ICMS

Instituto de Ciencia de Materiales de Sevilla

Memoria de Actividades
Annual Report

Consejo Superior de Investigaciones Científicas

Universidad de Sevilla

Junta de Andalucía

2020

Consejo Superior de Investigaciones Científicas
Universidad de Sevilla
Junta de Andalucía

INSTITUTO DE CIENCIA DE MATERIALES DE SEVILLA

Memoria de Actividades
Annual Report
2020

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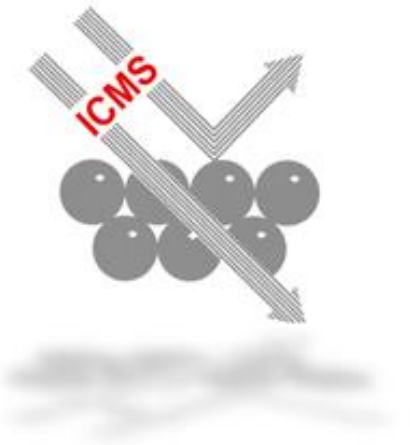

EDITA
Instituto de Ciencia de Materiales de Sevilla (ICMS)
Centro Mixto entre el Consejo Superior de Investigaciones
Científicas y la Universidad de Sevilla
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José Carlos Rivero Cabello

COMISIÓN MEMORIA-ICMS
Gerardo Colón Ibáñez - Francisco José Gotor Martínez – Pedro José Sánchez Soto

COORDINADORA DE DIVULGACIÓN CIENTÍFICA ICMS
T. Cristina Rojas Ruiz

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El ICMS en 2020
ICMS in 2020

Presentación Presentation

A través de esta Memoria 2020, el Instituto de Ciencia de Materiales de Sevilla (ICMS) pretende comunicar lo mejor de las actividades científicas realizadas a lo largo del pasado año. El ICMS es un centro mixto del Consejo Superior de Investigaciones Científicas (CSIC) y la Universidad de Sevilla (US), cofinanciado por la Junta de Andalucía. Fue creado en 1986 y está integrado por personal científico de la Universidad de Sevilla y el CSIC. A finales de 2020, contaba con 113 empleados, de los cuales 53 eran científicos permanentes.

De acuerdo con la estructura establecida en el vigente Plan Estratégico 2018-2021, nuestro centro se organiza en torno a 5 departamentos, cuyos miembros abordan temáticas tanto fundamentales como aplicadas. Así, nuestras investigaciones abordan problemas candentes de la Química y Física del Estado Sólido, la Catálisis Heterogénea, la Cerámica, la Óptica, la Ciencia de Superficies, las Energías Renovables y el Medio Ambiente, etc. Entre ellas, podemos resaltar el aprovechamiento de las energías solar y de biomasa, la generación de H_2 y otros combustibles limpios y de origen renovable, el aprovechamiento y conversión química del CO_2, la depuración de efluentes químicos, el secuestro de residuos radioactivos, la mejora de la selectividad y rendimiento de reacciones químicas básicas, el aumento de la eficiencia de los dispositivos generadores de energía renovable (celdas fotovoltaicas, electrodos, generadores piezoeléctricos, celdas de combustible o baterías) y de los emisores de luz (LEDs), el desarrollo y la mejora de sensores químicos y agentes de contraste radiológico, de materiales biocompatibles para implantes quirúrgicos, de pigmentos cerámicos, de recubrimientos hidrofóbicos o hidrofílicos, recubrimientos hielofóbicos,… y un largo etcétera.

Nuestros principales empeños para los años venideros son realizar investigaciones que se sitúen en la vanguardia de la Ciencia e Ingeniería de Materiales y que éstas sirvan de apoyo al desarrollo en Andalucía de un nuevo sistema económico, en el que las bases científica y tecnológica pasen a ser pilares fundamentales.

Through this Activity Report 2020, the Institute of Materials Science of Seville (ICMS) is communicating the best of its scientific activities carried out for the last year. The ICMS is a joint centre of the Spanish Research Council (CSIC) and the University of Seville (US), also funded by the Junta de Andalucia. It was founded in 1986, and integrated by scientific staff of the University of Seville and the CSIC. At the end of last year, it includes 113 people, 53 of them as permanent scientific staff.

The current Strategic Plan 2018-2021 organizes our Centre in 5 departments, which personnel address both fundamental and applied research. Thus, our researchers address hot topics related with disciplines of Solid State Chemistry and Physics, Heterogeneous Catalysis, Ceramics, Optics, Surface Science, Renewable Energy and Environment, etc., Among them, we could mention the exploitation of solar energy and biomass, the generation of H_2 and other clean and renewable fuels, the exploitation and chemical conversion of CO_2, the removal of pollutants, the storage of radioactive wastes, the improvement of the selectivity and yield of basic chemical reactions, the efficiency of renewable energy generators (photovoltaic cells, electrodes, piezoelectric devices, fuel cells and batteries) and light emitting devices (LEDs), the development and improvement of chemical sensors and radiological contrast agents, the production of biocompatible scaffolds for surgical implants, the production of ceramic pigments, many different coatings and thin films, … and a long et cetera.

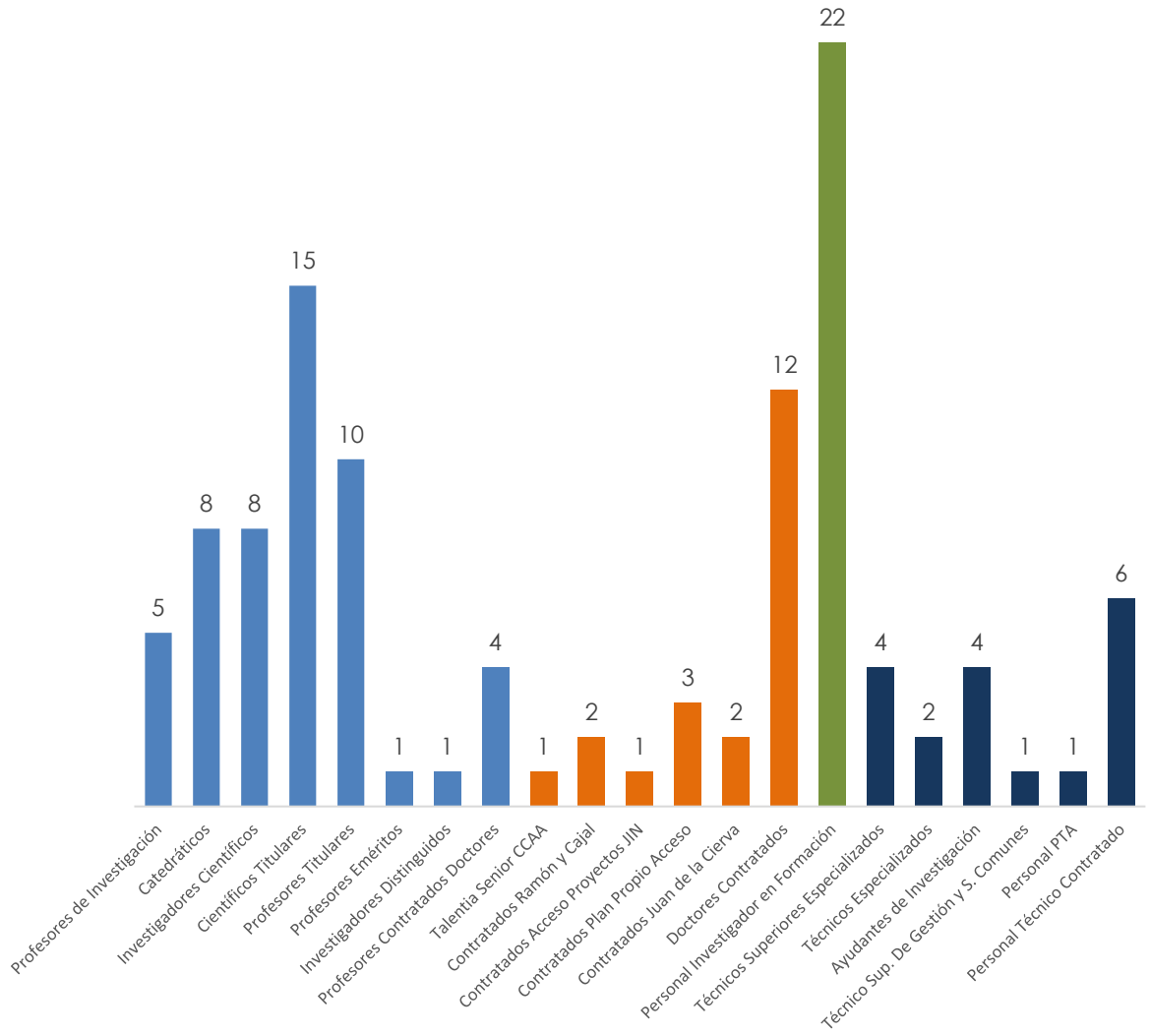
Our main efforts for near future are to cover the most modern and innovative aspects of the current Material Science and Material Engineering, as well as to support in the region of Andalusia, the development of a new economic system, in which scientific and technological basis should become fundamental pillars.

Dr. Juan Pedro Espinós Manzorro
Director del Instituto de Ciencia de Materiales de Sevilla

Datos Estadísticos del ICMS
Statistical Data of ICMS

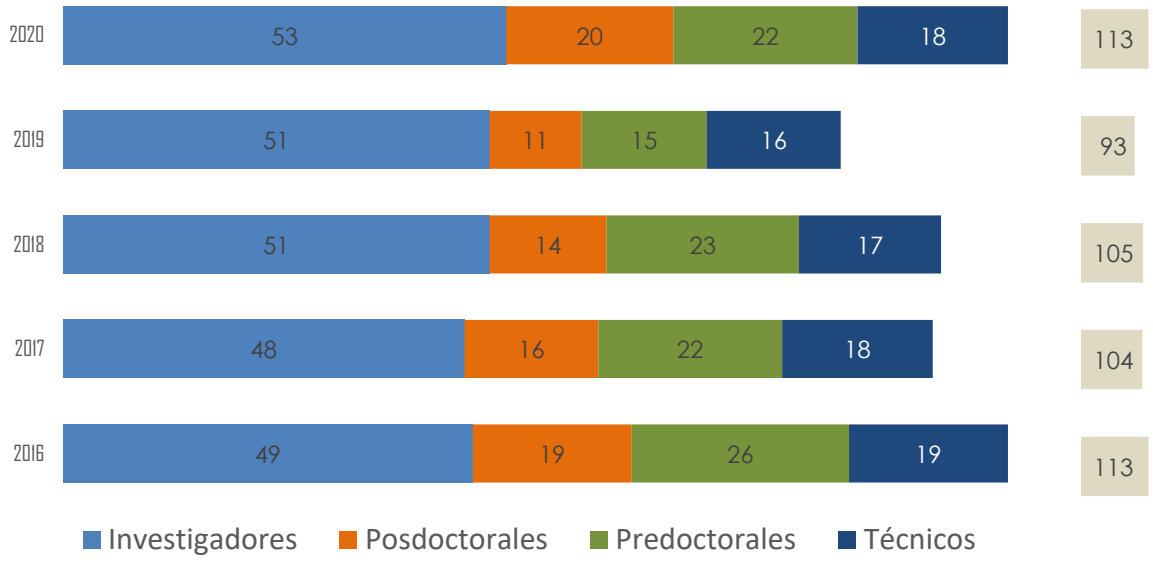
RECURSOS HUMANOS / HUMAN RESOURCES

Distribución del personal por categorías – 2020*
Distribution by professional category – 2020

