au clusters on C: Structure and electron irradiation effects

Gontard, L.C.; Dunin-Borkowski, R.E.
Micron, **70** (2015) 41-49
March, 2015 | **DOI:** 10.1016/j.micron.2014.12.001

High-resolution transmission electron microscopy is used to study interactions between thiol-capped Au clusters and amorphous C support films. The morphologies of the clusters are found to depend both on theis size and on the local structure of the underlying C. When the C is amorhous, larger Au clusters are cystalline, while smaller clusters are typically disordered. When the C is graphitic, the Au particles adopt either elongated shapes that maximize their contact with the edge of the C film or planar arrays when they contain few Au atoms. We demonstrate the influence of electron beam irradiation on the structure, shape and stability of the Au clusters, as well as on the formation of holes bounded by terraces of graphitic lamellae in the underlying C.

Atomic scale characterization of SiO_2/4H-SiC interfaces in MOSFETs devices

Beltran, AM; Duguay, S; Strenger, C; Bauer, AJ; Cristiano, F; Schamm-Chardon, S
Solid State Communications, **221** (2015) 28-32
November, 2015 | **DOI:** 10.1016/j.ssc.2015.08.017

The breakthrough of 4H-SiC MOSFETs is stemmed mainly due to the mobility degradation in their channel in spite of the good physical intrinsic material properties. Here, two different n-channel 4H-SiC MOSFETs are characterized in order to analyze the elemental composition at the SiC/SiO_2 interface and its relationship to their electrical properties. Elemental distribution analyses performed by EELS reveal the existence of a transition layer between the SiC and the SiO_2 regions of the same width for both MOSFETs despite a factor of nearly two between their electron mobility. Additional 3D compositional mapping by atom probe tomography corroborates these results, particularly the absence of an anomalous carbon distribution around the SiC/SiO_2 interface.

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

9th International Symposium Hydrogen & Energy
25 – 30 enero [Emmetten, Suiza]

Investigation of a Pt containing washcoat on SiC foam for hydrogen combustion applications
A. Fernández, G.M. Arzac, U.F. Vogt, C.F. Hosoglu, A. Borgschulte, M.C. Jiménez, O. Montes, A. Züttel
Comunicación oral

Preparation of Co and Co-B supported catalysts by magnetron sputtering: a step forward in understanding the active phase and deactivation processes in sodium borohydride hydrolysis
V. Godinho, G.M. Arzac, M. Paladini, D. Hufschmidt, L.C. Contard, M.C. Jiménez, A. Fernández
Comunicación oral

Hydrogen-oxygen recombination reaction for treatment of exhaust gases from fuel cells
G.M. Arzac, D. Hufschmidt, A. Fernández
Poster

VIII Congreso Ibérico de Tribología | IBERTRIB 2015
18 – 19 junio [Cartagema, España]

Tribological properties of TiC/a-C:H nanocomposite coatings prepared by HIPIMS
J.C. Sánchez-López, S. Dominguez-Meister, T.C. Rojas, M. Colasuonno, M. Bazzan, A. Pate
Comunicación oral

Biotribological behavior of anti-bacterial Ag-C coatings prepared by DC-pulsed magnetron sputtering
S. Domínguez-Meister, T. C. Rojas, J. C. Sánchez-López
Poster

5th International Conference on Accelerated Carbonation for Environmental and Materials Engineering
21 – 24 junio [Nueva York, Estados Unidos de América]

Improving technological viability of carbon sequestration technologies with structured agents and industrial wastes
L. Romero-Hermida, V. Morales-Florez, A. Santos, L. Esquivias
Comunicación oral

14th International Conference of the European Ceramic Society

21 – 25 junio [Toledo, España]

Effect of the addition of carbon nanotubes on the hydrothermal degradation of Y-TZP ceramics
F. Gutiérrez Mora, A. Gallardo López, R. Poyato, A. Muñoz, A. Domínguez Rodríguez
Poster

Exploring how the amount of carbon nanotubes at grain boundaries controls the tetragonal to monoclinic transformation of zirconia in 3Y-TZP/SWNT composites
A. Morales-Rodríguez;Á. Gallardo-López;R. Poyato;A. Muñoz;A. Domínguez-Rodríguez
Poster

Processing and Impedance Spectroscopy of Multifunctional 3YTZP/SWNT Ceramic Nanocomposites
R. Poyato, J. Macías-Delgado, Á. Gallardo-López, A. Muñoz;A. Domínguez-Rodríguez
Poster

Processing and room temperature properties of agglomerate-free 3Y-TZP / SWNTs Composites
Á. Gallardo-López, J. Vega-Padillo, A. Morales-Rodríguez, R. Poyato, A. Muñoz, A. Domínguez-Rodríguez
Poster

22ND international Symposium on Metastable, Amorphous and Nanostructured Materials | ISMANAN 2015

13 – 17 julio [Paris, Francia]

Proton elastic scattering cross-section on helium from 0.6 to 3.0 MeV at laboratory angles between 110 and 165 degrees
A. FernándezV. GodinhoJ. Caballero-Hernández
Conferencia invitada

Porous amorphous-Silicon coatings fabricated by magnetron sputtering with Helium as process gas
F.J. Ferrer, V. Godinho, B. Fernández, J.P. Fernández-García, J. Caballero-Hernández, A. Fernández-Camacho, J. Gómez-Camacho
Poster

16th European Conference On Applications of Surface and Interface Analysis ECASIA' 15

28 septiembre – 1 octubre [Granada, España]

On the self-lubricity and chemical resistance of WSex films
J.C. Sánchez-López, S. Dominguez-Meister, M. Conte, T.C. Rojas, A. Igartua
Comunicación oral

4th Magnetron, Ion Processing & Arc Technologies European Conference. 14th International Symposium on Reactive Sputter Deposition

8 – 11 diciembre [Paris, Francia]

Porous Amorphous-Silicon Coatings Fabricated By Magnetron Sputtering With Helium As Process Gas
V. Godinho, J. Caballero-Hernández, A. Fernández
Cnferencia Plenaria

CONGRESOS Y REUNIONES NACIONALES / NATIONAL CONGRESSES AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

Catálisis, Confluencia Interdisciplinar: Modelos, Catalizadores y Reactores SECAT'15

13 – 15 julio [Barcelona, España]

Óxidos mixtos basados en zirconio como conductores iónicos
N. García Moncada, R. Poyato Galán, F. Romero Sarria, M.A. Centeno Gallego, J.A. Odriozola Gordón
Poster

XIV Congreso Nacional de Tratamientos Térmicos y de Superficie | TRATERMAT 2015

30 septiembre – 1 octubre [Pontevedra, España]

Preparación de membranas de SAPO-34 soportadas sobre mallas metálicas
M. Romero, M.I. Domínguez, R. Poyato, J.A. Odriozola
Poster

Formacion / Training

Formación de Graduados / Master Degree Thesis

Título:	**Procesado y microestructura: su efecto sobre las propiedades eléctricas de compuestos de 3YTZP con nanotubos de carbono monocapa**
Autor:	Julio Macías Delgado
Directoras:	Dra. Rosalía Poyato Galán y Dra. Ángela Gallardo López
Grado:	Trabajo Fin de Master (Máster en Ciencia y Tecnología de Nuevos Materiales)
Año Académico:	2013-2014 (Junio 2014)

Docencia / Teaching

Máster “Láser, Plasma y Tecnología de Superficies”

Interacción de partículas y radiación con la materia láseres / Interacción de los electrones con el sólido

Asunción Fernández Camacho

Interacción de partí

Lugar: Universidad de Córdoba

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 detectores FID y TCD.

Buretas automáticas para medición de gases.

Cámaras de deposición de recubrimientos y películas delgadas y tratamientos con plasma.

Sistema de tratamiento con plasmas Diener.
Tres cámaras de deposición por la técnica de pulverización catódica (magnetrón sputtering). Con una dotación total de 7 cabezas magnetrón, 2 fuentes DC, 2 fuentes RF y 1 fuente pulsada, portamuestras girables, calentables y "biased".
Una cámara de deposición por la técnica de pulverización catódica con fuente HIPIMS.

Equipamiento para microscopía electrónica
Sistema de preparación de muestras en película delgada TXP de Leica.
Pulidora, trípode y microscopio óptico.
TEM de 300kV Tecnai F30 dotado de modo STEM, detector HAADF, analizador EDX Oxford Max80 y filtro de energías GIF Quantum.

Propiedades mecánicas de superficies y recubrimientos
Tribómetro CSM (movimiento lineal y rotativo) para evaluación de coeficientes de fricción y desgaste.
Tribómetro de alta temperatura (hasta 800ºC) –Microtest
Calotest para medida de espesores y evaluación del desgaste.
Equipo de rayado (Scratch-test) hasta 200N (Tribotechnic).
Perfilómetro-rugosímetro (Mahr) de tipo táctil y resolución vertical nanométrica.

Propiedades eléctricas de materiales
Sistema de medida de espectroscopía de impedancia compleja, formado por un impedancímetro Agilent modelo 4294A, un horno Hobersal ST115020, y una celda de medida hermética para la realización de medidas en atmósfera.