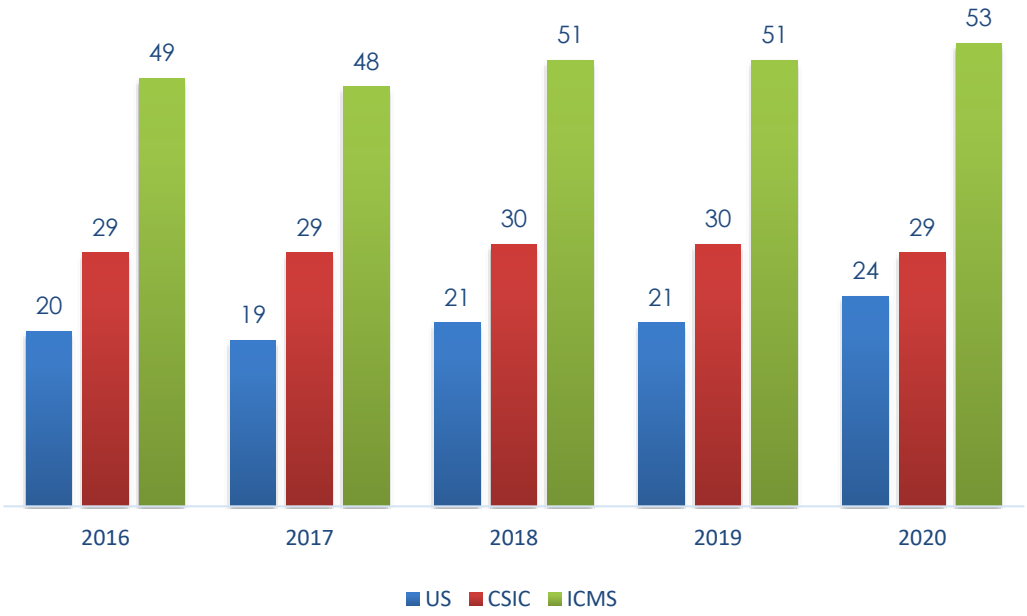


* Datos a 31 de Diciembre de 2020

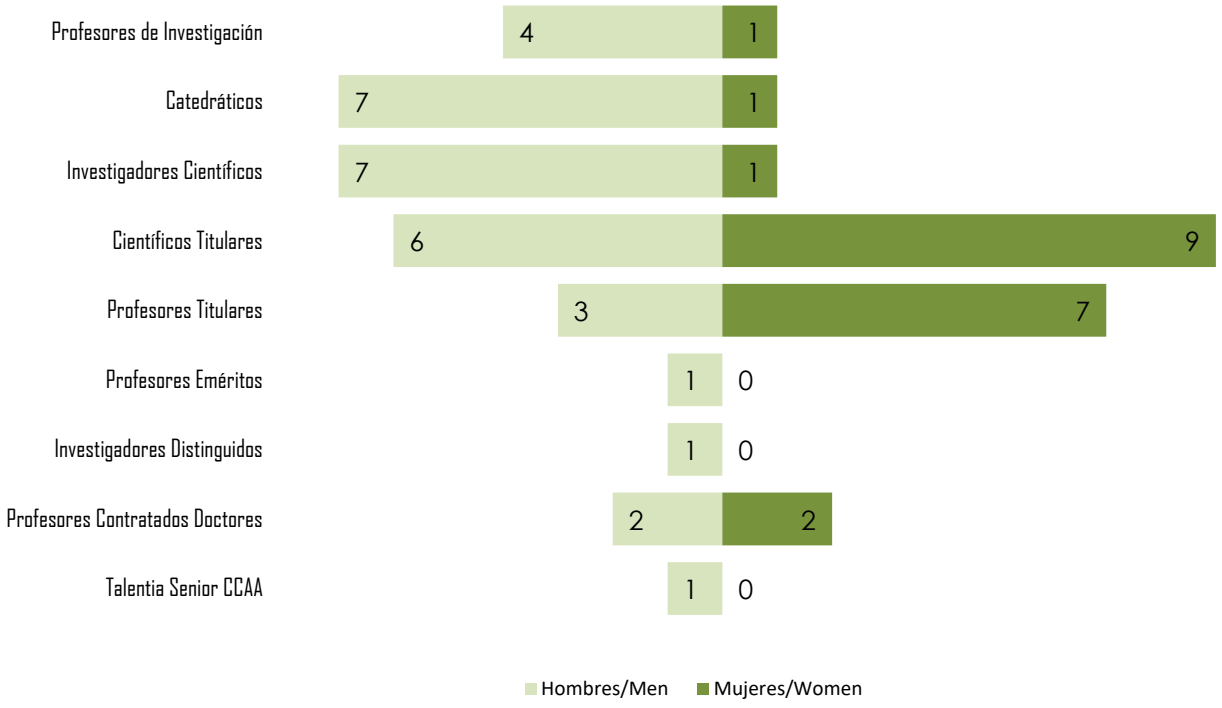
Evolución 2016-2020 del personal
Evolution of Staff

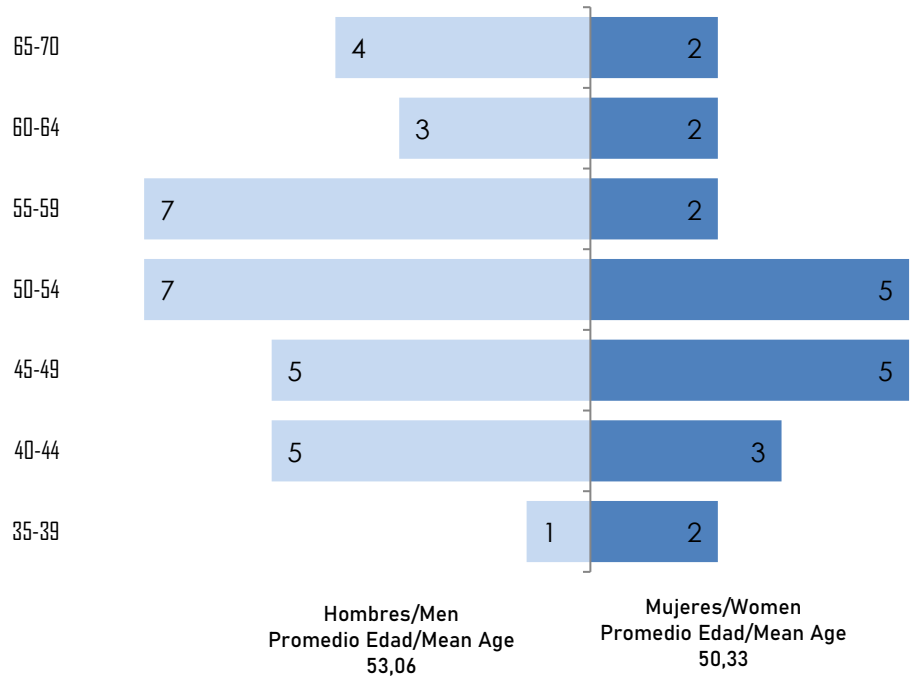


Evolución 2016-2020 del Personal Investigador
Evolution of Research Staff



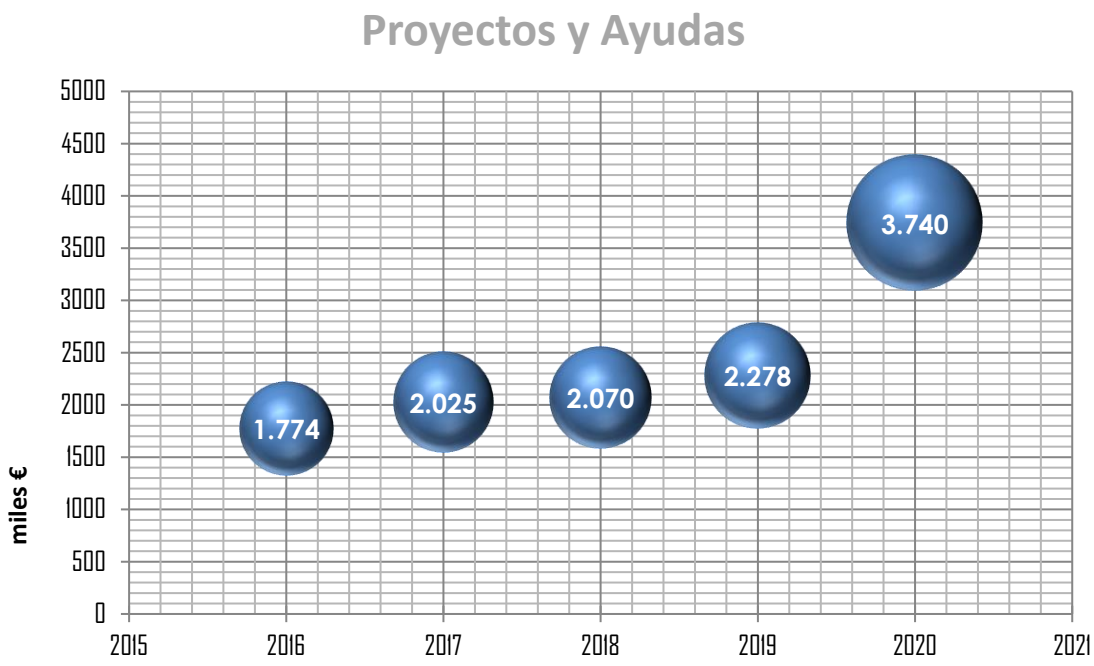
Distribución del personal Investigador (sexo y edad)
Distribution by Scientific staff (gender and age)





FINANCIACIÓN / FUNDING

Evolución 2016-2020 de la Financiación conseguida por año (miles€)(PCO)
Evolution of the funding of the ICMS



PRODUCCIÓN CIENTÍFICA / SCIENTIFIC PRODUCTION

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

REVISTA JOURNAL	ARTÍCULOS PAPERS	FACTOR DE IMPACTO IMPACT FACTOR (*)	Mejor PCT
ADVANCED ENERGY MATERIALS	1	29,368	Q1
APPLIED CATALYSIS B-ENVIRONMENTAL	4	19,500	Q1
ACS CATALYSIS	1	13,084	Q1
ADVANCES IN COLLOID AND INTERFACE SCIENCE	1	12,980	Q1
JOURNAL OF ADVANCED RESEARCH	1	10,479	Q1
ADVANCED OPTICAL MATERIALS	2	9,926	Q1
CARBON	1	9,594	Q1
JOURNAL OF CLEANER PRODUCTION	1	9,297	Q1
ACS APPLIED MATERIALS & INTERFACES	1	9,229	Q1
MATERIALS TODAY CHEMISTRY	1	8,301	Q1
ACS SUSTAINABLE CHEMISTRY & ENGINEERING	1	8,198	Q1
JOURNAL OF COLLOID AND INTERFACE SCIENCE	3	8,130	Q1
RENEWABLE ENERGY	1	8,001	Q1
MATERIALS & DESIGN	1	7,991	Q1
NANOSCALE	1	7,790	Q1
SENSORS AND ACTUATORS B-CHEMICAL	1	7,460	Q1
JOURNAL OF MATERIALS CHEMISTRY C	2	7,393	Q1
FUEL PROCESSING TECHNOLOGY	1	7,303	Q1
SOLAR ENERGY MATERIALS AND SOLAR CELLS	1	7,267	Q1
ENERGY	1	7,147	Q1
CHEMOSPHERE	1	7,086	Q1
JOURNAL OF PHYSICAL CHEMISTRY LETTERS	1	6,475	Q1
CURRENT OPINION IN GREEN AND SUSTAINABLE CHEMISTRY	2	6,457	Q1
CATALYSIS TODAY	2	6,291	Q1
INTERNATIONAL JOURNAL OF HYDROGEN ENERGY	2	5,816	Q2
CHEMCATCHEM	1	5,686	Q2
SCRIPTA MATERIALIA	1	5,611	Q1

REVISTA JOURNAL	ARTÍCULOS PAPERS	FACTOR DE IMPACTO IMPACT FACTOR (*)	Mejor PCT
JOURNAL OF ANALYTICAL AND APPLIED PYROLYSIS	1	5,541	Q1
APPLIED CLAY SCIENCE	1	5,467	Q1
MICROPOROUS AND MESOPOROUS MATERIALS	2	5,455	Q1
JOURNAL OF BUILDING ENGINEERING	1	5,318	Q1
JOURNAL OF ALLOYS AND COMPOUNDS	1	5,316	Q1
JOURNAL OF THE EUROPEAN CERAMIC SOCIETY	1	5,302	Q1
FRONTIERS IN CHEMISTRY	2	5,221	Q2
ARABIAN JOURNAL OF CHEMISTRY	1	5,165	Q2
ACS APPLIED NANO MATERIALS	1	5,097	Q2
NANOMATERIALS	2	5,076	Q1
MOLECULAR CATALYSIS	1	5,062	Q2
DYES AND PIGMENTS	1	4,889	Q1
MATERIALS RESEARCH BULLETIN	1	4,641	Q2
JOURNAL OF THERMAL ANALYSIS AND CALORIMETRY	2	4,626	Q1
CERAMICS INTERNATIONAL	3	4,527	Q1
POLYMERS	2	4,329	Q1
JOURNAL OF PHOTOCHEMISTRY AND PHOTOBIOLOGY A-CHEMISTRY	3	4,291	Q2
JOURNAL OF MATERIALS SCIENCE	2	4,220	Q2
SURFACE & COATINGS TECHNOLOGY	3	4,158	Q1
CATALYSTS	2	4,146	Q2
JOURNAL OF PHYSICAL CHEMISTRY C	4	4,146	Q2
MATERIALS SCIENCE IN SEMICONDUCTOR PROCESSING	1	3,927	Q1
PLASMA PROCESSES AND POLYMERS	1	3,872	Q1
CHEMPHOTOCHEM	1	3,849	Q2
JOURNAL OF THE AMERICAN CERAMIC SOCIETY	1	3,784	Q1
INDUSTRIAL & ENGINEERING CHEMISTRY RESEARCH	1	3,720	Q2
PHYSICAL CHEMISTRY CHEMICAL PHYSICS	2	3,676	Q1
MATERIALS	3	3,623	Q1
JOURNAL OF LUMINESCENCE	1	3,599	Q1
JOURNAL OF NON-CRYSTALLINE SOLIDS	1	3,531	Q1
MATERIALS LETTERS	3	3,423	Q2
MATERIALS TODAY COMMUNICATIONS	1	3,383	Q2

REVISTA JOURNAL	ARTÍCULOS PAPERS	FACTOR DE IMPACTO IMPACT FACTOR (*)	Mejor PCT
JOURNAL OF BIOMEDICAL MATERIALS RESEARCH PART B-APPLIED BIOMATERIALS	1	3,368	Q2
NUCLEAR FUSION	1	3,179	Q1
PHYSICAL REVIEW A	1	3,140	Q2
THERMOCHIMICA ACTA	1	3,115	Q2
TOPICS IN CATALYSIS	1	2,910	Q2
JOURNAL OF APPLIED PHYSICS	1	2,546	Q2
METALS	2	2,351	Q2
APPLIED OPTICS	1	1,980	Q3
INTERNATIONAL JOURNAL OF APPLIED CERAMIC TECHNOLOGY	1	1,968	Q2
JOURNAL OF MOLECULAR MODELING	1	1,810	Q3
MATERIALS RESEARCH EXPRESS	1	1,620	Q4
Total	**102**		

(*) Factor de Impacto correspondiente al año 2020
Journal Citation Reports of 2020