UNIDAD EXTERNA DE INVESTIGACIÓN: FÍSICA DE MATERIALES

EXTERNAL UNIT: PHYSICS OF MATERIALS

PERSONAL / PERSONNEL

Catedrático

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

Profesor Titular

Dr. Javier S. Blázquez Gámez
Dra. Josefa María Borrego Moro
Dra. María Dolores Estrada de Oya
Dra. M. del Carmen Gallardo Cruz
Dra. Angela Gallardo López
Dr. Felipe Gutiérrez Mora
Dr. José María Martín Olalla
Dra. Ana Morales Rodríguez
Dr. Francisco Javier Romero Landa

Investigador Contratado

Dr. Jhon J. Ipus Bados

Becario Predoctoral

Ldo. Luis M. Moreno Ramírez

PROYECTOS DE INVESTIGACIÓN / RESEARCH PROJECTS

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 / 31-12-2016
Organismo Financiador/Financial source: Ministerio de Ciencia e Innovación
Importe total/Total amount: 118.530,61 €
Investigador responsable/Research head: Alejandro Conde Amiano / Victorino Franco García
Componentes/Research group: Clara F. Conde Amiano, Josefa María Borrego Moro, Javier S. Blázquez Gámez , Jhon J. Ipus Bados, Laszlo F. Kish

RESUMEN / ABSTRACT

Se estudiarán materiales magnéticos con aplicacabilidad en sistemas para un uso eficiente de la energía, centrando nuestra atención, fundamentalmente, en dos aspectos interrelacionados: la refrigeración magnética y los materiales compuestos para la recuperación de energía. En lo referente al efecto magnetocalórico, se estudiarán materiales con una transición de fase de primer orden, en los que el control de la microestructura, la composición y campos externos como la presión o el campo eléctrico pueden alterar la respuesta termomagnética. Se abordará la problemática actual de dos familias de materiales magnetocalóricos con alto interés tecnológico: los compuestos del tipo LaFeSi y las aleaciones Heusler. Los primeros, especialmente los compuestos hidrogenados, son altamente prometedores para aplicaciones industriales, pero sufren una migración del hidrógeno que da lugar a una disminución del pico de respuesta magnetocalórica que se desdobla en dos picos menores. El control de la microestructura y la modificación de la composición debe dar lugar a una mejora en la estabilidad de las propiedades. Las aleaciones Heusler, a pesar de contar con una elevada respuesta magnetocalórica, presentan dificultades para su aplicación tecnológica, debido a la significativa irreversibilidad de la transición microestructural. Esfuerzos combinados de modificación de la composición y modelado de la transición de fase deben permitirnos minimizar esta histéresis térmica y mejorar la aplicabilidad de las aleaciones Heusler. El estudio experimental de materiales magnetocalóricos requiere de técnicas apropiadas para la caracterización de la variación de entropía magnética y de la variación de temperatura adiabática. La primera puede obtenerse mediante medidas indirectas de imanación, pero los protocolos de medida para materiales con una transición de fase de primer orden existentes en la actualidad implican procedimientos costosos en tiempo (y, consecuentemente, de alto coste económico). La modelización de las transiciones de fase nos permitirá diseñar algoritmos de medida más eficientes. Por otra parte, la caracterización de la respuesta dinámica de los materiales magnetocalóricos sólo puede hacerse mediante la medida

directa de la variación de temperatura adiabática, existiendo un número muy limitado de dispositivos comerciales que permitan realizar estas medidas de manera fiable, especialmente para muestras de reducida masa. Realizaremos estudios para desarrollar y optimizar un equipo de medida directa de la variación de temperatura adiabática para el estudio de la respuesta dinámica de los materiales, lo que tendrá implicaciones directas en el análisis de la viabilidad de emplear estos materiales en dispositivos refrigeradores. En especial, se estudiará la posibilidad de realizar medidas con excitación AC para la detección de pequeñas respuestas. El tercer bloque de estudio de este proyecto se centra en el análisis de otros efectos cruzados en materiales con transiciones de fase magnéticas o magnetoestructurales. Mediante el desarrollo de materiales multiferroicos artificiales, preparados por procedimientos combinados de aleado mecánico y "spin coating", estudiaremos la viabilidad de la recuperación de energía por medios termomagnéticos.

In this project, we will study magnetic materials with applicability in systems for energy efficiency. Our interest will be focused in two interrelated topics: magnetic refrigeration and composite materials for energy harvesting. Concerning magnetocaloric effect, we will study materials with a first order phase transition, which thermomagnetic response can be controlled through tailoring microstructure and composition. Other external excitations, such as pressure and electric field, can also affect the thermomagnetic response. We will focus on some of the currently open problems of two families of magnetocaloric materials with high technological interest: LaFeSi-type compounds and Heusler alloys. In hydrogenated compounds of the former family, despite their promising industrial applicability, hydrogen migrates yielding a reduction of the peak of the magnetocaloric response, which splits in two smaller peaks. The control of the microstructure and the compositional tailoring should lead to an enhancement of the stability of the properties. Heusler alloys, despite their high magnetocaloric response, show a strong irreversibility at the microstructural transition, which hinders their technological application. Tailoring the composition along with modelling of the phase transition should allow us to minimize the detrimental thermal hysteresis and to enhance the applicability of Heusler alloys. The experimental study of magnetocaloric materials requires appropriate techniques for the characterization of the isothermal magnetic entropy change and the adiabatic temperature change. Although the former can be obtained from indirect measurements of magnetization, the measurement protocols for materials with a first order phase transition used nowadays demand long time measurements (and, consequently, have a high economical cost). Modeling of the phase transitions will allow us to design more efficient measurement algorithms. On the other hand, the characterization of the dynamic response of the magnetocaloric materials can only be performed by direct measurements of the adiabatic temperature change. However, there is a reduced number of commercial devices supplying reliable measurements of this magnitude, especially for samples with a low mass. We will carry out studies for developing and optimizing a setup for the characterization of the dynamic response of materials. This will have direct implications in the analysis of the viability to use these materials in refrigerators. Particularly, we will study the viability to perform AC measurements to detect weak reponses. Finally, the analysis of cross-effects in materials with magnetic or magnetostructural transitions will be undertaken. Especially, we will study the viability for thermomagnetic energy harvesting through the development of artificial multiferroic materials prepared by combined mechanical alloying and spin coating techniques.

PROYECTOS EN LOS QUE PARTICIPAN INTEGRANTES DE LA UNIDAD

Avalanchas en Biofísica, Materiales y Plasmas

Código/Code:	MAT2015-69777-REDT
Periodo/Period:	27-11-2015 / 28-11-2017
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total (UEI)/Total amount (UEI):	30.000 €
Investigador responsable/Research head:	Eduard Vives-Santa-Eulalia (U. Barcelona)
Investigador responsable (UEI) Research head (UEI):	María del Carmen Gallardo Cruz
Componentes/Research group:	José María Martín Olalla, Francsico Javier Romero Landa

RESUMEN / ABSTRACT

Bajo la denominación genérica de “avalanchas” se incluyen una serie de fenómenos físicos que consisten en la respuesta discontinua, estocástica e intermitente de un sistema cuando se conduce de forma suave y continua: variaciones repentinas del parámetro de orden ocurren separadas por intervalos sin ninguna actividad aparente. Existen ejemplos muy variados y a distintas escalas espaciales y temporales que incluyen la fractura de un material poroso nanoestructurado bajo compresión, las transiciones de fase estructurales en sólidos, el ruido Barkhausen en un material ferromagnético cuando se imanta, algunos fenómenos de condensación y ebullición, la respuesta de los tejidos neuronales, las verdaderas avalanchas (aludes) de nieve, los terremotos o incluso los plasmas de fusión y las llamaradas solares. Las dimensiones espaciales, energéticas y las duraciones de las avalanchas suelen presentar distribuciones estadísticas que, si no son de tipo ley de potencias, en general tienen colas largas. Es decir su comportamiento se aleja del comportamiento gaussiano esperable. El objetivo de esta red es discutir desde una aproximación multidisciplinar y holística las diferencias y similitudes entre estos fenómenos y la posibilidad que se puedan describir bajo un marco físico-estadístico común. Tambien se pretende importar y exportar las técnicas experimentales y estadísticas para el estudio de las correlaciones temporales y espaciales de las avalanchas entre las diferentes disciplinas:

física de materiales, biofísica, geofísica, física de plasmas. El reto final sería contribuir de forma importante al conocimiento y a la mejora de los métodos de caracterización, evaluación de riesgos, predicción y alarma de estos fenómenos, desde las fracturas en materiales hasta los terremotos y las llamaradas solares.

Under the name of “avalanches” we designate many physical phenomena that consist in the discontinuous, stochastic and intermittent response of a system under smooth and continuous external driving: sharp variations of the order parameter occur separated by intervals with apparently no activity. There are many examples at different spatial and temporal scales including: the failure of nanostructured porous materials under compression, the structural

transitions in solids, Barkhausen noise in ferromagnetic materials, some condensation and boiling phenomena, the response of neural tissues, the real snow avalanches, earthquakes and even fusion plasmas and solar flares. The spatial and energetic dimensions as well as the durations of the avalanches display often power-law behavior or, at least, fat tails. Their behaviour clearly differs from the expected gaussian behaviour. The goal of this network is to discuss from a multidisciplinar and holistic approximation the differences and similarities of these phenomena and the possibility to describe all of them in a comon statistical mechanics framework. We also pretend to export and import the experimental and statistical analysis techniques for the study of the spatial and temporal correlations of the avalanches in the different disciplines: materials physics, biophysics, geophysics and plasma physics. The final goal will be to contribute to the knowledge and improvement of the methods for characterization, risk evaluation, forecast and alarm of these phenomena, from materials failure to earthquakes and solar flares.

CONVENIOS Y CONTRATOS / CONTRACTS AND AGREEMENTS

Caracterización de aceros eléctricos

Periodo/Period: 01-09-2014 / 30-09-2015
Organismo Financiador/Financial source: ThyssenKrupp Steel Europe AG
Investigador responsable/Research head: Victorino Franco García

Magnetocaloric research with vibrating sample magnetometers: Algorithms for data analysis and development of new measuring protocols

Periodo/Period: 20-01-2014 / 30-06-2015
Organismo Financiador/Financial source: Lake Shore Cryotronics Inc. (EE.UU.)
Investigador responsable/Research head: Victorino Franco García

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

Influence of hot compaction on microstructure and magnetic properties of mechanically alloyed Fe(Co)-based amorphous compositions

J.J. Ipus, J.M. Borrego, J.S. Blázquez, M. Stoica, V. Franco, A. Conde
Journal of Alloys and Compounds, **653** (2015) 546-551
DOI: 10.1016/j.jallcom.2015.09.074

Amorphous Fe75Nb10B15 and (Fe70Co30)75Nb10B15 alloys were prepared by mechanical alloying from the elemental constituents and hot compacted at different temperatures within the supercooled liquid region. After compaction, the microstructure studied by X-ray diffraction shows an increase in the crystalline fraction for both compositions. Magnetic properties and magnetic entropy change of compacted samples of the Co-free alloy were enhanced with respect to the powder sample, meanwhile, a deterioration in these properties was observed for the Co containing alloy after compaction. Both changes could be ascribed to an enrichment in boron in the amorphous phase.