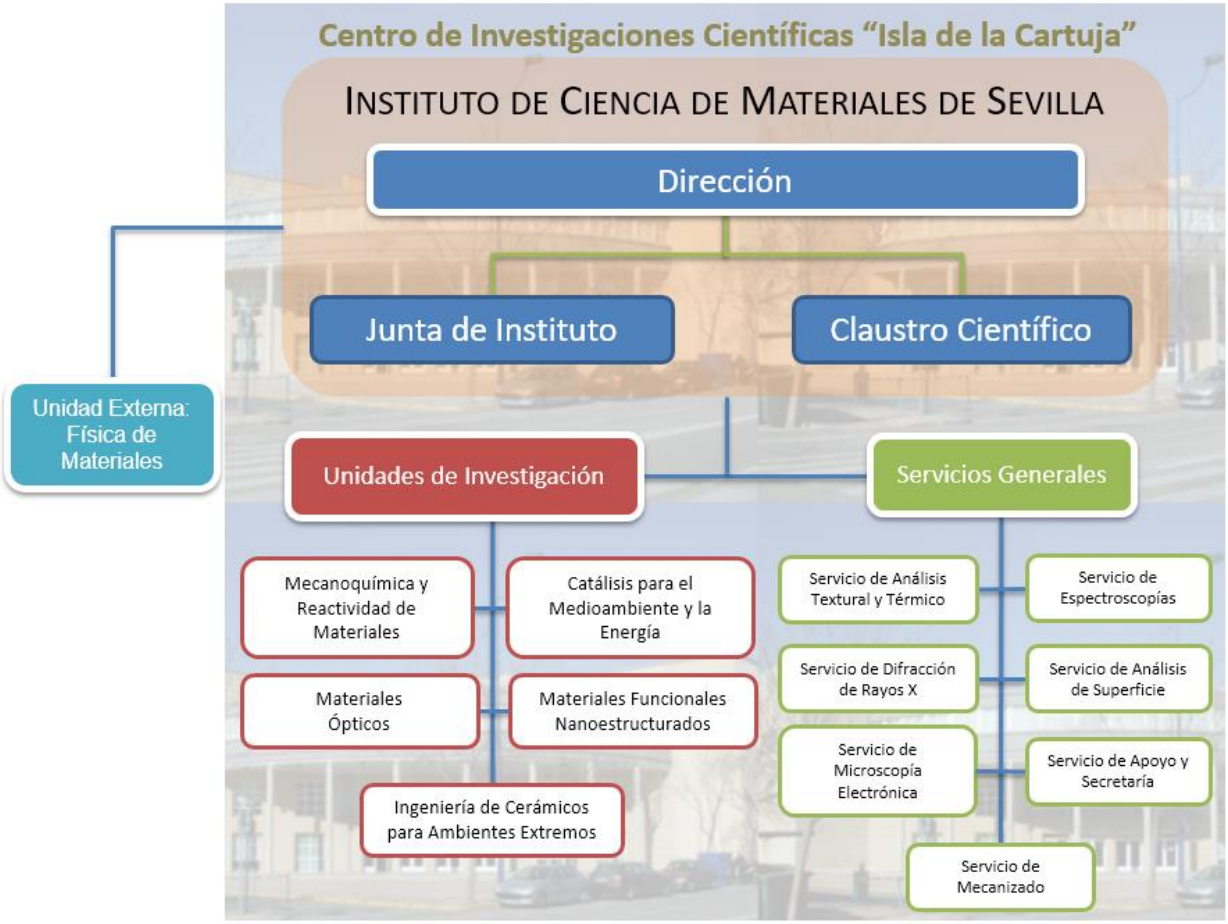
Composición y Estructura
Structure and Organisation

EL INSTITUTO / THE INSTITUTE

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

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

ORGANIGRAMA / ORGANIZATION CHART



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Gdo. Ahmed Taibi

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Dr. Gabriel Lozano Barbero
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CATÁLISIS PARA EL MEDIOAMBIENTE Y LA ENERGÍA
CATALYSIS FOR ENVIRONMENT AND ENERGY

GRUPOS DE INVESTIGACIÓN

Fotocatálisis Heterogénea: Aplicaciones
Heterogeneus Photocatalysis: Applications [642005]
http://materphotocat.ciccartuja.es

Materiales y Procesos Catalíticos de Interés Ambiental y Energético
Materials and catalytic processes for environment and energy [642004]
http://matproner.icms.us-csic.es

Química de Superficies y Catálisis
Surface Chemistry and Catalysis [642006]
http://surfcat.icms.us-csic.es

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PROYECTOS DE INVESTIGACIÓN / RESEARCH PROJECTS

Procesos Power-to-X para la Valorización de CO_2 en Reactores Catalíticos Estructurados (CO_2-Ptx)

Power-to-X processes for CO_2 valorization in structured catalytic reactors (CO_2-PTX)

Código/Code:	RTI2018-096294-B-C33
Periodo/Period:	01-01-2019 / 31-12-2021
Organismo Financiador/Financial source:	Ministerio de Ciencia, Innovación y Universidades
Importe total/Total amount:	260.150 €
Investigador responsable/Research head:	José Antonio Odriozola Gordón y Francisca Romero Sarria
Componentes/Research group:	Luis F. Bobadilla Baladrón, María Isabel Domínguez Leal, Anna Dimitrova Penkova, Lola de las Aguas Azancot Luque, Marta Romero Espinosa, Juan Carlos Navarro De Miguel

RESUMEN / ABSTRACT

La tecnología Power-to-X (PTX) tiene como objetivo el almacenamiento de energía (preferentemente renovable) en productos químicos. Dichos productos pueden usarse luego como combustibles o como moléculas plataforma para otras síntesis químicas. Por tanto, esta tecnología juega un papel fundamental incrementando la fracción renovable del mix energético en línea con los objetivos de la UE para la reducción de emisiones de gases con efecto invernadero.

La producción de H_2 por electrólisis de agua para PTX es una tecnología madura disponible comercialmente que puede ser usada durante los periodos valle de consumo de energía renovables.

Por otro lado, el CO_2 es una fuente de carbono desaprovechada por lo que el uso combinado de H_2 renovable y CO_2 añade un importante plus al proceso PTX ya que el CO_2 asociado a las emisiones de gases de efecto invernadero es reintegrado contribuyendo a la economía circular y la descarbonización. Esta es la idea central que guía la presente propuesta. En particular, se trata de llevar a cabo las siguientes reacciones: hidrogenación de CO_2 a metano (también llamada metanación de CO_2 o reacción de Sabatier), la reacción reversa Water-Gas-Shift (activación del CO_2 y ajuste de la relación H_2/CO), síntesis de biocombustibles (dimetil éter y SFT) y producción de ácido acético. Estas reacciones ofrecen notables retos químico-ingenieriles en aspectos como: i) desarrollo de catalizadores multifuncionales adecuados; ii) gestión térmica de reacciones fuertemente exotérmicas; iii) control de la selectividad en reacciones múltiples en serie por acción conjunta de la temperatura, el tiempo de residencia, la formulación del catalizador y el diseño del reactor. El conocimiento adquirido por el consorcio en los proyectos previos (MAT2006-12386, ENE2009-14522, ENE2012-37431 y ENE2015-66975)

nos permite proponer de una manera sólida y fundamentada el uso de catalizadores y reactores estructurados para superar estos retos.

Por tanto, el objetivo fundamental de esta propuesta es el estudio de sistemas catalíticos estructurados para reacciones relevantes del proceso Power-To-X con CO_2 (CO_2-PTX). Por otro lado, esperamos que la intensificación que aportan los sistemas estructurados sobre metales y los patrones de flujo desarrollados en sistemas como espumas de poro abierto jueguen papeles determinantes en el control de la temperatura y la selectividad de la reacción. En este sentido se estudiarán diferentes arquitecturas de sustrato junto a las variables principales como la densidad de celda o poro, el espesor de película catalítica o la aleación metálica del sustrato. Finalmente, para aproximarnos a la aplicación industrial de estos sistemas CO_2-PtX se considerará la valorización de CO_2 presente en corrientes diluidas como los gases de combustión. Esto supone nuevos retos debido a la baja concentración de CO_2, altos caudales volumétricos y efectos negativos de otros componentes (H_2O, SOx, etc.) en la actividad y estabilidad de los catalizadores. Se investigarán nuevas formulaciones de catalizadores junto con estrategias avanzadas de adsorción-desorción-reacción de CO_2 sobre los sustratos estructurados estudiados.

Globalmente, el proyecto se estructurará en forma matricial con tareas transversales de cada grupo basadas en sus líneas de especialización (modelado, estructuración y caracterización avanzada) junto a reacciones concretas de cada laboratorio que conformarán las tareas longitudinales del proyecto.

The main idea underlying the term "Power-to-X" is the storage of energy (preferably renewable) in the form of chemical products.

Thereafter, these products may be employed in energy-related applications or as platform chemicals. As a result, the Power-to-X (PTX) processes play a key role in increasing the penetration rate of renewables in the energy mix in line with European Unions long-term objective of reducing greenhouse gas (GHG) emissions by 80-95 % by 2050 when compared to 1990 levels. Production of hydrogen by water electrolysis is a mature and commercially available technology that can be used during periods of low demand for renewable energy.

On the other hand, CO_2 is the only abundant carbon source within the EU and the combined use of renewable hydrogen and CO_2 remarkably results in additional benefits in the PTX concept since CO_2-associated GHG emissions is reintegrated in the value chain contributing to circular economy and decarbonization. This main idea drives CO_2-PTX proposal. Specifically, our proposal aims to carry out the following reactions in structured catalytic reactors: CO_2 hydrogenation to methane (also called methanation or Sabatier reaction), the reverse Water-Gas Shift reaction (CO_2 activation and adjustment of the H_2/CO ratio) and the direct synthesis of biofuels (dimethylether and FTS) and acetic acid. This set of reactions provides remarkable challenges in key catalytic engineering aspects such as: i) development of suitable multifunctional structured catalysts; ii) management of the thermal effect of highly exothermic reactions; iii) control of the selectivity of multiple reactions in series through the joint action of the reaction temperature, the residence time and suitable catalyst formulation and reactor configuration. The know-how acquired by the consortium during previous projects (MAT2006-12386, ENE2009- 14522, ENE2012-37431 and ENE2015-66975) allows us to propose the use of structured catalysts and reactors as a very convenient way of addressing that challenges. Heat and mass transfer rates intensification provided by metallic substrates-based structured systems as well as the flow patterns characteristic of open-cell foams are expected to play a determinant role in temperature and selectivity control. In this regard, several catalytic-wall reactor

configurations as parallel-channels monoliths and open-cell foams will be considered, as well as other characteristics that directly affect the transport properties of the structured systems (monolith cell density, pore density of foams, metal alloy used as substrate and catalyst layer thickness).

To be closed to practical applications it will be also considered within the CO_2-PTX project the valorization of CO_2 present in dilute streams, typically flue gases. This entails additional challenges arising from the low concentration of CO_2, high volumetric flow rates and negative effects of other components (H_2O, SO_x, etc.) on the catalytic activity and stability. Improved catalyst formulations as well as sorption-enhanced CO_2 conversion strategies in structured reactors will be investigated.

Overall, the project is organized as a series of transversal tasks for which each group contributes with his main field of specialization and vertical tasks associated to a more intense dedication of each group to one or more of the processes investigated.

Desarrollo de nuevos materiales nanoestructurados para la valorización de metano a hidrógeno y olefinas C2-C4 Development of new nanostructured materials for methane valorization to C2-C4 olefins

Código/Code:	ENE2017-82451-C3-3-R
Periodo/Period:	01-01-2018 / 31-12-2020
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	205.700 €
Investigador responsable/Research head:	Alfonso Caballero Martínez y Gerardo Colón Ibáñez
Componentes/Research group:	Rosa María Pereñíguez Rodríguez, Francisco Jesús Platero Moreno, Ángeles María López Martín, Juan Pedro Holgado Vázquez

RESUMEN / ABSTRACT

El desarrollo de nuevos materiales con propiedades singulares en distintos campos de aplicación se ha convertido en las últimas décadas en una prioridad en multitud de áreas de la ciencia y la tecnología. Entre ellas, además de materiales micro y mesoporosos de composición variable, pueden destacarse los sólidos basados en estructura perovskita ABO_3. La versatilidad que presentan estos últimos mediante la sustitución parcial en las posiciones A y B por distintos metales alcalinos, alcalino-térreos y de transición los convierte en una alternativa interesante, y de hecho tienen aplicaciones en campos relacionados con sus propiedades eléctricas, ópticas, térmicas, catalíticas y como adsorbentes. En el presente proyecto coordinado se plantea la preparación de un conjunto de materiales, entre ellos algunos con estructura perovskita (Fe, Co, Mn, Cu y Bi en posiciones B; Ca, Mg, Ce y La en posiciones A), y estudiar su aplicación en distintos procesos de catálisis heterogénea y de adsorción de contaminantes. Para ello se emplearán una serie de técnicas de preparación recientemente descritas (Cristalización por Microondas,

Proceso de Autocombustión, Mesoestructuración por Nanocasting y Porosidad Jerarquizada) que permiten obtener sistemas de alta superficie específica y nanoestructura controlada. De esta forma, y combinando los metales en posiciones A y B para que actúen tanto como agentes promotores como precursores de aleaciones metálicas en los sistemas reducidos, se obtendrán sistemas con propiedades muy variadas y versátiles. Así, en el subproyecto 1 se estudiarán sus propiedades catalíticas en procesos de enorme interés para la valorización de metano, principal componente del gas natural y una de las fuentes de energía más abundantes en la actualidad. En concreto, y junto con sistemas soportados en materiales mesoporosos y otros, se estudiará en primer lugar la actividad de perovskitas de níquel para la reacción de reformado seco de metano con el fin de obtener gas de síntesis. El objetivo será obtener sistemas activos y sobre estables frente a los fenómenos de desactivación habituales por deposición de coque. En segundo lugar, se estudiarán sistemas basados principalmente en Fe y Co para la reacción de Fisher-Tropsch a olefinas C2-C4, productos de gran interés económico por ser precursores de una gran cantidad de otros productos de alto valor añadido. Por otro lado, los trabajos propuestos en el subproyecto 2 están relacionados con la aplicación de estos sólidos de estructura perovskita para el desarrollo de procesos de eliminación de contaminantes emergentes, un nuevo tipo de desechos que suelen ser resistentes a los procesos de degradación biológico convencionales, constituyendo por tanto un problema medioambiental de primer orden. En concreto, el proyecto pretende desarrollar tratamientos integrados de depuración (adsorción-POA), utilizando perovskitas para la eliminación de contaminantes emergentes (Ibuprofeno, Salicílico, Ciprofloxacina, Cafeína, Gemfibrozil y Benzafibrato), optimizando parámetros como el rendimiento, la reciclabilidad de los catalizadores, la aplicabilidad y la sostenibilidad. De especial relevancia en este tipo de procesos es la utilización de procesos de fotocatálisis heterogénea, por lo que el desarrollo de nuevos óxidos semiconductores como las perovskitas, con características fisicoquímicas y estructurales superiores a las del TiO_2, es un objetivo de primer orden del presente subproyecto.