Enhanced cryogenic magnetocaloric effect in $Eu_8Ga_{16}Ge_{30}$ clathrate nanocrystals
Anis Biswas, Sayan Chandra, Stevce Stefanoski, J.S. Blázquez, J.J. Ipus, A. Conde, M.H. Phan, V. Franco, G. S. Nolas, H. Srikanth
Journal of Applied Physics, **117** (2015) 033903
DOI: 10.1063/1.4906280

We observe an enhanced magnetic entropy change (−ΔSM) at cryogenic temperatures (T < 20 K) in Eu8Ga16Ge30clathrate (type-I) nanocrystals prepared by a ball milling method. With reduction in the crystal size to 15 nm, −ΔSM is enhanced at low temperatures, reaching the highest value (∼10 J/kg K) at 5 K for a field change of 5 T. For all samples investigated, there is a cross-over temperature (∼25 K) in −ΔSM (T) above which −ΔSM decreases with crystal size, opposite to that observed at low temperatures. A careful analysis of the magnetic and magnetocaloric data reveals that as the crystal size decreases the magnetic interaction between Eu2+ ions on the Eu2 site governing the primary ferromagnetic transition at ∼35 K becomes gradually weaker, in effect, altering the interaction between Eu2+ ions occupying the Eu1 and Eu2 sites responsible for the secondary ferromagnetic transition at 15 K. As a result, we have observed a strong change in magnetization and the enhancement of −ΔSM at low temperature.

Structure, magnetic properties and giant magnetocaloric effect of $Tb_4Gd_1Si_{2.035}Ge_{1.935}Mn_{0.03}$ alloy
J.X. Min, X.C. Zhong, V. Franco, H.C. Tian, Z.W. Liu, Z.G. Zheng, D.C. Zeng
Intermetallics, **57** (2015) 68-72
DOI: 10.1016/j.intermet.2014.10.002

$Tb_4Gd_1S_{i2.035}Ge_{1.935}Mn_{0.03}$ alloy was prepared by arc melting followed by annealing at 1193 K for 168 h. Structural characterizations reveal that monoclinic $(Tb, Gd)_5Si_2Ge_2$-type phase, secondary phase with orthorhombic 5-4 type structure and hexagonal 5-3 type structure coexist in the alloy. The paramagnetic Curie temperature (θp) is 120 K, indicating that the dominant exchange interaction is ferromagnetic or ferrimagnetic. That the thermal hysteresis of 13 K between heating and cooling and the negative slopes of Arrott plots derived from M–H curves between 116 K and 170 K confirm a typical first-order magnetic transition from ferromagnetism to paramagnetism occurs. The maximum magnetic entropy changes of the $Tb_4Gd_1Si_{2.05}Ge_{1.95}Mn_{0.03}$ alloy for magnetic field changes of 0–1 T, 0–2 T, 0–3 T, 0–4 T and 0–5 T are about 3.3, 8.6, 14.0, 18.9 and 22.4 J/kg K, respectively. And the effective refrigeration capacity (RCeff) value is 231 J/kg with a subtracted magnetic hysteresis loss of 30 J/kg for a magnetic field change from 0 to 5 T. Large −ΔSM and RCeff suggest that $Tb_4Gd_1Si_{2.035}Ge_{1.935}Mn_{0.03}$ alloy is an attractive potential magnetocaloric material working in the vicinity of 143 K.

Enhancement of magnetocaloric effect in B-rich FeZrBCu amorphous alloys
L.F. Kiss, T. Kemény, V. Franco, A. Conde
Journal of Alloys and Compounds, **622** (2015) 756-760
DOI: 10.1016/j.jallcom.2014.10.127

Magnetic properties and magnetocaloric effect were studied in the $Fe_{92-x}Zr_7B_xCu_1$ amorphous alloy series (x = 0–23 at.%). Enhancement of the magnetocaloric effect was observed in the B-rich side (x > 15 at.%) which correlates well with the anomalous increase of the saturation magnetic moment per Fe atom in this concentration range. Research into Fe2(B1−yETMy) amorphous alloys (ETM = Zr, Nb, Ti and V; y = 0–0.55 atomic fraction for Zr and to 0.25 atomic fraction for the rest) reveals an unexpected increase of the iron magnetic moment when early transition metals are substituted for B up to y = 0.1–0.25 atomic fraction. This behavior is attributed to the highly attractive B–ETM interaction. Similar mechanism is thought to explain the anomalous increase of the iron magnetic moment and hence the magnetocaloric effect in the boron-rich $Fe_{1-x}Zr_7B_xCu_1$ amorphous alloys. Universal scaling of the magnetic entropy change curves is also used to detect differences between $Fe_{92-x}Zr_7B_xCu_1$ alloys.

Analysis of magnetocaloric effect of ball milled amorphous alloys: Demagnetizing factor and Curie temperature distribution

L.M. Moreno-Ramírez, J.J. Ipus, V. Franco, J.S. Blázquez, A. Conde
Journal of Alloys and Compounds, **622** (2015) 606-609
DOI: 10.1016/j.jallcom.2014.10.134

The magnetocaloric effect (MCE) has been measured for an amorphous mechanically alloyed (MA) Co62Nb6Zr2B30. It is demonstrated that sample shape must be taken into account for a correct interpretation of the results, especially to correctly determine the field dependence of magnetic entropy change and refrigerant capacity. However, although correcting the influence of the demagnetizing factor allows us to recover the theoretically predicted behavior for these field dependencies, they differ from those obtained for a rapidly quenched (RQ) amorphous alloy of the same composition. This deviation is assigned to a broader Curie temperature distribution in MA alloys with respect to RQ alloys. Theoretical simulations based on the Arrott–Noakes equation of state and a Gaussian distribution of TC support this hypothesis.

Effect of alpha-Fe impurities on the field dependence of magnetocaloric response in LaFe11.5Si1.5

J.S. Blazquez, L.M. Moreno-Ramirez, J.J. Ipus, L.F. Kiss, D. Kaptas, T. Kemeny, V. Franco, A. Conde
Journal of Alloys and Compounds, **646** (2015) 101-105
DOI: 10.1016/j.jallcom.2015.06.085

In this work, the theoretical field dependence of the magnetic entropy change far away from the transition is used to analyze the field dependence of composite materials formed by fcc La(Fe,Si)13 and bcc α-Fe(Si) phases. A non-interacting phases approximation is followed in the analysis and results are in good agreement with microstructural data obtained from X-ray diffraction and Mössbauer spectroscopy. The range of validity of the approximation is estimated. It is concluded that the quadratic field dependence of magnetic entropy change is reached a few tens of kelvin above the transition temperature at 1.5 T. However, the linear dependence (characteristic of ferromagnets well below its Curie temperature) is only reached a few hundred kelvin below the transition. The results presented here can be used in the deconvolution of the contribution of impurities to the MCE signal in composites.

On the use of classical JMAK crystallization kinetic theory to describe simultaneous processes leading to the formation of different phases in metals

J.S. Blazquez, C.F. Conde, A. Conde
International Journal of Thermal Sciences, **88** (2015) 1-6
DOI: 10.1016/j.ijthermalsci.2014.09.004

The classical theory of Johnson–Mehl–Avrami–Kolmogorov (JMAK) is widely used to describe the kinetics of crystallization even when the premises required for its application are not strictly fulfilled. In this paper we propose a procedure to obtain the JMAK parameters of the independent transformations that simultaneously occur during a crystallization process (e.g. leading to the formation of several crystalline phases). The predictions of the analysis have been used to describe the crystallization process of two amorphous alloys with Fe90Zr10 and (Fe0.7Co0.3)Zr10 composition, respectively, which consists of two overlapped processes ascribed to the formation of α-Fe(Co) phase and a Zr-rich intermetallic.

Influence of microstructure on the enhancement of soft magnetic character and the induced anisotropy of field annealed HITPERM-type alloys

J.S. Blazquez, J. Marcin, M. Varga, V. Franco, A. Conde, I. Skorvanek
Journal of Applied Physics, **117** (2015) 17A301
DOI: 10.1063/1.4906173

Hitperm-type rapidly quenched ribbons were submitted to field annealing, both longitudinal field (LF) and transversal field (TF) to the axis of the ribbon. LF annealing yields a reduction of the magnetic anisotropy and results can be explained in the frame of random anisotropy model. A coercivity of 3 A/m is obtained for Fe39Co39 Nb 6B15 Cu 1 alloy. The addition of Cu to these Nb-containing Hitperm-type alloys is a key factor to refine the microstructure in order to reach this very low coercivity value. TF annealing produces samples with sheared hysteresis loops suitable for sensor and high frequency applications.

Characterization of the magnetic interactions of multiphase magnetocaloric materials using first-order reversal curve analysis

V. Franco, F. Beron, K.R. Pirota, M. Knobel, M.A. Willard
Journal of Applied Physics, **117** (2015) 17C124

In order to understand the magnetocaloric response of materials, it is important to analyze the interactions between the different phases present in them. Recent models have analyzed the influence of these interactions on the magnetocaloric response of composites, providing an estimate value of the interaction field that is consistent with experimental results. This paper analyzes to which extent magnetization first-order reversal curve (FORC) method can be used to calculate these interactions. It is shown that the different field ranges that are explored using these techniques (inside the hysteretic region for FORC; close to magnetic saturation for magnetocaloric effect) produce interaction field values that differ in order of magnitude, with FORC being sensitive to the lower values of the interaction field and magnetocaloric analysis accounting for the larger interactions.

Table-like magnetocaloric effect of Fe88-xNdxCr8B4 composite materials
J.W. Lai, Z.G. Zheng, X.C. Zhong, V. Franco, R. Montemayor, Z.W. Liu, D.C. Zeng
Journal of Magnetism and Magnetic Materials, **390** (2015) 87-90

The narrow working temperature range due to the sharp magnetic entropy change vertical bar Delta S-M vertical bar peak and large thermal or magnetic hysteresis restricts the practical application of magnetocaloric materials. In this work, the table-like magnetocaloric effect (MCE) was obtained in the multilayer composite of Fe88-xNdxCr8B4 alloys with various Nd substitutions for Fe (x=5, 8, 10, 12, and 15), which were prepared by arc-melting followed by melt-spinning. The substation of Nd was found to enhance the glass-forming ability. For the alloys with Nd substitution from 5 at% to 15 at%, the Curie temperature (T-C) ranged from 322 K to 350 K and the peak value of vertical bar Delta S-M vertical bar remained almost constant, 3.4-3.5 J/(kg K) under an applied held of 0-5 T The composite with various Nd contents was prepared by stocking the ribbons layer by layer. The vertical bar Delta S-M vertical bar of the composite approached a nearly constant value of similar to 3.2 J/(kg K) in a held change of 0-5 T in a wide temperature span over 40 K, resulting in large refrigerant capacity value of >408 J/kg. This vertical bar Delta S-M vertical bar value was much larger than the previous reported Fe-based amorphous composite Fe78-xCexSi4Nb5B12Cu1. This composite can be used as the working material in the Ericsson-cycle magnetic regenerative refrigerator around room temperature.

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

Analysis of the Magnetocaloric Effect in Powder Samples Obtained by Ball Milling
J.S. Blázquez, J.J. Ipus, L.M. Moreno-Ramírez, M. Borrego, S. Lozano-Pérez, V. Franco, C.F. Conde, A. Conde
Metallurgical and Materials Transactions E, **2** (2015) 131-138
DOI: 10.1007/s40553-015-0050-0

Since the discovery of the giant magnetocaloric effect (MCE) close to room temperature in FeRh and particularly in Gd5Si2Ge2 compounds, the study of this phenomenon has experienced an exponential growth. Among the different techniques used to produce magnetocaloric materials, ball milling has been shown as a very versatile one and presents several advantages over other preparation techniques (e.g., easy scale-up to industrial production). Although a general decrease of the peak value of the magnetic entropy change is observed for milled samples, it can be compensated by the large broadening of the MCE peak, leading to an increase of the refrigeration capacity. In this short review, several aspects inherent to powder samples affecting MCE will be discussed, such as the relevant effect of the demagnetizing field, the possible multiphase character, and the presence of Curie temperature distributions. In mechanically alloyed samples, the two latter factors are typically affected by the degree of integration of the different starting constituents.

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

144th Annual Meeting
15 – 19 marzo [Orlando, Estados Unidos de América]

Tips and tricks for the correct analysis of the field dependence of the magnetocaloric effect
V. Franco, L.M. Moreno-Ramírez, C. Romero-Muñiz, J.J. Ipus, J.S. Blázquez, A. Conde
Conferencia invitada

Intermag 2015
15 mayo [Beijing, China]

Dynamic effects in the characterization of the magnetocaloric effect of LaFeSi-type alloys
C. Romero-Torralva, C. Mayer, V. Franco, A. Conde
Comunicación oral

The influence of magnetocrystalline anisotropy on the magnetocaloric effect of Co2B
M. Fries, V. Franco, K.P. Skokov, O. Gutfleisch
Comunicación oral

Assessment of the magnetic entropy change of (La0.6Pr0.4)(Fe,Si)13 under cycling
B. Kaeswurm, V. Franco, K.P. Skokov, O. Gutfleisch
Poster

20th International Conference on Magnetism
5 – 10 julio [Barcelona, España]

Magnetocaloric effect in type-I Eu8Ga16Ge30 clathrate nanocrystals
H. Srikanth, A. Biswas, S. Chandra, S. Stefanoski, J. Blázquez, J. Ipus, A. Conde, M.H. Phan, V. Franco, G. Nolas
Conferencia invitada

Gd-Zn biphasic magnetic composites synthesized in a single preparation step: increasing refrigerant capacity without decreasing magnetic entropy change
J.Y. Law, L.M. Moreno-Ramírez, J.S. Blázquez, V. Franco, A. Conde
Comunicación oral

Study of the induced anisotropy in field annealed Hitperm alloys by Mössbauer spectroscopy and Kerr microscopy
J. S. Blázquez, J. Marcin, F. Andrejka, V. Franco, A. Conde, I. Skorvanek
Comunicación oral

Analysis of the field dependence of magnetocaloric effect at the transition temperatures for biphasic systems
J.J. Ipus, L.M. Moreno-Ramírez, J.S. Blázquez, V. Franco, A. Conde
Poster

2015 First Order Reversal Curve (FORC) Workshop
23 julio [Minneapolis, Estados Unidos de América]

FORC analysis of magnetocaloric materials
V. Franco
Conferencia invitada

22th Soft Magnetic Conference |SMM22
13 – 16 septiembre [Sao Paulo, Brasil]

Anisotropy field distribution in soft Hitperm-type alloys submitted to different field annealing processes
J.S. Blázquez, J. Marcin, F. Andrejka, V. Franco, A. Conde, I. Skorvanek
Comunicación oral

4th International Conference on rare earth materials Advances in Synthesis, Studies and Applications | REMat2015
26 – 28 octubre [Wroclaw, Polonia]

Rare earth based composites for magnetic refrigeration
V. Franco, J.Y. Law, L.M. Moreno-Ramírez, J.S. Blázquez, A. Conde
Conferencia invitada

FORMACION / TRAINING

FORMACIÓN DE GRADUADOS / MASTER DEGREE THESIS

Título:	**Efecto magnetocalórico y exponentes críticos en aleacciones NiFeSiB**
Autor:	Miriam Sánchez Pérez
Directores:	Victorino Franco García
Grado:	Trabajo Fin de Master (Ciencia y Tecnología de Nuevos Materiales)
Año Académico:	2014-2015 (2 Julio 2015)
Calificación:	10

Título:	**Modelos cinéticos de cristalización**
Autor:	Alejandro F. Manchón Gordón
Directores:	Javier S. Blázquez Gámez / Clara F. Conde Amiano

Grado:	Trabajo Fin de Grado
Año Académico:	2014-2015 (13 Julio 2015)
Calificación:	Notable (8,5)

Título:	**Espectroscopía Mössbauer en función de la temperatura**
Autor:	Rodrigo Gómez Rodríguez
Directores:	Javier S. Blázquez Gámez / Jhon J. Ipus Bados
Grado:	Trabajo Fin de Grado
Año Académico:	2014-2015 (13 Julio 2015)
Calificación:	Notable (8)

Docencia / Teaching

Máster Profesorado de ESO y Bachillerato, FP

Complementos de formación disciplinar en Física y Química
Dr. Javier S. Blázquez
Lugar: Universidad de Sevilla

Equipamiento Científico Available Equipment

- Calorímetro de barrido diferencial (Perkin-Elmer DSC7)
- Criostato para espectrómetro Móssbauer
- Balanza termogravimétrica (Perkin-Elmer TGA-7)
- Espectrómetro Mössbauer (Wissel MB-500) con horno y criostato.
- Coercímetro (desarrollado en el laboratorio)
- Magnetómetro de muestra vibrante (LakeShore 7000) con horno y criostato.
- Equipo de medida directa de temperatura adiabática (Advanced Magnetic Technologies).
- Molino Planetario (Fritszch Pulverisette Vario 4)
- Equipo de solidificación por enfriamiento ultrarrápido (melt spinning, Bühler)
- Equipo de fusión por arco (MAM1, Bühler)
- Calorímetro de conducción, resolución en la medida del flujo de calor mejor que 0,1⍰W, fluctuaciones en temperatura del orden de 10-6 K, velocidad de barrido menor de 0,01K/h, rango 80-320K, es posible aplicar tensión uniaxial hasta 30 kg/cm2 y campo eléctrico hasta 800V/cm
- Calorímetro de conducción, rango 80-400K, campo eléctricos 2000V/cm
- Medida de constante dieléctrica, en los mismos calorímetros,
- Medida de ciclo de histéresis en Ferroeléctricos.
- Analizador de Impedancia

SERVICIOS GENERALES

GENERAL SERVICES

SERVICIO DE ESPECTROSCOPIAS / SPECTROSCOPY SERVICE

El Servicio de Espectroscopias incluye las Unidades de Espectroscopía de Resonancia Magnética Nuclear, Espectroscopía Raman, Espectroscopía Infrarroja y Espectroscopía Ultravioleta-Visible. Este servicio está dedicado a la determinación de la estructura molecular de los compuestos químicos y la caracterización de materiales.

This Service consists of four different spectroscopies: Nuclear Magnetic Resonance Spectroscopy, Raman Spectroscopy, Infrared Spectroscopy and Ultraviolet-Visible Spectroscopy. It is devoted to the determination of molecular structure of chemical compounds and materials.

RESONANCIA MAGNÉTICA NUCLEAR EN SU CONFIGURACIÓN DE ESTADO SÓLIDO / NUCLEAR MAGNETIC RESONANCE IN ITS SOLID-STATE CONFIGURATION

La Resonancia Magnética Nuclear en estado sólido se utiliza, fundamentalmente, para el estudio de la estructura atómica de materiales amorfos o poco cristalinos. Se basa en el comportamiento de los momentos magnéticos de ciertos núcleos atómicos bajo la influencia de un campo magnético externo y de los campos locales creados por los núcleos vecinos y por la distribución de carga electrónica en torno al núcleo. Los espectros más comunes son representaciones de la intensidad de absorción frente a la frecuencia de resonancia y presentan señales cuya posición, forma y tamaño están íntimamente relacionados con el entorno químico del núcleo estudiado. Permite el estudio del orden a corto alcance de materiales estructuralmente desordenados y diferencia átomos con números atómicos similares.