In the present project the preparation of a set of materials, including some with perovskite structure (Fe, Co, Mn, Cu and Bi in positions B; Ca, Mg, Ce and La in positions A), and the study of its application in different processes of heterogeneous catalysis and adsorption of pollutants has been proposed. For this purpose, a number of recently described preparation techniques will be used to obtain high surface specific and controlled nanostructure systems. In this way, and combining the metals in positions A and B to act both as promoters and precursors of metal alloys in the reduced systems, systems with very varied and versatile properties will be obtained.

Thus, we will study its catalytic properties in processes of great interest for the valorization of methane, the main component of natural gas and one of the most abundant energy sources today. In particular, and together with systems supported on mesoporous materials and others, the activity of nickel perovskites for the dry methane reforming reaction will be studied first in order to obtain synthesis gas. The aim will be to obtain active and above all stable systems in the face of the usual deactivation phenomena by deposition of coke. Secondly, systems based mainly on Fe and Co for the Fisher-Tropsch reaction to C2-C4 olefins will be studied, products of great economic interest as precursors to a large number of other high added value products.

Aprovechamiento de biomasa y producción sostenible de energía mediante (foto)catalizadores y reactores estructurados basados en materiales carbonosos Biomass valorization and sustainable energy production over (photo)catalysts and structured reactors based on carbonaceous materials

Código/Code:	ENE2017-82451-C3-3-R
Periodo/Period:	01-01-2018 / 31-12-2020
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	193.600 €
Investigador responsable/Research head:	Miguel Ángel Centeno Gallego y Svetlana Ivanova
Componentes/Research group:	Leidy Marcela Martínez Tejada, María Isabel Domínguez Leal, Regla Ayala Espinar, Carlos López Cartes

RESUMEN / ABSTRACT

El objetivo principal del presente proyecto coordinado entre la U. de Zaragoza, el ICMS y la U. de Cádiz, es el desarrollo de catalizadores multifuncionales y estructurados basados en materiales catalíticos carbonosos, tanto de carácter biomórfico, como grafénico-grafítico. Estos materiales catalíticos han de ser activos, selectivos y estables en reacciones directamente relacionadas con el aprovechamiento de la biomasa lignocelulósica (producción de 5-HMF, ácido levulínico, FDCA, o γ-valerolactona) y la producción sostenible de energía (producción de H_2), así como la valorización química y fotoquímica de CO_2 (hidrogenación de CO_2, descomposición de biogás, foto-reformado de bio-alcoholes), usando H_2 de origen renovable ("water splitting"). Este proyecto trata de mejorar procesos actualmente implementados que están relacionados con la producción de energía, y otros más novedosos, como el aprovechamiento de la luz solar, que sin lugar a dudas están llamados a tener un papel importante en este campo. De hecho, la utilización de la energía solar haría más viable energéticamente, por ejemplo, la reacción de metanación de CO_2 al usar H_2 de origen (foto)renovable producido por "water splitting". Se busca también la generación de productos de alto valor añadido por procesos de biorefinería, que sustituyan los obtenidos actualmente a partir de fuentes fósiles. Se pretende conseguir un conjunto de sólidos carbonosos con propiedades estructurales (porosidad jerárquica meso/micro), hidrofilicidad-hidrofobicidad, funcionalidades químicas, composición superficial etc. diseñados ad hoc para cada una de las reacciones consideradas por los distintos subproyectos, incluyendo la implementación de procesos en continuo mediante la utilización de reactores estructurados a partir de los catalizadores más eficientes. El desarrollo y utilización de sistemas catalíticos estructurados aumenta la viabilidad e intensificación de los procesos y por tanto la eficiencia energética y medioambiental. La complementariedad de los tres grupos proponentes abre la posibilidad de abordar en un solo proyecto todos estos objetivos, permitiendo aplicar distintas metodologías emergentes para la síntesis de nuevos materiales carbonosos, como son la mineralización biomórfica, la expansión/funcionalización de

compuestos intercalados de grafito, grafitos especiales (e.g. “graphite nanolayers” o “nanoflakes”), uso de plantillas inorgánicas para generación de carbones mesoporosos, su funcionalización avanzada y su aplicación en procesos de alto impacto en el área de la energía, tecnología química y tecnologías ambientales.

The main goal of ENERCARB, project coordinated among the U. of Zaragoza, the ICMS and the U. of Cádiz, is the development of multifunctional and structured catalysts based on carbonaceous catalytic materials of biomorphic and/or graphenic-graphitic character. These materials must be active, selective and stable in catalytic reactions related to i) the production and use of chemicals derived from lignocellulosic biomass, i.e. 5-HMF, levulinic acid, FDCA and g-valerolactone; ii) to sustainable energy vector production (H_2), and iii) to chemical and photochemical utilization of CO_2 (CO_2 hydrogenation), biogas decomposition, photo-reforming of bio-alcohols) using H_2 of renewable origin (“water splitting”). This project tries to improve currently implemented processes for energy production, and to propose other more innovative processes, such as use of sunlight, undoubtedly called to play an important role in this field. In fact, the use of solar energy would make more energy-efficient, the CO_2 methanation reaction by using H_2 of (photo)renewable origin produced by “water splitting”. ENERCARB also intends to generatre high added value products by bio-refinery processes, as alternative to currently obtained chemicals from fossil sources. A set of carbonaceous solids with tunned structural properties (meso/micro hierarchical porosity), hydrophilicity-hydrophobicity, chemical functionalities, surface composition, etc., will be designed ad hoc for each of the reactions considered by the different subprojects. The implementation of continuous processes through the use of structured reactors is the next logical step to increase the efficiency of the the proposed proceses. The development and use of structured catalytic systems increases the viability and intensifies the processes, and therefore leads to higher energy and environmental efficiency. The complimentary nature of the three participating groups opens the possibility of addressing all these objectives in one single project. It will allow the application of different emerging methodologies for the synthesis of new carbonaceous materials, such as biomorphic mineralization, the expansion-functionalization of graphite intercalation compounds, special graphites (e.g. graphite nanolayers or nanoflakes), use of inorganic templates for the generation of mesoporous carbons, and also its advanced functionalization and its application in processes of high impact in the area of energy, chemical and environmental technologies.

Diseño racional de fotocatalizadores altamente eficientes mediante control a nivel atómico Rational design of highly effective photocatalysts with atomic-level control

Código/Code:	RATOCAT PCIN-2017-056
Periodo/Period:	02-10-2017 / 01-10-2020
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad. Unión Europea
Importe total/Total amount:	97.000 €
Investigador responsable/Research head:	Gerardo Colón Ibañez
Componentes/Research group:	Alfonso Caballero Martínez, Ángeles Martín

RESUMEN / ABSTRACT

El uso de la energía solar para la generación de hidrógeno a partir de agua es probablemente uno de los procesos más limpios y sostenibles para la obtención de energía. Sin embargo, los catalizadores que dan mejores rendimientos son demasiado caros para ser económicamente viables. El proyecto RATOCAT tiene como objetivo el desarrollo de materiales fotocatalíticos optimizados. De esta forma las prestaciones fotocatalíticas de sistemas basados en TiO_2 y gC_3N_4 podrían optimizarse mediante el diseño de su superficie con nanoestructuras de composición, nanoarquitectura, tamaño y estado químico altamente controladas. Se empleará para ello estudios de simulación teórica para proponer las nanoestructuras óptimas que serán depositadas de forma controlada y precisa mediante atomic layer deposition (ALD). Los test de actividad fotocatalítica se llevarán a cabo tanto a escala de laboratorio (ICMS) como en planta piloto (Plataforma Solar de Almería).

Using the sun's energy to generate hydrogen from water is probably the cleanest and most sustainable source of fuel that we can envisage. Unfortunately, catalysts that do this are currently too expensive to be commercially viable. The RATOCAT project aims to develop improved photocatalyst materials, along with the processes for their production. The catalytic performance of cheap TiO_2 and C_3N_4 powders will be improved by tailoring their surface with nanostructured oxides as co-catalysts of highly-controlled composition, nanoarchitecture, size and chemical state. First principles simulations will be used to design the optimum nanostructures, which will then be deposited onto powders with the required precision using atomic layer deposition, again supported by simulation. Lab-scale tests of photocatalytic activity will provide feedback for the optimisation of the material and process, before the most promising materials are tested in the field on both pure water and wastewater.

Valorización de CO_2: obtención de hidrocarburos mediante procesos catalíticos de hidrogenación CO_2 valorization: obtaining hydrocarbons through catalytic hydrogenation processes

Código/Code:	US-1263455
Periodo/Period:	01-02-2020 / 31-01-2022
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	80.000 €
Investigador responsable/Research head:	Alfonso Caballero Martínez y Juan Pedro Holgado Vázquez
Componentes/Research group:	Gerardo Colón Ibáñez, Rosa Pereñíguez Rodríguez, Andrew M. Beale (UCL), Ángeles M. López Martín, Francisco Jesús Platero Moreno

RESUMEN / ABSTRACT

En el presente proyecto se llevarán a cabo diversos estudios y desarrollos relacionados con la reducción de CO_2 a productos de alto valor añadido, como metano, olefinas ligeras, gasolinas y otros hidrocarburos funcionalizados, de gran interés económico, energético y medioambiental. El uso de hidrógeno como agente reductor, obtenido éste a su vez de fuentes renovables supone, además de la reducción de las emisiones de gases de efecto invernadero, una vía para el almacenamiento de la energía procedente de fuentes renovables, muchas de ellas de carácter intermitente y por tanto difícilmente acoplable a las necesidades de consumo.

Con todo ello en este proyecto se propone el desarrollo de nuevos sistemas catalíticos heterogéneos basados en Ni, Fe, Co, Ru e In, entre otros metales, los cuales han mostrado en los últimos años un gran potencial para esta reacción de hidrogenación. Dado el carácter bifuncional de los mecanismos de reacción involucrados en estas reacciones, se seleccionarán soportes micro y mesoporosos de composición variable (zeolitas, SBA-15, etc.), así como otros basados en estructura perovskita ABO_3. Para ello se emplearán una serie de técnicas de preparación recientemente descritas (Cristalización por Microondas, Proceso de Autocombustión, Mesoestructuración por Nanocasting y Porosidad Jerarquizada) que permiten obtener sistemas de alta superficie específica y nanoestructura controlada. La combinación de diferentes elementos en las posiciones A y B de la estructura perovskita, que actúen tanto como agentes promotores de los sistemas catalíticos como de precursores de aleaciones metálicas en los sistemas catalíticos reducidos, permitirá obtener materiales con propiedades catalíticas modulables, muy variadas y versátiles.

This project will carry out several studies and developments related to the reduction of CO_2 to valuable products, such as methane, light olefins, gasolines and other functionalized hydrocarbons, of economic, energetic and environmental interest. The use of hydrogen as a reducing agent, obtained from renewable sources, in addition to the reduction of greenhouse gas emissions, is a way to store energy from renewable sources, many of which are intermittent and therefore difficult to match with consumption needs.

Therefore, this project proposes the development of new heterogeneous catalytic systems based on Ni, Fe, Co, Ru and In, among other metals, which have shown in recent years

a great potential for this hydrogenation reaction. Given the bifunctional character of the reaction mechanisms involved in these reactions, micro and mesoporous supports of variable composition (zeolites, SBA-15, etc.) will be selected, as well as others based on ABO_3 perovskite structure. For this purpose, a series of recently described preparation techniques (Microwave Crystallization, Self-Combustion Process, Mesostructuring by Nanocasting and Hierarchical Porosity) will be used to obtain systems with high specific surface area and controlled nanostructure. The combination of different elements in the A and B positions of the perovskite structure, acting both as promoting agents of the catalytic systems and as precursors of metallic alloys in the reduced catalytic systems, will allow obtaining materials with modulable, varied and versatile catalytic properties.