Solid State Nuclear Magnetic Resonance is a widely use technique to study the structural properties of materials, including amorphous and not very crystalline solids. This technique is based on the behavior of the magnetic moments of the nuclei under an external magnetic field and the local magnetic field due to the environment. Thus, the position, shape and intensity of the signals inform of the chemical environment of the nuclei.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- Espectrómetro de RMN multinuclear Bruker, mod. DRX-400
  Multinuclear RMN Spectrometer (Bruker, mod. DRX-400)
- Sonda CP/MAS de 4 mm dotada de doble canal, 1H y BB (en el rango de frecuencia desde 15N a 31P)
  CP/MAS sound of 4 mm with double channel, 1H and BB (at a frequency range from 15N to 31P)
- Unidad neumática, que permite alcanzar velocidades de giro bajo ángulo mágico de hasta 15000 Hz
  Neumatic unit, that allows to reach turn velocities under a magic angle up to 15000 Hz

- Accesorio de temperatura variable dotado de una línea propia de N_2, así como de un sistema de control de temperatura que permite trabajar en un rango entre 223 K y a 373 K.
  Accessory of variable temperature with a line of its own of N_2, and with a temperature control system that allows to work at a range of 223 K - 373 K.

Responsable Científico/Scientific Responsible: Dra. María Dolores Alba Carranza
Personal Técnico/Technical Assistant: Dr. Miguel Angel Avilés Escaño

ESPECTROSCOPÍA MICRO-RAMAN / MICRO-RAMAN SPECTROSCOPY

La espectroscopía Raman se basa en un proceso fotónico en el que la radiación incidente es dispersada por la muestra, produciéndose transiciones de tipo vibracional y rotacional. En general, el espectro Raman se interpreta como un espectro vibracional que ofrece información muy similar al espectro de infrarrojo, aunque las vibraciones que se ven reflejadas en el espectro Raman no son siempre las mismas que en aquél. Para que un modo vibracional sea activo en espectroscopía Raman es necesario que se produzcan cambios en la polarizabilidad de los enlaces químicos o la molécula considerada, lo que conlleva la producción de momentos dipolares inducidos. Su campo de aplicación es muy extenso: semiconductores, compuestos del carbono (grafito, diamante, nanotubos, fibras…), catalizadores, pigmentos, etc.

Raman spectroscopy is based on a photonic process in which the incident radiation is dispersed by the sample. This latter is perturbed leading to vibrational and rotational transitions. In general, the Raman spectrum is interpreted like a vibrational one, providing information very similar to the infrared spectroscopy, although the Raman active vibrations are not always the same as those excited with infrared radiation. A Raman vibration mode is active if there is a change of polarizability of the chemical bonds or the considered molecule, which in turn results in the generation of induced dipolar momentam. Its application fields are very broad: semiconductors, carbon compounds (graphite, diamond, nanotubes, fibers…), catalysts, pigments, etc.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- LabRAM Horiba Jobin Yvon dotado de un microscopio confocal y 3 longitudes de excitación (785 cm^{-1} rojo, 532 cm^{-1} verde, y 325 cm^{-1} UV)
  LabRAM Horiba Jobin Yvon equipped with a confocal microscope and 3 excitation lasers (785 cm^{-1} red, 532 cm^{-1} green, and 325 cm^{-1} UV)

Responsables Científicos/ Scientific Responsibles: Dr. Juan Carlos Sánchez López, Dr. Hernán Míguez García y Dr. Miguel Angel Centeno Gallego
Personal Técnico/ Technical Assistant: Dr. Miguel Angel Avilés Escaño

ESPECTROSCOPÍA INFRARROJA / INFRARED SPECTROSCOPIES

La espectroscopía de Infrarrojos (FT-IR) se basa en la absorción de radiación infrarroja por parte de los materiales. Esta absorción supone un cambio en la energía vibracional de los enlaces, siempre que se produzca un cambio en la polarización de dicho enlace. El resultado obtenido es un espectro en el que se representa la radiación absorbida o transmitida en función del número de onda de la radiación, lo cual permite identificar el enlace correspondiente.
El equipo en el ICMS cubre un rango de número de ondas que va desde 5000 a 250 cm-1 (óptica de CsI) y se puede trabajar con purga o en vacío. Se halla equipado con accesorios para trabajar en los modos de Reflectancia Difusa (DRIFT), Reflectancia Total Atenuada (ATR) y Reflexión Especular. Dispone de un microscopio de Infrarrojos que tiene una resolución lateral de 10 µm.

Infrared spectroscopy (FT-IR) is based on the selective absorption of the infrared radiation by the materials. This absorption means a change in the vibrational energy of the chemical bonds, whenever it occurs a change in the polarization. The result is a spectrum showing the absorbed or transmitted radiation as a function of the wavenumber of the radiation, which can be assigned to the corresponding chemical bound.
The equipment at the ICMS works in a wavenumber range from 5000 to 250 cm^{-1} (CsI optic), and can operate with a gas purge or in vacuum. It is equipped with several accessories to do Diffuse Reflectance (DRIFT), Attenuated Total Reflectance (ATR) or Specular Reflectance. It has got an Infrared Microscope with a lateral resolution of 10 µm.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- JASCO FT/IR-6200 IRT-5000
  JASCO FT/IR-6200 IRT-5000

Responsables Científicos/ Scientific Responsibles: Dr. Manuel Ocaña Jurado y Dr. Ángel Barranco Quero
Personal Técnico/ Technical Assistant: Dr. Miguel Angel Avilés Escaño

ESPECTROSCOPÍA ULTRAVIOLETA / ULTRAVIOLET-VISIBLE SPECTROSCOPIES

La espectroscopía Ultravioleta-Visible (UV-Vis) informa sobre las diferencias de energía existentes entre los niveles electrónicos ocupados más externos y los desocupados más próximos.
Se dispone en el ICMS de dos equipos que cubren el rango de longitudes de onda que va desde 190 nm hasta 900 nm. Se puede trabajar tanto en el modo de Transmisión como en el de Reflectancia Difusa.

The Ultraviolet-Visible Spectroscopy (UV-Vis) reports on the existing energy differences between the more external occupied electronic levels and the nearer unoccupied ones.
There are two equipments in the laboratory, which work in the wavelength range of 190 nm to 900 nm. It can operate in the Transmission mode or in Diffuse Reflectance Modes.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- SHIMADZU UV-2101 PC
  SHIMADZU UV-2101 PC
- Perkin Elmer Lambda 12
  Perkin Elmer Lambda 12

Personal Técnico/ Technical Assistant: Dr. Miguel Angel Avilés Escaño

SERVICIO DE ANÁLISIS TEXTURAL Y TÉRMICO / TEXTURAL AND THERMAL ANALYSIS SERVICE

Este servicio incluye las siguientes Unidades: Análisis Térmico, Fisi-quimisorción, Análisis de Tamaño de Partícula y Potencial Z. Está dedicado a la determinación de la textura, estructura y comportamiento térmico de los materiales.

This Service includes the following units: Thermal Analysis, Physisorption and Chemisorption, Particle Size and Z-potential determination. It is devoted to the characterization of texture, microstructure and thermal behavior of advanced materials.

FISI-QUIMISORCIÓN / PHYSISORPTION-CHEMISORPTION

Este servicio constituye una herramienta básica para la caracterización microestructural de sólidos pulverulentos de distinta naturaleza, en cuanto a porosidad, superficie específica y superficie químicamente activa.

En el servicio se dispone de un analizador de adsorción de gases (Micromeritics, ASAP 2020) que proporciona isotermas de adsorción y desorción, a partir de los cuales se obtienen de ellas la superficie específica y distribución del tamaño de poro y de microporo de estos materiales, incorporando también los accesorios necesarios para medidas de quimisorción.

This service constitutes a basic tool for the microstructural characterization of powdered solids of different natures, regarding to their porosity, specific surface area and chemically active surface.

This service is composed by a phyisisorption analyser (Micromeritics, ASAP 2020) which provides the complete adsorption/desorption isotherms, from which the specific surface area, pore and micropore size distribution and concentration of reactive sites are obtained. The instrument is also equipped for carrying out chemisorption of different reactive molecules, as O_2, H_2, CO, etc.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- Analizador científico de fisisorción ASAP2010 (Micromeritics)
  Physisorption analyser ASAP 2010 (Micromeritics)
- Analizador de quimisorción ASAP2010 (Micromeritics)
  Chemisorption analyser ASAP 2010 (Micromeritics)
- Analizador de fisisorción multimuestra TRISTAR II (Micromeritics)
  Multisample physisorption analyser TRISTAR II (Micromeritics)
- Analizador de fisisorción multimuestra TRISTAR II-Kr (Micromeritics)
  Multisample physisorption analyser TRISTAR II-Kr (Micromeritics)

Responsables Científicos/ Scientific Responsibles: Dr. Gerado Colón Ibáñez y Dr. Miguel Angel Centeno Gallego
Personal Técnico/ Technical Assistant: Dª Cristina Gallardo López

ANÁLISIS TÉRMICO / THERMAL ANALYSIS

Las técnicas de análisis térmico permiten estudiar aquellos cambios físicos o químicos que ocurren en los sólidos en función de la temperatura y que conlleven modificaciones en su masa o intercambios de calor con su entorno.
En el servicio se pueden realizar experimentos desde temperatura ambiente hasta 1500ºC, tanto en atmósfera inerte (N_2) como reactiva (aire, O_2,…).
Se dispone de dos técnicas: Análisis Termogravimétrico (TG) y Análisis Térmico Diferencial (ATD).

Thermal analysis techniques allow to studying physical or chemical changes occurring in solid in samples as a function of the temperature. Those changes should involve either a mass change or a heat flow.
The experiments can be performed in the range from room temperature to 1500ºC, both under inert (N_2), or reactive (air, O_2,…) atmospheres.
Two different techniques are available: Thermogravimetry (TG) and Differential Thermal Analysis (DTA)

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- Equipo de análisis térmico simultáneo TG/ATD/CDB TA Instruments Q600
  TA Instruments Q600 Simultaneous TG/DTA/DSC instrument
- Equipo termogravimétrico TG, TA Instruments Q5000
  Thermogravimetric instrument TG, TA Instruments Q5000

Responsable Científico/ Scientific Responsible: Dr. Luis A. Pérez Maqueda
Personal Técnico/ Technical Assistant: Dª Cristina Gallardo López

TAMAÑO DE PARTÍCULAS Y POTENCIAL Z / PARTICLE SIZE AND Z POTENTIAL ANALYSIS

Se dispone de las técnicas de Dispersión Dinámica de Luz y de Difracción Láser (LD), que permiten determinar la distribución de tamaños de partícula de sistemas coloidales en suspensión (disolvente acuoso u orgánico) en los rangos que van de 3 a 3000 nanómetros (DLS) o de 0.05 a 900 micras (LD).