CO_2 como fuente de carbono para la producción de compuestos químicos de alto valor añadido Formic acid as energy vector: feasability of hydrogen charge/discharge cycles

Código/Code:	US-1263288
Periodo/Period:	01-02-2020 / 31-10-2020
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	90.000 €
Investigador responsable/Research head:	José Antonio Odriozola Gordón y Svetlana Ivanova
Componentes/Research group:	Anna Dimitrova Penkova, Ligia Amelia Luque Álvarez, Débora Álvarez Hernández

RESUMEN / ABSTRACT

El presente proyecto se encuadra en la actual tendencia a nivel mundial de búsqueda de tecnologías para la captura y uso del dióxido de carbono (Carbon dioxide Capture and Utilization CCU). Su interés radica en la utilización directa del CO_2 atmosférico para almacenar hidrógeno verde, esto es, producido con la ayuda de energías renovables, en forma de ácido fórmico, usado como vector energético. Desde el punto de vista medioambiental, el desarrollo de esta tecnología permitiría preservar la huella de CO_2 durante el ciclo completo de generación, almacenamiento y liberación de energía, sin generar más gases de efecto invernadero. La posibilidad de almacenar hidrógeno de esta forma facilitaría su transporte y su uso en aplicaciones deslocalizadas diversas, tanto móviles como estacionarias. Indirectamente, esta tecnología racionalizaría el almacenamiento de las energías renovables, haciéndolas independientes de las condiciones climáticas. Este proyecto pretende estudiar la viabilidad de la tecnología basándose en el desarrollo de un único catalizador, estable y selectivo para los ciclos de carga y descarga de hidrógeno (CO_2/HCOOH).

This project is part of the current trend for future technologies of Carbon dioxide Capture and Utilization (CCU). His interest lies in a direct use of atmospheric CO_2 to store green hydrogen (produced with the help of renewable energies) as formic acid directly used as an

energy vector. From an environmental point of view, the development of this technology would make possible the preservation of the CO_2 footprint during the complete cycle of energy generation, storage and release, without generating more greenhouse gases. The possibility of storing hydrogen in this way would facilitate its transport and its use in diverse applications, both mobile and stationary. Indirectly, this technology would rationalize the storage of renewable energies, making them independent of climatic conditions. This project aims to study the feasibility of the technology based on the development of one unique stable and selective catalyst for both, hydrogen charge and discharge cycles (CO_2/HCOOH).

OTROS PROYECTOS / OTHER PROJECTS

Incentivo al Grupo de Investigación TEP-106

Tipo de Proyecto/Ayuda:	Ayudas a Consolidación de Grupos de la Junta de Andalucía
Código/Code:	2019/TEP-106
Periodo/Period:	01-01-2020 / 30-06-2021
Organismo Financiador/Financial source:	Junta de Andalucía. Universidad de Sevilla
Investigador responsable/Research head:	José Antonio Odriozola Gordón

Incentivo al Grupo de Investigación FQM-181

Tipo de Proyecto/Ayuda:	Ayudas a Consolidación de Grupos de la Junta de Andalucía
Código/Code:	2019/FQM-181
Periodo/Period:	01-01-2020 / 30-06-2021
Organismo Financiador/Financial source:	Junta de Andalucía. Universidad de Sevilla
Investigador responsable/Research head:	José Antonio Navío Santos

Incentivo al Grupo de Investigación FQM-015

Tipo de Proyecto/Ayuda:	Ayudas a Consolidación de Grupos de la Junta de Andalucía
Código/Code:	2019/FQM-015
Periodo/Period:	01-01-2020 / 30-06-2021
Organismo Financiador/Financial source:	Junta de Andalucía. Universidad de Sevilla
Investigador responsable/Research head:	Alfonso Caballero Martínez

AYUDAS PARA LA ADQUISICIÓN DE EQUIPOS

Biorefinería: Aprovechamiento de biomasa residual para la obtención de biocombustibles y compuestos de alto valor añadido (EQC2019-005458-P)
Financia: Ministerio de Ciencia e Innovación y Consejo Superior de Investigaciones Científicas
Importe Concedido: 196.856,00 €
Periodo: 1-1-2019 / 31-12-2021
Cofinanciado por el Grupo de Investigación "Química de Superficies y Catálisis"

COOPERACIÓN INTERNACIONAL Y OTROS INTERNATIONAL COOPERATION AND OTHERS

CO_2 Value Europe

Tipo de Proyecto/Ayuda:	Plan Propio de la Universidad de Sevilla
Modalidad:	Acciones especiales de internacionalización
Código/Code:	PP2019-12554
Organismo Financiador/Financial source:	Universidad de Sevilla
Investigador responsable/Research head:	José Antonio Odriozola Gordón

Estudio teórico-experimental de la estructura y conductividad iónica de silicofosfatos de litio vítreos

Tipo de Proyecto/Ayuda:	Agencia Española de Cooperación Internacional
Modalidad:	Acciones especiales de internacionalización
Código/Code:	AECI/2001/00000143
Organismo Financiador/Financial source:	Agencia Española de Cooperación Internacional
Investigador responsable/Research head:	José Antonio Odriozola Gordón

PATENTES / PATENTS

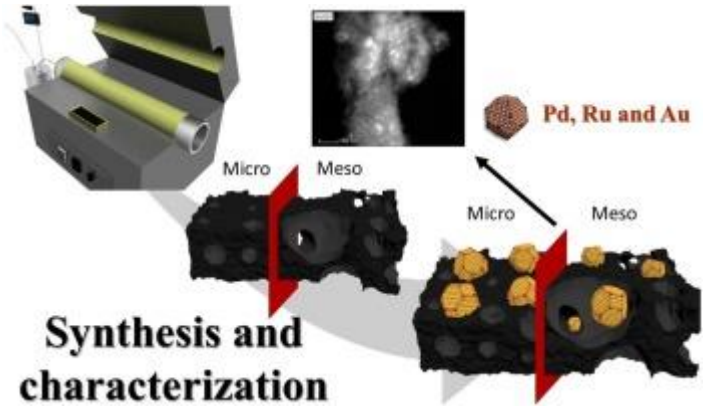
Procedimiento de obtención de monolitos integrales de carbono

Inventores: Miguel Ángel Centeno Gallego, José Antonio Odriozola Gordón, Nicolás Rodríguez Riaño, José Luis Santos Muñoz
Tipo de Patente: Internacional
Número de Solicitud: PCT/ES20/070731
Fecha Solicitud: 24 de noviembre de 2020
Entidad Titular: Universidad de Sevilla, Universidad de Colombia, Consejo Superior de Investigaciones Científicas

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

Metal catalysts supported on biochars: Part I synthesis and characterization

Santos, JL; Maki-Arvela, P; Monzon, A; Murzin, DY; Centeno, MA
Applied Catalysis B: Enviromental, **258** (2020) 118423
Julio, 2020 | **DOI:** 10.1016/j.apcatb.2019.118423

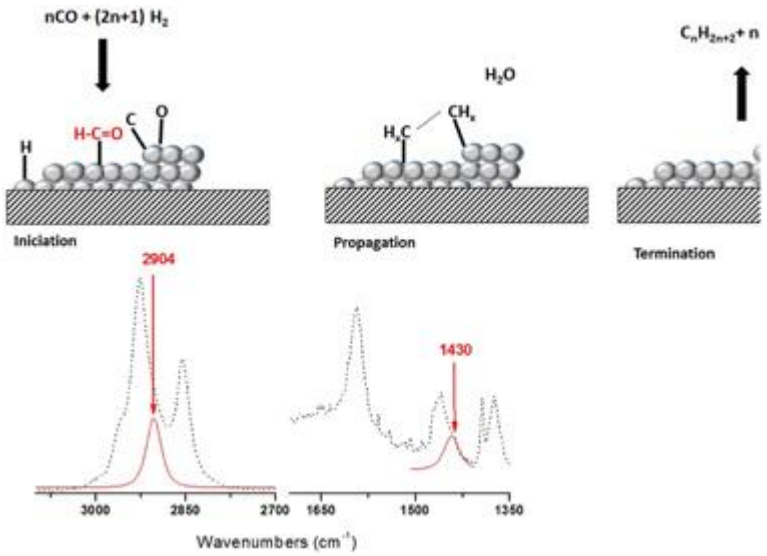


In the current study, synthesis and detailed characterization of cellulose biochars as a waste biomass model component and vine shoot biochars as a real waste biomass catalyst was performed. Although initially biochars exhibit poor textural properties, a simple activation process can make them much more suitable as a catalyst supports. A combination of physical (CO_2) and chemical activation ($ZnCl_2$) was evaluated. The characterization results indicated that the surface area and pore volume of the biochars have increased significantly by chemical activation treatment with $ZnCl_2$. A series of metal catalysts (Pd, Au and Ru) supported on biochars was prepared and characterized. The prepared materials represent a set of noble metal catalysts supported on biochars with different textural and surface properties, which can be used to evaluate the catalytic role of the active phase and carbon support nature in catalytic reactions of interest, such as hydrodeoxygenation, described in the part II.

Experimental evidence of HCO species as intermediate in the fischer tropsch reaction using operando techniques

Díaz-Sánchez, RM; de-Paz-Carrion, A; Serrera-Figallo, MA; Torres-Lagares, D; Barranco, A; Leon-Ramos, JR; Gutiérrez-Pérez, JL
Applied Catalysis B: Enviromental, **272** (2020) 119032
Septiembre, 2020 | **DOI:** 10.1016/j.apcatb.2020.119032

Fischer Tropsch's reaction, known from 1925, receives special attention nowadays due to its key role in the CO_2 or biomass valorization to liquid fuels and chemicals. Several aspects on the exact mechanism or the role of water in this reaction are not yet completely clear. Formyl species, HCO, have been proposed as the most probable reaction intermediate, but they have never been observed under operation conditions closed to the real ones. In this work, using DRIFTS-MS operando techniques, HCO intermediates are detected under a H_2/CO flow and 200 °C. IR bands at 2900 cm^{-1} and 1440 cm^{-1} attributed to ν(C–H) and δ(HCO) vibrations modes characterize these species. Evolution of these bands with the reaction time evidences its high reactivity with OH groups, which explains the positive effect of water on the CO conversion previously observed.



Hydrodeoxygenation of vanillin over noble metal catalyst supported on biochars: Part II: Catalytic behaviour

Santos, JL; Maki-Arvela, P; Warna, J; Monzon, A; Centeno, MA; Murzin, DY

Applied Catalysis B: Enviromental, **268** (2020) 118425

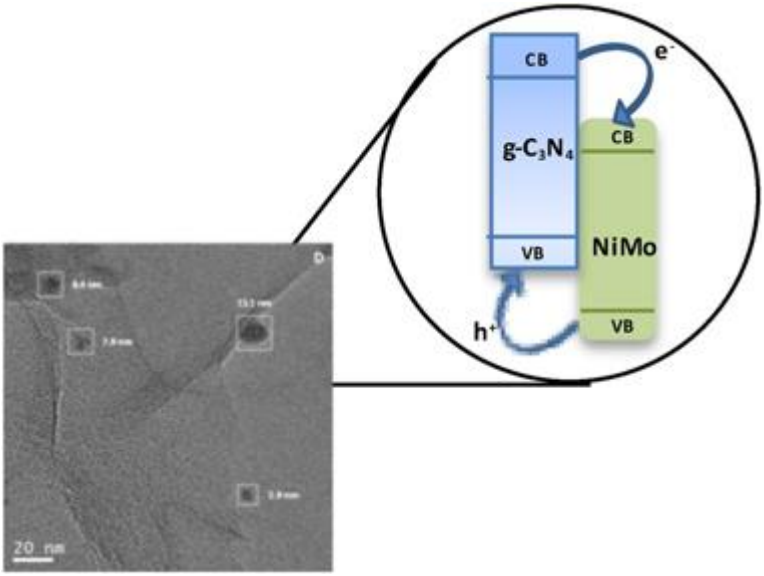
Julio, 2020 | **DOI:** 10.1016/j.apcatb.2019.118425

Vanillin hydrodeoxygenation was investigated using noble metal (Pd, Au, Ru) supported on active carbon prepared from waste derived biochars, which were produced via pyrolysis in CO_2 atmosphere. Chemical activation with $ZnCl_2$ and HNO_3 was also used in the preparation of active carbon to enhance the specific surface area and demineralize material, respectively. Both fresh and spent catalysts were characterized with X-ray diffraction, DRIFTS, zeta potential measurement and HR-TEM. The highest selectivity to p-creosol, 92 % selectivity at complete vanillin conversion after 3 h was obtained in vanillin hydrodeoxygenation at 100 °C under 30 bar in hydrogen in water with Pd/C catalyst prepared via pyrolysis under CO_2 from wine waste and using $ZnCl_2$ as a chemical activation agent. Hydrodeoxygenation activity increased with increasing metal dispersion. A kinetic model including adsorption of vanillin described well the experimental data.