Así mismo, se dispone de la técnica de Análisis de Movilidad Electroforética para la evaluación del potencial "Z" de sistemas coloidales en suspensión (disolvente acuoso u orgánico).

Dynamic light scattering (DLS) and Laser diffraction (LD) are available for the determination of particle size distributions of colloidal systems (dispersed in aqueous or organic dispersions solutions) in the range 3-3000 nanometers (DLS) and 0.05-900 microns (LD).

Electrophoretic mobility measurements can be also performed for the evaluation of Z potential in colloidal systems (aqueous or organic dispersions).

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- Malvern modelo Zetamaster (DLS) y MalvernSizer (LD)
  Malvern model Zetamaster (DLS) and MalvernSizer (LD)

Responsable Científico/ Scientific Responsible: Dr. Manuel Ocaña Jurado
Personal Técnico/ Technical Assistant: Dª Cristina Gallardo López

SERVICIO DE MICROSCOPÍA ELECTRÓNICA / ELECTRON MICROSCOPY SERVICE

El servicio está dedicado a la caracterización química y estructural de muestras sólidas mediante técnicas de microscopía electrónica. Las técnicas de caracterización disponibles en el servicio son la Microscopía Electrónica de Transmisión (TEM) y la Microscopía Electrónica de Barrido (SEM), con el equipamiento anexo de preparación de muestras para TEM y SEM.

This Service is devoted to the chemical and structural characterization of solid samples by means of electron microscopies. The characterization techniques available at ICMS are Transmission Electron Microscopy (TEM) and Scanning Electron Microscopy (SEM), with additional equipment for TEM and SEM sample preparation.

MICROSCOPÍA ELECTRÓNICA DE BARRIDO / SCANNING ELECTRON MICROSCOPY

La microscopía electrónica de barrido proporciona información microestructural, morfológica y de composición química en escala microscópica. Se puede aplicar a todo tipo de materiales y problemáticas de estudio en ciencia de materiales: cerámicas, plásticos, metales, minerales, catalizadores, muestras de patrimonio histórico, capas finas, recubrimientos, interfases, nanopartículas, etc. El equipo SEM es un microscopio de emisión de campo de cátodo frío que permite realizar imágenes de la morfología y textura superficial de las muestras con una resolución de 1 nm a 15kV. También permite trabajar a bajo voltaje en muestras sin metalizar y en modo transmisión (STEM-in-SEM) en muestras electrón-transparentes. Acoplado al detector de rayos-X (EDX) permite análisis elementales y mapas composicionales.

The scanning electron microscopy provides information about the microstructure, morphology and chemical composition at the microscopic scale of solid samples. It can be applied to all type of materials including ceramics, polymers, metals, minerals, catalysts, samples from cultural heritage, thin films, coatings, interfaces, nanoparticles, etc. The SEM microscope is a field emission cold cathode equipment which enables images of the surface morphology and texture of samples with a resolution of 1 nm at 15kV. It also allows working at low voltages with non-metalized samples and in transmission mode for electron-transparent samples (STEM-in-SEM). Coupled to the X-ray detector (EDX) enables compositional analysis and elemental mapping.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- Microscopio SEM, modelo Hitachi S4800 SEM-FEG: cañón de emisión de campo de cátodo frío y voltaje de 0.5-30 kV, resolución de 1 nm a 15kV. Dotado de analizador EDX Bruker-X Flash-4010 con una resolución de 133 eV (en la línea MnKα) y detector con portamuestras para trabajar en modo transmisión (STEM-in-SEM).
  Hitachi S4800 SEM-FEG microscope: cold cathode field emission gun with voltage from 0.5 to 30 kV, resolution of 1nm at 15 kV. Equipped with a Bruker-X Flash-4010 EDX detector with a resolution of 133 eV (at the MnKα line), and a detector with sample holder to work in transmission mode (STEM-in- SEM).
- Equipamiento adicional en el “laboratorio de preparación de muestras para microscopía electrónica” (ver sección abajo)
  Additional equipment in the “electron microscopy samples preparation laboratory” (see the section below)

Responsable Científico/ Scientific Responsible: Dra. Asunción Fernández Camacho
Personal Técnico/ Technical Assistant: Dra. M. Carmen Jiménez de Haro

MICROSCOPÍA ELECTRÓNICA DE TRANSMISIÓN / TRANSMISSION ELECTRON MICROSCOPY

La microscopía electrónica de transmisión es una técnica ampliamente utilizada para la caracterización estructural y química de materiales a escala microscópica y nanoscópica, proporcionando imágenes bidimensionales de la textura de la muestra, forma y tamaño de grano y/o de partícula, grado de homogeneidad a escala microscópica, grado de cristalinidad de la muestra, identificación de fases cristalinas, e imágenes de alta resolución que identifican dominios cristalinos. El equipo está dotado de un analizador EDX para el análisis composicional. Puede aplicarse a todo tipo de materiales y campos de estudio en ciencia y tecnología de materiales trabajando sobre muestras electrón-transparentes preparadas en su caso ad-hoc para este fin. El servicio realiza microscopía en modo transmisión: Imágenes en campo claro y campo oscuro, difracción de electrones de área selecta y microscopía electrónica de alta resolución así como análisis elemental de area selecta. No se dispone de modo STEM.

The transmission electron microscopy is a widely used technique for the microstructural and chemical characterization at micro and nanoscales, providing two-dimensional images of the sample texture and shape as well as grain and/or particle size, degree of homogeneity at the microscopic scale, degree of crystallinity of the sample, identification of crystalline phases, and high resolution images to identify the crystalline domains. The microscope is equipped with an EDX analyzer for compositional analysis. It can be applied to all type of materials and research topics in materials science and technology working with electron-transparent samples prepared ad-hoc for this end. The service performs transmission electron microscopy: Imaging in bright and dark field, selected area electron diffraction and high resolution electron microscopy, as well as elemental analysis of selected areas. It does not provide STEM mode.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- Microscopio Philips CM20 (200kV) con una resolución estructural de 0.14 nm entre líneas y 0.23 nm entre puntos, portamuestras de uno y dos giros y de calentamiento. Acoplados al equipo se dispone de un sistema de análisis por Energías Dispersivas de Rayos X (EDX X-Max 80T, Oxford Instruments) y una cámara CCD (Gatan) para registro de imágenes.
  Philips CM200 microscope (200kV) with a structural resolution of 0.14 nm between lines and 0.23 nm between points. Sample holders with one and two angles and heating. It is equipped with a X-ray Energy Dispersive Analyzer (EDX X-Max 80T, Oxford Instruments) and a CCD GATAN camera for image acquisitions.
- Equipamiento adicional en el “laboratorio de preparación de muestras para microscopía electrónica” (ver sección abajo)
  Additional equipment in the “electron microscopy samples preparation laboratory” (see the section below)

Responsable Científico/ Scientific Responsible: Dra. Asunción Fernández Camacho
Personal Técnico/ Technical Assistant: Dª Olga Montes Amorín (CicCartuja) y Dª María Inmaculada Roja Cejudo

LABORATORIO DE PREPARACIÓN DE MUESTRAS PARA MICROSCOPÍA ELECTRÓNICA / ELECTRON MICROSCOPY SAMPLES PREPARATION LABORATORY

El laboratorio de preparación de muestras para TEM y SEM dispone de metalizador de oro, evaporador de carbón, metalizador de Cr y carbón, cortadora de disco, pulidora, “disc-grinder”, cortadora ultrasónica, pulidora cóncava (dimple) y adelgazador iónico (Fischione 1010).

The laboratory for TEM and SEM samples preparation has a gold coater, a carbon evaporator, a metallization system for Cr and carbon, a diamond wheel cutter, a grinder with disc-grinder device, an ultrasonic cutter, a concave polishing (dimple) and ion thinning (Fischione 1010).

Responsable Científico/ Scientific Responsible: Dra. Asunción Fernández Camacho
Personal Técnico/ Technical Assistants: Dª María Inmaculada Roja Cejudo, Dª Olga Montes Amorín (CicCartuja) y Dra. M. Carmen Jiménez de Haro

SERVICIO DE DIFRACCIÓN DE RAYOS X
X-RAY DIFFRACTION LABORATORY SERVICE

La difracción de rayos-X permite la identificación cualitativa y cuantitativa de sustancias cristalinas y su caracterización microestructural y textural.
El servicio dispone en la actualidad de cuatro difractómetros independientes, configurados específicamente para abordar el análisis de muestras policristalinas de muy distinta naturaleza, en lo referente a su composición, estabilidad química, cristalinidad, etc.
Asimismo, con alguno de ellos se pueden llevar a cabo, además de los análisis rutinarios (θ-2θ), otros varios mas avanzados, como pueden ser:

- Seguir las transformaciones de fase “in situ” provocadas por calentamientos en atmósfera inerte (vacio, Ar) o reactiva (H_2, O_2,..).
- 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/ MECHANICAL WORKSHOP

Se trata de un servicio horizontal fundamental para el Instituto y unidades externas adscritas al mismo, puesto que, además del mantenimiento del material y equipamiento científico, permite su mejora y adaptación a los distintos experimentos en curso, y de acuerdo con las necesidades de los investigadores y/o los proyectos que se llevan a cabo.

The Machining workshop is a basic and essential service that provides technical support to the maintenance, change, fitting and development of simple scientific equipments, according with the current requirements of the experiments and projects carried out by the researchers at the ICMS.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

Además de otras herramientas menores para cortar, soldar, doblar, taladrar, etc., el taller de mecanizado cuenta con los siguientes equipos:
Besides other minor tools for cutting, soldering, folding, drilling, etc, this workshop is equipped with:

- Centro de mecanizado de control numérico computerizado (CNC), modelo HASS TM1P
  A computer numeric control turing machine, model HASS TM1P
- Taladro vertical, modelo ERLO TSAR32
  A vertical drill, model ERLO TSAR32
- Torno paralelo manual EMCO SUPER 11CD y torno semiautomático PINACHO SMART TURN180
  A couple of turning machines: models PINACHO TURN180 and SUPER 11 CD EMCO

Responsable/ Responsible: Ing. D. Enrique Jiménez Roca
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

www.masteroficial.us.es

UNIVERSIDAD D SEVILLA

Título Oficial
de Máster
Universitario
en CIENCIA Y
TECNOLOGÍA DE
NUEVOS MATERIALES

Organizado por la Universidad de Sevilla
Coordinador: Dr. Joaquín Ramírez Rico
Fecha de Celebración: Curso Académico 2014-15
Créditos necesarios: 60
Dirigido a: Licenciados en Química, Física, así como, los Titulados en Ingeniería afines (Ingenieros Químicos, de Materiales, etc.)
Especialidades: Materiales para la Energía y el Medio Ambiente. Ingeniería de Superficies, Materiales Estructurales y Funcionales.
Mención de Calidad

Química del Estado Sólido (Créditos: 5) | Dra. Pilar Malet Maenner
Fisica del Estado Sólido (Créditos: 5) | Dra. Angela Gallardo López | Dra. Josefa Borrego | Dr. Manuel Jiménez Melendo
Técnicas de Caracterización de Materiales (Créditos: 8) | Dr. Julián Martínez Fernández | Dr. Juan M. Montes Martos (US)
Síntesis de Materiales y Nanoestructuras (Créditos: 7) | Dra. María Dolores Alcalá | Dra. Svetlana Ivanova | Dr. Francisco J. Gotor Martínez
Catalizadores para la Energía y el Medio Ambiente (Créditos: 5) | Dr. José Antonio Odriozola Gordón
Materiales con Funcionalidad Superficial (Créditos: 5) | Dra. Rosa María Pereñiguez Rodríguez
Recuperación y Transformación de Materiales (Créditos: 5) | Dra. Svetlana Ivanova
Procesado de Materiales Estructurales (Créditos: 5) | Dr. Alfonso Bravo León | Dr. José Antonio Rodríguez Ortíz
Comportamiento Mecánico (Créditos: 5) | Dr. Arturo Domínguez Rodríguez | Dr. Diego Gómez García
Comportamiento Magnético (Créditos: 5) Dr. Javier S. Blázquez Gámez | Dr. Victorino Franco García
Comportamiento Térmico, Dieléctrico y Óptico de Materiales (Créditos: 5) | Dr. Hernán Míguez García | Dr. Francisco Javier Romero Landa | Dra. Carmen Gallardo Cruz
Computación en Ciencia de Materiales (Créditos: 5) | Dra. Regla Ayala Espinar | Dr. Antonio Córdoba Zurita (US) | Dr. Felipe Gutiérrez Mora

Asimismo, el personal del ICMS ha impartido cursos regulares de primer y segundo ciclos de las correspondientes Licenciaturas y/o Grados en Química, Física y Farmacia e Ingeniería de Materiales y Biología en la Universidad de Sevilla.

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

11 de junio | **The Magnetocaloric Effect: From Energy Efficient Refrigeration to Fundamental Studies of Phase Transitions**
Dr. Victoino Franco García
Institute for Energy Technology, Physics Department, Kieller, Noruega

5 de octubre | **Ciencia de Materiales: Una herramienta multidisciplinar y una vía para la internacionalización de la tecnología**
Dr. José Antonio Odriozola Gordón
Conferencia Inaugural de la XIX Jornada de la Investigación en la Universidad Pedagógica y Tecnologica de Colombia (UPTC), Tunja, Colombia

11 de diciembre | **Analysis of the Magnetocaloric Effect in Powder Samples Obtained by Ball Milling**
Dr. Javier S. Blázquez Gámez
Reunión Anual del Capítulo Español de IEEE Magnetics y el Club Español de Magnetismo (CEMAG), Sevilla

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

24 de marzo| **Perspectivas de Investigación en Catálisis en Colombia – Análisis desde la Universidad Pedagógica y Tecnológica de Colombia**
Dr. Hugo Rojas Sarmiento y Dra. Julie J. Murcia
Grupo de Catálisis de la Universidad Pedagógica y Tecnológica de Colombia

7 de mayo| **Prospects for Plasmonic Nanomaterials**
Dr. Luis M. Liz-Marzán
Bionanoplasmonics Laboratory, CIC biomaGUNE

Metal nanoparticles display very interesting optical properties, related to localized surface plasmon resonances (LSPR), which give rise to well-defined absorption and scattering peaks in the visible and near-IR spectral range. Such resonances can be tuned through the size and shape of the nanoparticles, but are also extremely sensitive towards dielectric changes in the near proximity of the particles surface. Therefore, metal nanoparticles have been proposed as ideal candidates for biosensing applications. Additionally, surface plasmon resonances are characterized by large electric fields at the surface, which are responsible for the so-called surface enhanced Raman scattering (SERS) effect, which has rendered Raman spectroscopy a powerful analytical technique that allows ultrasensitive chemical or biochemical analysis, since the Raman scattering cross sections can be enhanced up to 10 orders of magnitude, so that very small amounts of analyte can be detected. This seminar will focus on recent developments and future prospects within the various areas involved in the development and application of plasmonic nanomaterials, from nanoparticle synthesis and assembly, through optical modeling, to the design of engineered substrates for sensing and diagnostics.

21 de mayo| **TOF-SIMS for the analysis of surfaces**
Dr. Miolaj Berczenko
University of Rzeszow (Polonia)

28 de mayo| **Silicon nanocrystals as enabler for silicon photonics**
Prof. Lorenzo Pavesi
Universidad de Trento (Italia)

Low dimensional silicon nanocrystals (Si-nc) have interesting optical properties which enables different applications. On one side quantum confinement effects turn silicon into a luminescent material where luminescence can be also excited by electrical injection. On the other side, small sizes, large surfaces and dielectric mismatch between the core and the surrounding matrix increase dramatically the nonlinear optical coefficients.

In this talk I will review our recent work on Si-Nc and their applications:

- The growth and physics of Si-nc [Silicon Nanocrystals; Fundamentals, Synthesis and Applications (Wiley-VCH 2010)]

- The development of high efficiency solar cells with Si-nc downshifting layer [Solar Energy Materials and Solar Cells 132, 267 (2015)]

-The development of bipolar light emitting diodes [Journal of Applied Physics 111, 063102 (2012) Applied Physics Letters (2015);]

- The use of silicon nanocrystals light emitting diode as entropy source for quantum random number generation, the key device for cryptography [Journal of Ligthwave Technology (2015)];

- The use of silicon nanocrystals as nonlinear material in bistable optical cavities, in waveguide optical mode monitors which are based on two photon excited luminescence detection, in wavelength shifter by sum-frequency generation [Optics Letters 38, 3562 (2013); Applied Physics Letters 106, 071109 (2015).].

29 de junio| **Exploring Artificial Ferroic Systems with Synchrotron X-rays**
Profesora Laura Heyderman
ETH Zürich y Paul Scherrer Institute, Suiza

In artificial ferroic systems [1], novel functionality can be engineered through the combination of structured ferroic materials and tailored interactions between the different components. With both careful design of the structures and appropriate selection of materials, artificial ferroic systems offer prospects for the discovery of new physics, both static and dynamic, as well as the development of novel devices.

I will present our work employing synchrotron x-rays to probe two classes of artificial ferroic systems. I will begin with hybrid mesoscopic structures incorporating two different ferromagnetic layers, which display magnetization dynamics that results from the mutual imprint of the magnetic domain configurations [2]. I will also discuss work on multiferroic composites, which have the potential to provide control of the state of the magnetic components with an electric field [3, 4]. Finally, I will describe our progress in a second class of artificial ferroic systems, artificial spin ice, which consists of arrays of dipolar-coupled nanomagnets arranged in frustrated geometries. This includes the observation of emergent magnetic monopoles in magnetic-field-driven systems [5] and more recent work on cooperative moment reorientations in thermally active systems [6, 7, 8].

L.J. Heyderman and R.L. Stamps, J. Phys.: Condens. Matter 25, 363201 (2013)
G. Heldt et al. Appl. Phys. Lett. 104, 182401 (2014); P. Wohlhüter et al. Submitted (2014)
R.V. Chopdekar et al. Phys. Rev. B 86, 014408 (2012)
M. Buzzi et al. Phys. Rev. Lett. 111, 027204 (2013)
E. Mengotti et al. Nature Physics 7, 68 (2011)
A. Farhan et al. Nature Physics 9, 375 (2013)
A. Farhan et al. Phys. Rev. Lett. 111, 057204 (2013)
V. Kapaklis et al. Nature Nanotechnology 9, 514 (2014)

15 de julio| **Plasma polymerization (diagnostic + growth mechanism)**
Prof. Dr. Rony Snyders
Head of the research laboratory ChIPS (Chimie des Interactions Plasma-Surface)

In the framework of our activities in the field of plasma polymerization, we are interested in contributing towards a better understanding of the growth mechanism of the plasma polymer films which is of tremendous importance for a better control of the material properties. To reach this goal, our systematic strategy is based on a multidisciplinary approach combining plasma diagnostic and thin film characterization. The latter is mainly achieved through XPS measurements most of the time combined with chemical derivatization methods as well as ToF-SIMS measurements. On the other hand, the plasma chemistry is investigated by using Mass Spectrometry and FTIR spectroscopy. For both methods, the data are evaluated using an original approach using DFT calculations. We have recently shown that this approach allows to significantly improve the understanding of the different reactions pathways encountered by the precursor. In the future, based on our recent achievements in clarifying the growth mechanism of several families of plasma polymers, our objective is to go one step further by investigating the chemical reactivity of film-forming species towards the growing film. In addition, in order to get a more complete picture of the mechanisms, strong efforts will be devoted to characterize the electrons as well as the ions by dedicated diagnostic methods.