$(NH_4)_4[NiMo_6O_{24}H_6].5H_2O$ / g-C_3N_4 materials for selective photo-oxidation of C—O and C=C bonds

Caudillo-Flores, U; Ansari, F; Bachiller-Baeza, B; Colon, G; Fernández-García, M; Kubacka, A
Applied Catalysis B: Enviromental, **278** (2020) 119299
Diciembre, 2020 | **DOI:** 10.1016/j.apcatb.2020.119299

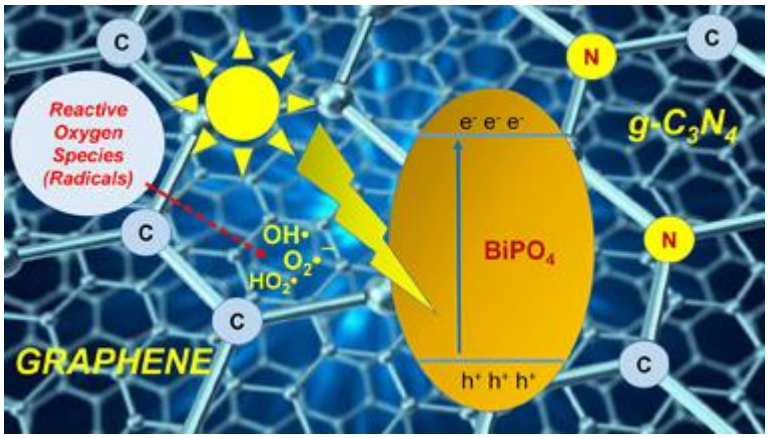
Novel composite photo-catalysts having $(NH_4)_4[NiMo_6O_{24}H_6]\cdot 5H_2O$ Polyoxometalate (POM) species deposited over g-C_3N_4 are synthesized. Materials were characterized through a multitechnique approach showing the stability of the carbon nitride component both through the synthesis process and under reaction. Contrarily, the POM component evolves under reaction conditions to maximize the interaction with the support. Such a behavior renders, as measured by the quantum efficiency, highly active photo-catalysts in the photo-oxidation of 2-propanol and styrene both under UV and sunlight illumination, setting up the basis for a green catalytic process. The material having a 4 wt. % POM showed improved activity with respect to both parent constituents but also higher selectivity to the partial oxidation of the alcohol and the aromatic hydrocarbon to generate added value chemical compounds. A multitechnique approach investigating charge carrier fate demonstrates the key role played by the interaction between components to promote activity and selectivity in selective oxidation reactions.



Recent progress on the enhancement of photocatalytic properties of $BiPO_4$ using π–conjugated materials

Naciri, Y., Hsini, A., Ajmal, Z., Navio, J.A., Bakiz, B., Albourine, A., Ezahri, M., Benlhachemi, A.
Advances in Colloid and Interface Science, **280** (2020) 102160
Junio, 2020 | **DOI:** 10.1016/j.cis.2020.102160

Semiconductor photocatalysis is regarded as most privileged solution for energy conversion and environmental application. Recently, photocatalysis methods using bismuth-based photocatalysts, such as $BiPO_4$, have been extensively investigated owing to their superior efficacy regarding organic pollutant degradation and their further mineralization into CO_2 and H_2O. It is well known that $BiPO_4$ monoclinic phase exhibited better photocatalytic performance compared to Degussa (Evonik) P25 TiO_2 in term of ultraviolet light driven organic pollutants degradation. However, its wide band gap, poor adsorptive performance and large size make $BiPO_4$ less active under visible light irradiation. However, extensive research works have been

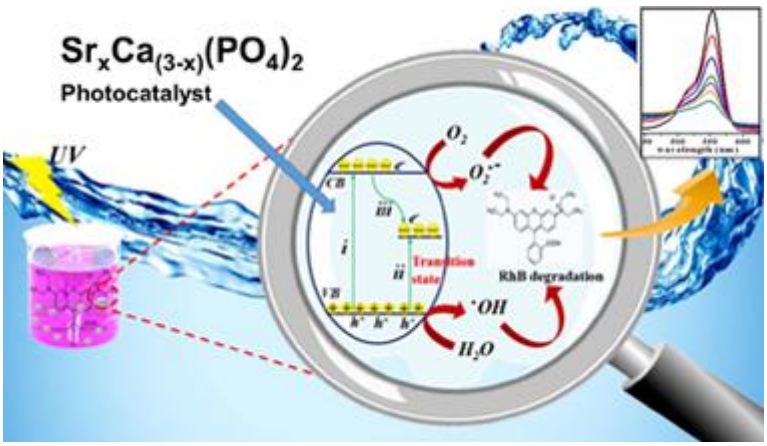


conducted in the past with the aim of improving visible light driven $BiPO_4$ activity by constructing a series of heterostructures, mainly coupled with π-conjugated architecture (e.g., conductive polymer, dye sensitization and carbonaceous materials). However, a critical review of modified $BiPO_4$ systems using π-conjugated materials has not been published to date. Therefore, this current review article was designed with the aim of presenting a brief current state-of-the-art towards synthesis methods of $BiPO_4$ in the first section, with an especial focuses onto its crystal-microstructure, optical and photocatalytic properties. Moreover, the most relevant strategies that have been employed to improve its photocatalytic activities are then addressed as the main part of this review. Finally, the last section presents ongoing challenges and perspectives for modified $BiPO_4$ systems using π–conjugated materials.

Influence of Sr-doping on structural, optical and photocatalytic properties of synthesized $Ca_3(PO_4)_2$

Y.Naciri; A.Hsini; Z.Ajmal; A.Bouddouch; B.Bakiz; J.A.Navío; A.Albourine; J-C.Valmalette; M.Ezahri; A.Benlhachemi

Journal of Colloid and Interface Science, **572** (2020) 269-280
Julio, 2020 | **DOI:** 10.1016/j.jcis.2020.03.105

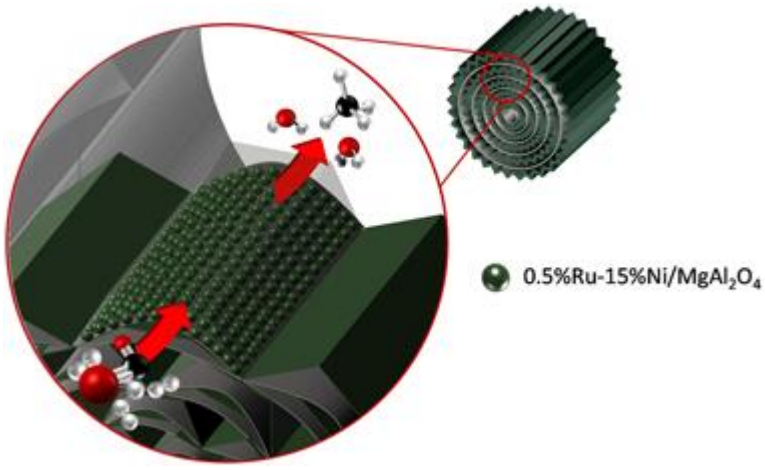


Well-crystallized $Ca_3(PO_4)_2$ doped and un-doped nano-particles with the maximum strontium content (40 wt% Sr) followed by calcination at 800 °C for 3 h were synthesized via facile co-precipitation method. DTA/TGA, X-ray diffraction (XRD), energy dispersive scanning electron microscopy (SEM/EDX), UV–vis diffuse reflectance spectrum (UV–vis DRS), Raman spectroscopy and photoluminescence (PL) techniques were used for material characterization. The (XRD) patterns of as-synthesized Sr-doped $Ca_3(PO_4)_2$ solid solution samples exhibited a systematic shift toward lower angles by possessing a single rhombohedral crystal structure without any secondary phases. The UV light driven photocatalytic activity was assessed for rhodamine B (RhB) degradation. As a result, ultrafast photodegradation activity was

observed after Sr doping. Moreover, the 30 wt% Sr-$Ca_3(PO_4)_2$ sample showed the highest photocatalytic degradation among the Sr-doped $Ca_3(PO_4)_2$ samples toward RhB. It was further suggested that as-synthesized 30 wt% Sr-$Ca_3(PO_4)_2$ superior photocatalytic performance is ascribed to the more proficient partition of photogenerated electron-hole pairs. Furthermore, the involved mechanism of superior photocatalytic performance of the 30 wt% Sr-$Ca_3(PO_4)_2$ solid solution was also investigated. In addition, regeneration cycles indicated the higher stability of the photocatalyst to be effectively recycled up to four times without any considerable reduction in photocatalytic performance. Thus, these informations further provides us a scalable pathway to fabricate Sr doped $Ca_3(PO_4)_2$ and its consequent use as an efficient photocatalyst for rhodamine B (RhB) contaminated wastewater treatment.

Ru-Ni/$MgAl_2O_4$ structured catalyst for CO_2 methanation

Navarro, Juan C.; Centeno, Miguel A.; Laguna, Oscar H.; Odriozola, José A.
Renewable Energy, **161** (2020) 120-132
Diciembre, 2020 | **DOI:** 10.1016/j.renene.2020.07.055

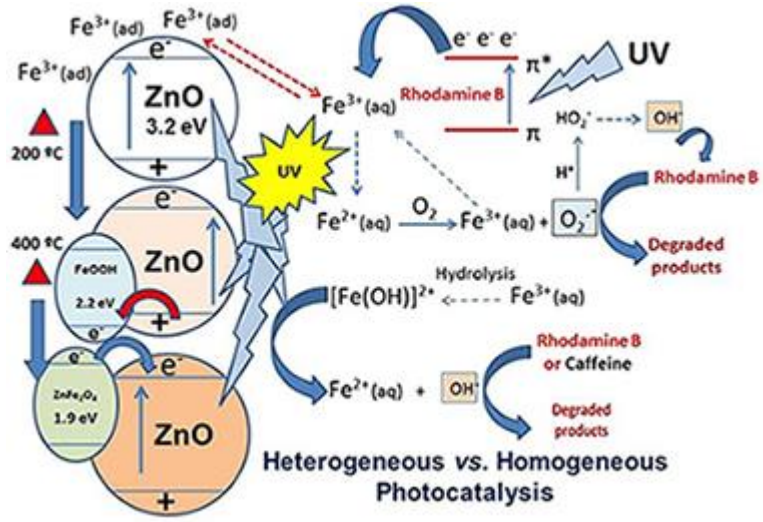


Semiconductor photocatalysis is regarded as most privileged solution for energy conversion and environmental application. Recently, photocatalysis methods using bismuth-based photocatalysts, such as $BiPO_4$, have been extensively investigated owing to their superior efficacy regarding organic pollutant degradation and their further mineralization into CO_2 and H_2O. It is well known that $BiPO_4$ monoclinic phase exhibited better photocatalytic performance compared to Degussa (Evonik) P25 TiO_2 in term of ultraviolet light driven organic pollutants degradation. However, its wide band gap, poor adsorptive performance and large size make $BiPO_4$ less active under visible light irradiation. However, extensive research works have been conducted in the past with the aim of improving visible light driven $BiPO_4$ activity by constructing a series of heterostructures, mainly coupled with π-conjugated architecture (e.g., conductive polymer, dye sensitization and carbonaceous materials). However, a critical review of modified $BiPO_4$ systems using π-conjugated materials has not been published to date. Therefore, this current review article was designed with the aim of presenting a brief current state-of-the-art towards synthesis methods of $BiPO_4$ in the first section, with an especial focuses onto its crystal-microstructure, optical and photocatalytic properties. Moreover, the most relevant strategies that have been employed to improve its photocatalytic activities are then addressed as the main part of this review. Finally, the last section presents ongoing challenges and perspectives for modified $BiPO_4$ systems using π–conjugated materials.

Role of Fe(III) in aqueous solution or deposited on ZnO surface in the photoassisted degradation of rhodamine B and caffeine

Tanji, Karim; Navio, J A; Martín-Gómez, A N; Hidalgo, M C; Jaramillo-Paez, C; Naja, Jamal; Hassoune, Hicham; Kherbeche, Abdelhak

Chemosphere, **241** (2020) 125009
Febrero, 2020 | **DOI:** 10.1016/j.chemosphere.2019.125009



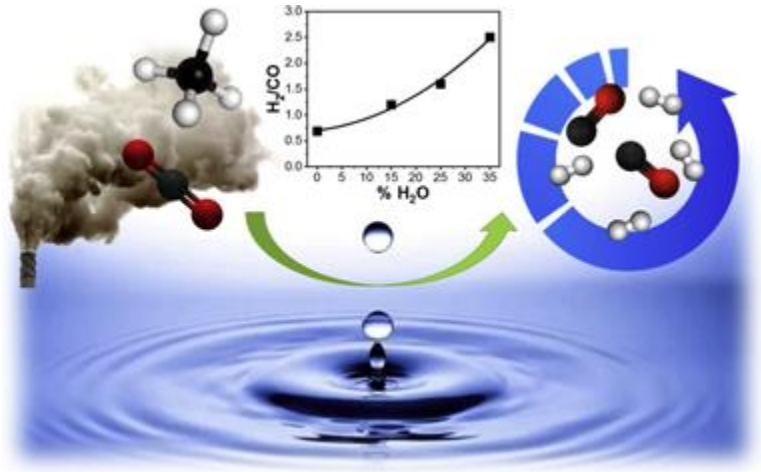
Iron (III) was incorporated, to the surface of a synthesized ZnO, using two nominal molar percentages of Fe (III): 1% and 5% Fe relative to ZnO. Samples dried and calcined at 200 °C and 400 °C for 2 h, were characterized by XRD, XPS, XRF, N2-adsorption-BET and (UV–vis)-DRS. Photocatalytic activities of the catalysts were assessed based on the degradation of rhodamine B (RhB) and caffeine (CAF) in aqueous solution under two irradiation conditions: UV and visible light illumination. Prior to the photocatalytic tests, the interaction of each one of the substrates with either Fe(III) or Fe(II) was studied in homogeneous medium under UV-illumination and oxygenated environment. It was found that Fe (III) can play an important role in homogeneous media in the photoassisted degradation, both of rhodamine B and caffeine, while Fe (II) does not exert a relevant role in the photoassisted degradation of the referred substrates. Fe–ZnO samples display similar or poorer performance than pure ZnO in the presence of UV light for both studied substrates. The phenomenon can be attributed to the formation of either goethite or $ZnFe_2O_4$ at the ZnO surface where the coupled Fe^{3+}/Fe^{2+} can act as recombination centers for the photogenerated charges. On the contrary, all Fe–ZnO samples showed enhanced photocatalytic activity under visible illumination which seems to be independent of the iron content. In this context, the mechanisms for photoassisted degradation of both the substrates in homogeneous medium and photocatalytic degradation are discussed, as well as the role of Fe in the photodegradation processes.

Flexible syngas production using a $La_2Zr_{2-x}Ni_xO_7$-delta pyrochlore-double perovskite catalyst: Towards a direct route for gas phase CO_2 recycling

Le Sache, E; Pastor-Pérez, L; Garcilaso, V; Watson, DJ; Centeno, MA; Odriozola, JA; Reina, TR

Catalysis Today, **357** (2020) 583-589
Noviembre, 2020 | **DOI:** 10.1016/j.cattod.2019.05.039

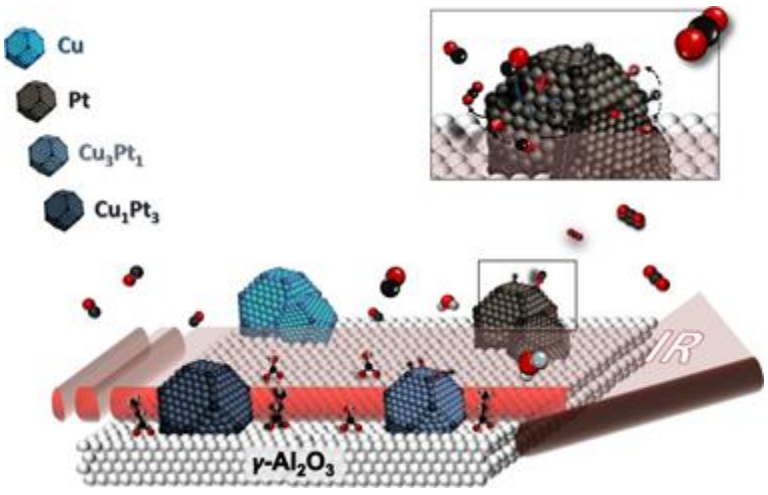
The bi-reforming of methane (BRM) has the advantage of utilising greenhouse gases and producing H_2 rich syngas. In this work Ni stabilised in a pyrochlore-double perovskite structure is reported as a viable catalyst for both Dry Reforming of Methane (DRM) and BRM. A 10 wt.%



Ni-doped $La_2Zr_2O_7$ pyrochlore catalyst was synthesised, characterised and tested under both reaction conditions and its performance was compared to a supported Ni/$La_2Zr_2O_7$. In particular the effect of steam addition is investigated revealing that steam increases the H_2 content in the syngas but limits reactants conversions. The effect of temperature, space velocity and time on stream was studied under BRM conditions and brought out the performance of the material in terms of activity and stability. No deactivation was observed, in fact the addition of steam helped to mitigate carbon deposition. Small and well dispersed Ni clusters, possibly resulting from the progressive exsolution of Ni from the mixed oxide structure could explain the enhanced performance of the catalyst.

Upgrading the PtCu intermetallic compounds: The role of Pt and Cu in the alloy

Castillo, R; García, ED; Santos, JL; Centeno, MA; Sarria, FR; Daturi, M; Odriozola, JA
Catalysis Today, **356** (2020) 390-398
Octubre, 2020 | **DOI:** 10.1016/j.cattod.2019.11.026



This work is devoted to the study of the role of both metals in the intermetallic PtxCuy/ γ Al_2O_3 catalysts commonly employed in CO-PROX reaction. Therefore, monometallic Pt and Cu based catalysts and PtCu intermetallic compound with different molar ratios (Pt_3Cu_1 and Pt_1Cu_3) supported catalysts were carefully synthesized and deeply characterized. Room temperature CO adsorptions by FTIR spectroscopy were carried out on the mono- and intermetallic catalysts being the monometallic catalyst determinant for the study. From the analysis of the nature of the platinum surface in Pt/ γ Al_2O_3, we have demonstrated that the role of Pt sites is based in the CO dissociation for the CO_2 formation and also how the platinum surface is partially blocked by leftovers from the synthesis. Moreover, the study of the Cu/ γ Al_2O_3 and the bimetallic catalysts PtxCuy/ γ Al_2O_3 allowed elucidating the effect of the copper in

the metallic site and support interphase as well as the role of copper in the hydrocarbon oxidation.

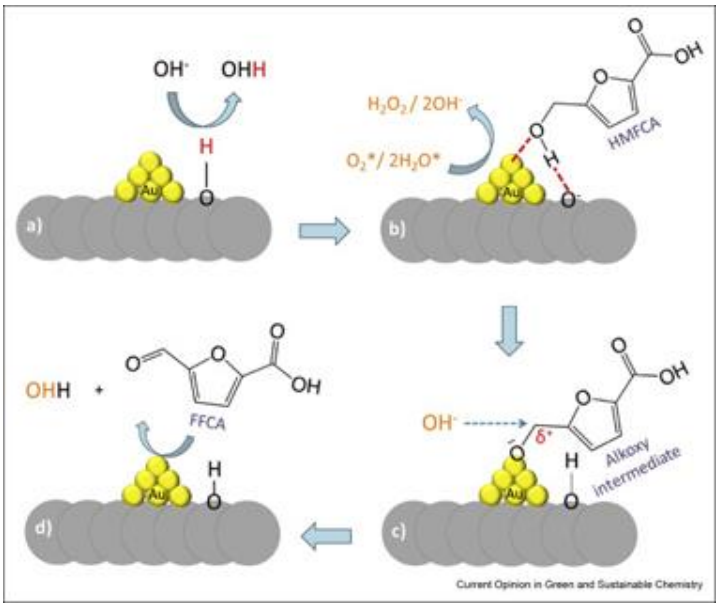
Cost-effective routes for catalytic biomass upgrading

Jin, W; Pastor-Pérez, L; Yu, J; Odriozola, JA; Gu, S; Reina, TR
Current Opinion in Green and Sustainable Chemistry, **23** (2020) 1-9
Junio, 2020 | **DOI:** 10.1016/j.cogsc.2019.12.008

Catalytic hydrodeoxygenation (HDO) is a fundamental and promising route for bio-oil upgrading to produce petroleum-like hydrocarbon fuels or chemical building blocks. One of the main challenges of this technology is the demand of high-pressure H_2, which poses high costs and safety concerns. Accordingly, developing cost-effective routes for biomass or bio-oil upgrading without the supply of commercial H_2 is essential to implement the HDO at commercial scale. This article critically reviewed the very recent studies relating to the novel strategies for upgrading the biofeedstocks with 'green' H_2 generated from renewable sources. More precisely, catalytic transfer hydrogenation/hydrogenolysis, combined reforming and HDO, combined metal hydrolysis and HDO, water-assisted *in-situ* HDO and nonthermal plasma technology and self-supported hydrogenolysis are reviewed herein. Current challenges and research trends of each strategy are also proposed aiming to motivate further improvement of these novel routes to become competitive alternatives to conventional HDO technology.

Recent advances in selective oxidation of biomass-derived platform chemicals over gold catalysts

Megias-Sayago, C; Navarro-Jaen, S; Castillo, R; Ivanova, S
Current Opinion in Green and Sustainable Chemistry, **21** (2020) 50-55
Febrero, 2020 | **DOI:** 10.1016/j.cogsc.2019.12.001



Gold is without a doubt the best known metal for chemical oxidation. The noblest of the noble metals gained its place because of its resistance to overoxidation, low temperature of operation, especially in gas-phase oxidation, and fairly good selectivity when required. The aim for sustainable development and the need for new technologies open the possibility to introduce new raw materials and new catalyst formulation. That is why new horizons appear in the otherwise uncertain future of gold catalysis. The old glory becomes now a glorious alternative, and this mini-review gives only a small example of it.

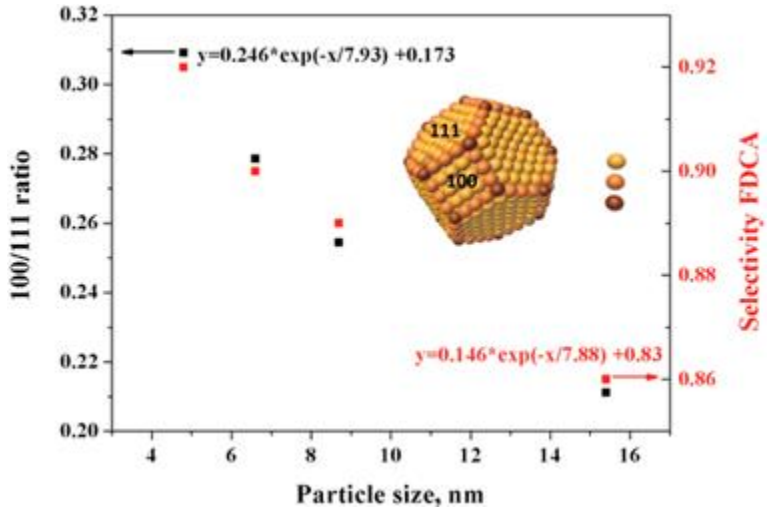
Bimetallic PdAu catalysts for formic acid dehydrogenation

Santos, JL; Leon, C; Monnier, G; Ivanova, S; Centeno, MA; Odriozola, JA
International Journal of Hydrogen Energy, **45** (2020) 23056-23068
Septiembre, 2020 | **DOI:** 10.1016/j.ijhydene.2020.06.076

A series of monometallic and bimetallic palladium gold catalyst were prepared and studied for the formic acid dehydrogenation reaction. Different Pd/Au compositions were employed (Pd_xAu_{100-x}, where x = 25; 50 and 75) and their impact on alloy structure, particle size and dispersion was evaluated. Active phase composition and reaction parameters such as temperature, formic acid concentration or formate/formic acid ratio were adjusted to obtain active and selective catalyst for hydrogen production. An important particle size effect was observed and related to Pd/Au composition for all bimetallic catalysts.

Effect of Gold Particles Size over Au/C Catalyst Selectivity in HMF Oxidation Reaction

Megias-Sayago, C; Lolli, A; Bonincontro, D; Penkova, A; Albonetti, S; Cavani, F; Odriozola, JA; Ivanova, S
Chemcatchem, **12** (2020) 1177-1183
Febrero, 2020 | **DOI:** 10.1002/cctc.201901742

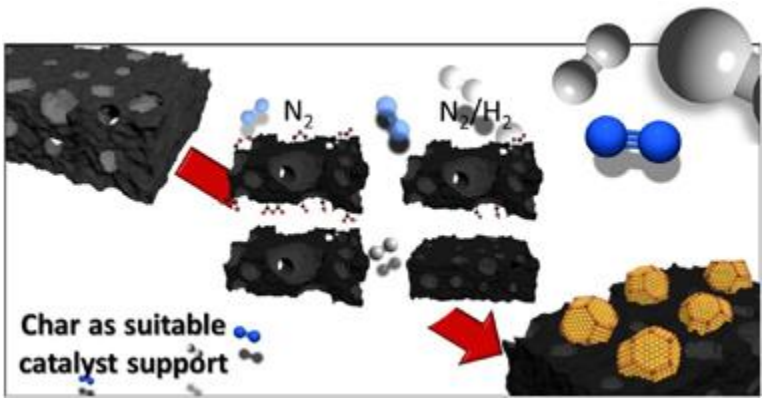


A series of gold nanoparticles in the 4-40 nm range were prepared, immobilized on activated carbon and further tested, at low base concentration, in the catalytic oxidation of 5-hydroxymethyl furfural (HMF) to 2,5-furandicarboxylic acid (FDCA). Gold particles size variation has no influence on HMF conversion but significantly affects product selectivity and carbon balance. This behavior is ascribed to the thermodynamically favorable oxygen reduction reaction on Au(100) faces. As the gold particle size decreases the Au(100)/Au(111) exposure

ratio, estimated by using the van Hardeveld-Hartog model, increases as well as the FDCA selectivity. The smaller the gold particle size the smaller the 5-hydroxymethyl-2-furancarboxylic acid (HMFCA) to FDCA ratio pointing to the gold size dependent behavior of the oxidation of the alcohol function of the HMF molecule.

Reductant atmospheres during slow pyrolysis of cellulose: First approach to obtaining efficient char -based catalysts in one pot

Santos, JL; Centeno, MA; Odriozola, JA
Journal of Analytical and Applied Pyrolysis, **148** (2020) 104821
Junio, 2020 | **DOI:** 10.1016/j.jaap.2020.104821



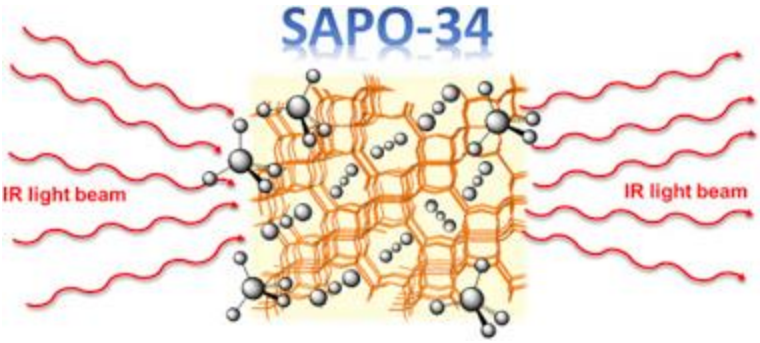
Char based metallic (Pd-Au-Ru-Pt/C) catalysts have drawn increasing research interest due to their versatility in biomass related industrial reactions. Recent studies dealing with the synthesis of char-based catalysts in one single step (one-pot) use reductant atmospheres for biomass pyrolysis. In this work, the influence of the use of a reductant N_2/H_2 atmosphere on the physicochemical properties of the resulting chars was evaluated in comparison with the use of an inert N_2 atmosphere. Specifically, the fundamental parameter of the pyrolysis process, the temperature, was evaluated in the 500–900 °C range. Produced chars were fully characterized by N_2 isotherms, ultimate CHNS analysis, X-ray Diffraction, Raman spectroscopy, Diffuse Reflectance Infrared spectroscopy, X-ray Photoelectron spectroscopy, helium Temperature Programmed Decomposition and Isoelectric Point analysis. Slow pyrolysis under reductant atmosphere favours deoxygenation reaction against dehydrogenation ones, reduces the carbon yield and results in chars with a more hydrophobic and graphitic character, higher thermal stability and weak surface functionalization. The use of intermediates temperatures (700 °C) favours the obtaining of chars with suitable physicochemical properties and good surface functionalization, which will facilitate the anchoring of the active phase on the surface, improving the metallic dispersion of the resulting one pot catalyst. This leads us to affirm that the use of reducing atmospheres at intermediate temperatures, is superior to the use of inert atmospheres for this purpose. This analysis on the impact of the use of a reductant atmosphere during slow pyrolysis of microcrystalline cellulose opens a new working path for the optimization of char-based catalysts obtained in a single stage.