24 de septiembre| **Sunlight, free electrons and molecular bonds: Design of catalysts and reactive interfaces for energy storage and conversion**
Dr. Peter Strasser
The Electrochemical Energy, Catalysis, and Materials Science Laboratory. Departament of Chemistry, Chemical Engineering Division. Technical University Berlin. Alemania

Catalytic materials are key components of (photo)electrochemical devices and technologies for storing and converting renewable electricity. Their successful development and optimization requires insight into the relation between the atomic-scale chemical structure of the electrified interface and its catalytic activity, selectivity, and stability.
In this talk, we will highlight some of our recent work on the fundamental understanding of electrocatalytic nanomaterials and their liquid-solid interface at the atomic-scale. We will outline the preparation, characterization, and catalytic performance of a number of different nanomaterials and solar fuel reactions and discuss relations between structure, activity, selectivity, and stability. Topics will touch upon the acid and alkaline oxygen electrode(1-17) and the electroreduction of CO2(18-21).

1. P. Strasser, Science, 2015, 349, 379-380.
2. B. H. Han, C. E. Carlton, A. Kongkanand, R. S. Kukreja, B. R. Theobald, L. Gan, R. O'Malley, P. Strasser, F. T. Wagner and Y. Shao-Horn, Energy Environ. Sci., 2015, 8, 258-266.
3. N. R. Sahraie, J. P. Paraknowitsch, C. Göbel, A. Thomas and P. Strasser, J. Am. Chem. Soc., 2014, 136, 14486-14497.
4. L. Gan, C. Cui, M. Heggen, F. Dionigi, S. Rudi and P. Strasser, Science, 2014, 346, 1502-1506.
5. L. Gan, M. Heggen, R. O'Malley, B. Theobald and P. Strasser, Nano Lett., 2013, 13, 1131-1138.
6. C. Cui, L. Gan, M. Heggen, S. Rudi and P. Strasser, Nature Mat., 2013, 12, 765.
7. P. Strasser, S. Koh, T. Anniyev, J. Greeley, K. More, C. F. Yu, Z. C. Liu, S. Kaya, D. Nordlund, H. Ogasawara, M. F. Toney and A. Nilsson, Nature Chem., 2010, 2, 454-460.
8. P. Strasser, Rev. Chem. Eng., 2009, 25, 255-295.
9. R. Srivastava, P. Mani, N. Hahn and P. Strasser, Angewandte Chemie International Edition, 2007, 46, 8988-8991.
10. S. Koh and P. Strasser, J. Am. Chem. Soc., 2007, 129, 12624-12625.
11. H.-S. Oh, H. N. Nong and P. Strasser, Adv. Funct. Mater., 2015, 25, 1074-1081.
12. H. S. Oh, H. N. Nong, T. Reier, M. Gliech and P. Strasser, Chem. Sci., 2015, 6, 3321-3328.
13. H. N. Nong, H. S. Oh, T. Reier, E. Willinger, M. G. Willinger, V. Petkov, D. Teschner and P. Strasser, Angew. Chem., 2015, 54, 2975-2979.
14. T. Reier, D. Teschner, T. Lunkenbein, A. Bergmann, S. Selve, R. Kraehnert, R. Schlögl and P. Strasser, J. Electrochem. Soc., 2014, 161, F876-F882.

15. H. N. Nong, L. Gan, E. Willinger, D. Teschner and P. Strasser, Chem. Sci., 2014, 5, 2955-2963.
16. A. Bergmann, I. Zaharieva, H. Dau and P. Strasser, Energy Environ. Sci., 2013, 6, 2745-2755.
17. H. Dau, C. Limberg, T. Reier, M. Risch, S. Roggan and P. Strasser, Chemcatchem, 2010, 2, 724-761.
18. A. S. Varela, N. Ranjbar, J. Steinberg, W. Ju, H.-S. Oh and P. Strasser, Angewandte Chemie - International Edition, 2015, online DOI: 10.1002/anie.201502099.
19. R. Reske, H. Mistry, F. Behafarid, B. Roldan Cuenya and P. Strasser, J. Am. Chem. Soc., 2014, 136, 6978-6986.
20. H. Mistry, R. Reske, Z. Zeng, Z.-J. Zhao, J. Greeley, P. Strasser and B. Roldan Cuenya, J. Am. Chem. Soc., 2014, 136, 16473-16476.
21. Ana Sofia Varela, Matthias Kroschel, Tobias Reier and P. Strasser, Catal. Today, 2015, doi:10.1016/j.cattod.2015.1006.1009.

12 de noviembre| **Design of Hierarchically Organized Zeolite Catalysts**
Prof. Javier Pérez-Ramírez
Institute of Chemical and Bioengineering ETH Zurich, Switzerland

This lecture overviews recent progress towards the design of hierarchically-structured zeolites for application in catalysis. I will illustrate the benefits attained due to the improved access and molecular transport in zeolite crystals with auxiliary pore networks, emphasizing recent advances that contribute to the integral design of these materials for established and new catalytic processes. This includes preparative aspects by strategic post-synthetic modifications, the development of advanced characterization tools to assess descriptors, and the transition from laboratory practices with powders to the multi-ton scale up and structuring of the hierarchical zeolites into technical form. Current needs and future directions will be discussed.

1. J. Pérez-Ramírez, Nature Chem. 2012, 4, 250.
2. S. Mitchell, N.-L. Michels, K. Kunze, J. Pérez-Ramírez, Nat. Chem. 2012, 4, 825.
3. S. Mitchell, N.-L. Michels, J. Pérez-Ramírez, Chem. Soc. Rev. 2013, 42, 6094.
4. M. Milina, S. Mitchell, N.-L. Michels, J. Kenvin, J. Pérez-Ramírez, J. Catal. 2013, 308, 398.
5. L. Gueudré, M. Milina, S. Mitchell, J. Pérez-Ramírez, Adv. Funct. Mater. 2014, 24, 209.
6. T. C. Keller, S. Isabettini, D. Verboekend, E. G. Rodrigues, J. Pérez-Ramírez, Chem. Sci. 2014, 5, 677.
7. M. Milina, S. Mitchell, P. Crivelli, D. Cooke, J. Pérez-Ramírez, Nat. Commun. 2014, 5:3922, DOI: 10.1038/ncomms4922
8. N.-L. Michels, S. Mitchell, J. Pérez-Ramírez, ACS Catal. 2014, 4, 2409.
9. M. Milina, S. Mitchell, P. Crivelli, D. Cooke, J. Pérez-Ramírez, Angew. Chem. Int. Ed. 2015, 54, 1591.
10. M. Morales, P.Y. Dapsens, I. Giovinazzo, J. Witte, C. Mondelli, S. Papadokonstantakis, K. Hungerbühler, J. Pérez-Ramírez, Energy Environ. Sci. 2015, 8, 558

Otras Actividades / Other Activities

Feria de la Ciencia / Fair of Science

La Feria de la Ciencia (7 al 9 de mayo de 2015, Palacio de Congreso y Exposiciones de Sevilla, FIBES) constituye un punto de encuentro donde se desarrollaron actividades de divulgación de la Ciencia y la Tecnología, realizando demostraciones y experimentos para facilitar la comprensión de contenidos científicos. El Instituto de Ciencia de Materiales de Sevilla presentó la actividad: "Tecnología del Plasma: Aplicaciones Funcionales".
Coordinadora: T Cristina Rojas Ruiz
Participaron un total de 19 monitores

The Fair of Science (7 to 9 May 2015, in Seville) constituted a meeting point where many activities for spreading of science and technology were carried out. Demonstrations and experiments where presented to facilitate the understanding of scientific aspects. The Materials Science Institute of Seville presented the activity: "Tecnología del Plasma: Aplicaciones Funcionales"
Coordinadora: T Cristina Rojas Ruiz
Participaron un total de 19 monitores

Semana de la Ciencia y la Tecnología / Science and Technology Week

Jornadas de puertas abiertas. La Semana de la Ciencia y la Tecnología está dirigida a todos los públicos. Este evento de carácter europeo está diseñado para demostrar cómo la ciencia y la tecnología nos afectan y cómo éstas pueden ser utilizadas para mejorar nuestras vidas y el mundo que nos rodea.

Coordinación: Dra. T. Cristina Rojas Ruiz

Café con Ciencia

El café con ciencia es una actividad que acerca de forma original y atractiva la ciencia y sus protagonistas. Esta actividad de divulgación genera un punto de contacto entre profesionales de la ciencia y alumnos de secundaria, promoviendo la reflexión sobre diversos asuntos en un entorno cercano y participativo.

En esta edición se compartirán desayuno con estudiantes de bachillerato para dialogar sobre temas concretos de sus estudios y sobre sus respectivas trayectorias profesionales. Esta actividad cumple el triple objetivo de comunicar la ciencia a través de sus propios protagonistas, promover la cultura científica y fomentar vocaciones investigadoras.

Las mesas de encuentro tienen una hora de duración, y se desarrollan con grupos reducidos de quince alumnos.

Mesa: Dr. Angel Justo Erbez. Coloquio con estudiantes. Tema: “La importancia de la investigación científica para el conocimiento y la conservación del Patrimonio Histórico”

IES San Alberto Magno (14 alumnos)

Visitas guiadas: Dr. A. Ruiz Conde

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 25 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

Asunción Fernández Camacho. Carpa del “Corner Europeo”. La revolución nanotecnológica a través del microscopio electrónico

Mauricio Calvo Roggiani. Carpa 3 /CSIC. ¿Cómo nos protegeremos del sol en un futuro?

Instituto de Ciencia de Materiales de Sevilla
CSIC/US
c/ Américo Vespucio, nº 49
41092 Isla de la Cartuja. Sevilla
http://www.icms.us-csic.es
buzon@icmse.csic.es