Time-resolved operando DRIFTS-MS study of the moisture tolerance of small-pore SAPO-34 molecular sieves during CH_4/CO_2 separation

Romero, M; Navarro, JC; Bobadilla, LF; Domínguez, MI; Ivanova, S; Romero-Sarria, F; Centeno, MA; Odriozola, JA

Microporous and Mesoporous Materials, **298** (2020) 110071

Mayo, 2020 | **DOI:** 10.1016/j.micromeso.2020.110071



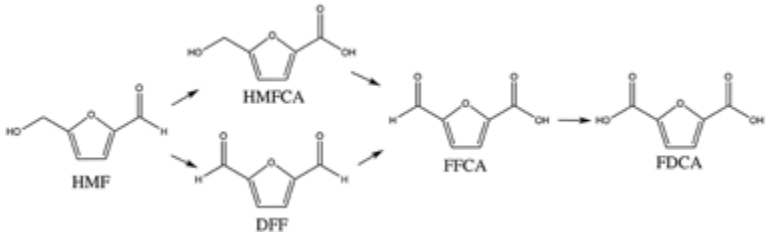
This study pretends to evaluate and understand the effect of moisture presence during CO_2/CH_4 separation on small-pore SAPO-34 molecular sieves. Two SAPO-34 samples with different physicochemical properties (composition, crystal size and texture) were prepared by hydrothermal synthesis using either one or a mixture of two templates. Transient operando DRIFTS-MS measurements revealed that the sample's hydrophobic character is associated to the presence of Si islands, which enhanced sample's moisture tolerance during repetitive adsorption/desorption cycles. This knowledge is fundamental to achieve the rational design of efficient SAPO-34 membranes under realistic conditions.

5-Hydroxymethyl-2-Furfural Oxidation Over $Au/Ce_xZr_{1-x}O_2$ Catalysts

Megias-Sayago, C; Bonincontro, D; Lolli, A; Ivanova, S; Albonetti, S; Cavani, F; Odriozola, JA

Frontiers in Chemistry, **8** (2020) 461

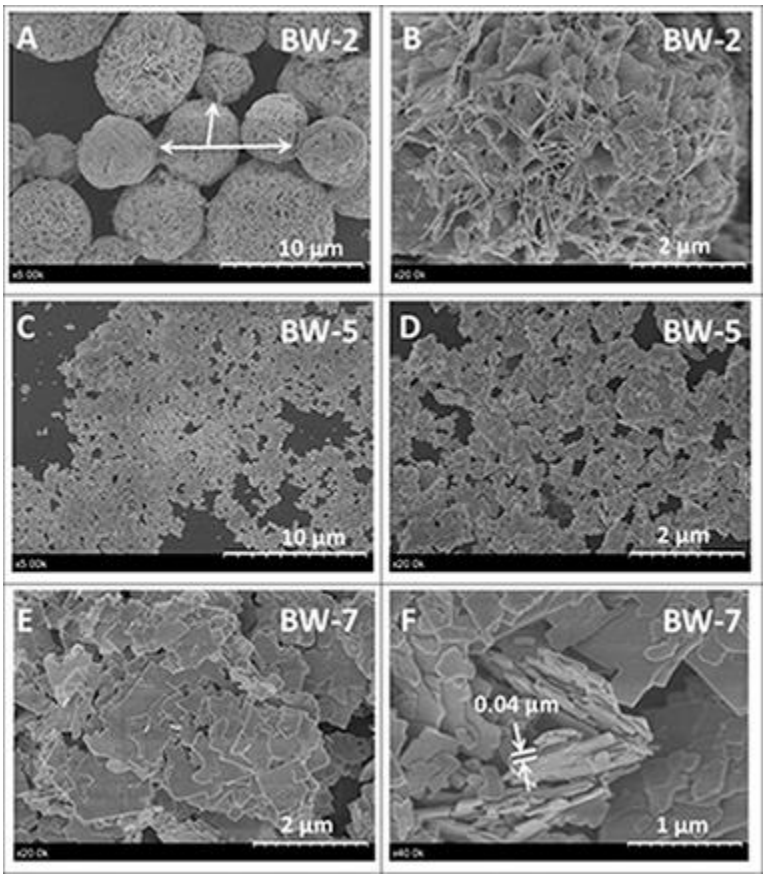
Junio, 2020 | **DOI:** 10.3389/fchem.2020.00461



A series of gold catalysts supported on pure CeO_2, ZrO_2, and two different Ce-Zr mixed oxides have been prepared and tested in the 5-hydroxymethyl-2-furfural oxidation reaction. All catalysts show high catalytic activity (100% conversion) and important selectivity (27-41%) to the desired product i.e., 2,5-furandicarboxylic acid at low base concentration. Products selectivity changes with the support nature as expected, however, the observed trend cannot be related neither to gold particle size, nor to catalyst reducibility and oxygen mobility. An important relation between the FDCA selectivity and the support textural properties is observed, conducing to the general requirement for optimal pore size for this reaction.

Effect of synthesis pH on the physicochemical properties of a synthesized Bi_2WO_6 and the type of substrate chosen, in assessing its photo-catalytic activities

Jaramillo-Páez, C.; Navío, J.A.; Hidalgo, M.C.
Arabian Journal of Chemistry, **13** (2020) 431-443
Enero, 2020 | **DOI:** 10.1016/j.arabjc.2017.05.014



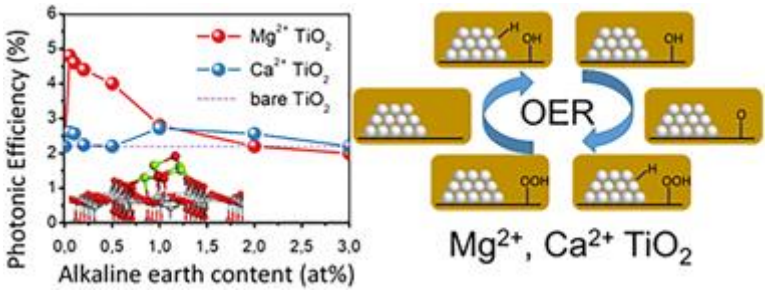
Crystalline orthorhombic Bi_2WO_6 powders were synthesized by a hydrothermal method from aqueous solutions of $Bi(NO_3)_3 \cdot 5H_2O$ and $Na_2WO_4 \cdot 2H_2O$ over a range of three selected pH values (2.0, 5.0 and 7.0), using NaOH as precipitating agent. The as-prepared catalysts were characterized by XRD, BET, FE-SEM, TEM, XPS and UV-vis spectroscopy. The effect of pH-synthesis on crystallinity, morphologies, surface area and optical absorption properties, were investigated.

Although the pH has a marked influence on morphology, the nature of the precipitating agent (NaOH or TEA) also influences the morphology and surface structure composition, as it is observed in the present work. Three different probe molecules were used to evaluate the photocatalytic properties under two illumination conditions (UV and Visible): Methyl Orange and Rhodamine B were chosen as dye substrates and Phenol as a transparent substrate. The photo-catalytic activities are strongly dependent not only on the pH used in the synthesis but also on the nature of the chosen substrate in assessing the photo-catalytic activities. Results were compared with those obtained when using TiO_2(P25, Evonik) in the same experimental conditions. The photocatalytic activity of one of the synthesised samples has been evaluated by exposing a mixture of Rhodamine B and Phenol in water, to different illumination conditions. Our results provide new evidences about the issue of whether dyes are suitable substrates to assess the activity of a photo-catalyst.

Surface Modification of Rutile TiO_2 with Alkaline-Earth Oxide Nanoclusters for Enhanced Oxygen Evolution

Rhatigan, S; Sukola, E; Nolan, M; Colon, G
ACS Applied Nano Materials, **3** (2020) 6017-6033
Junio, 2020 | **DOI:** 10.1021/acsanm.0c01237

The oxygen (O_2) evolution reaction (OER) is accepted as the bottleneck in the overall water splitting and has seen intense interest. In this work, we prepared rutile TiO_2 modified with nanoclusters of alkaline-earth metal oxides for the OER. Photocatalytic OER was performed over



rutile TiO_2 surface-modified with alkaline-earth oxide nanoclusters, namely, CaO and MgO. The O_2 evolution activity is notably enhanced for MgO-modified systems at low loadings and a combination of characterization and first-principles simulations allows interpretation of the role of the nanocluster modification in improving the photocatalytic performance of alkaline-earth-modified rutile TiO_2. At such low loadings, the nanocluster modifiers would be small, and this facilitates a close correlation with theoretical models. Structural and surface characterizations of the modified systems indicate that the integrity of the rutile phase is maintained after modification. However, charge-carrier separation is strongly affected by the presence of surface nanoclusters. This improved performance is related to surface features such as higher ion dispersion and surface hydroxylation, which are also discussed with first-principles simulations. The modified systems are reducible so that Ti^{3+} ions will be present. Water dissociation is favorable at cluster and interfacial sites of the stoichiometric and reduced modified surfaces. Pathways to water oxidation at interfacial sites of reduced MgO-modified rutile TiO_2 are identified, requiring an overpotential of 0.68 V. In contrast, CaO-modified systems required overpotentials in excess of 0.85 V for the reaction to proceed.

Influence of Water on the Oxidation of NO on Pd/TiO_2 Photocatalysts

M.J. Hernández Rodríguez; E. Pulido Melián; J. Araña; J.A. Navío; O.M. González Díaz; Dunia E. Santiago; J.M. Doña Rodríguez
Nanomaterials, **10** (2020) 2354
Diciembre, 2020 | **DOI:** 10.3390/nano10122354

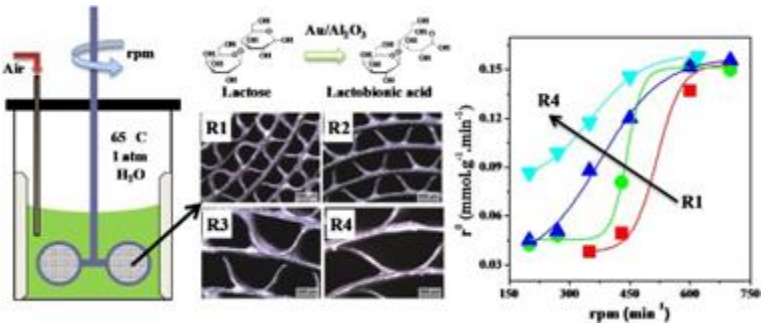
Two series of new photocatalysts were synthesized based on modification with Pd of the commercial P25 photocatalyst (EVONIK®). Two techniques were employed to incorporate Pd nanoparticles on the P25 surface: photodeposition (series Pd-P) and impregnation (series Pd-I). Both series were characterized in depth using a variety of instrumental techniques: BET, DRS, XRD, XPS, TEM, FTIR and FESEM. The modified series exhibited a significant change in pore size distribution, but no differences compared to the original P25 with respect to crystalline phase ratio or particle size were observed. The Pd0 oxidation state was predominant in the Pd-P series, while the presence of the Pd^{2+} oxidation state was additionally observed in the Pd-I series. The photoactivity tests were performed in a continuous photoreactor with the photocatalysts deposited, by dip-coating, on borosilicate glass plates. A total of 500 ppb of NO was used as input flow at a volumetric flow rate of 1.2 L·min−1, and different relative humidities from 0 to 65% were tested. The results obtained show that under UV-vis or Vis radiation, the presence of Pd nanoparticles favors NO removal independently of the Pd incorporation method employed

and independently of the tested relative humidity conditions. This improvement seems to be related to the different interaction of the water with the surface of the photocatalysts in the presence or absence of Pd. It was found in the catalyst without Pd that disproportionation of NO_2 is favored through its reaction with water, with faster surface saturation. In contrast, in the catalysts with Pd, disproportionation took place through nitro-chelates and adsorbed NO_2 formed from the photocatalytic oxidation of the NO. This different mechanism explains the greater efficiency in NO_x removal in the catalysts with Pd. Comparing the two series of catalysts with Pd, Pd-P and Pd-I, greater activity of the Pd-P series was observed under both UV-vis and Vis radiation. It was shown that the Pd0 oxidation state is responsible for this greater activity as the Pd-I series improves its activity in successive cycles due to a reduction in Pd^{2+} species during the photoactivity tests.

Monolithic stirrer reactor: The selective lactose oxidation in liquid phase over Au/Al_2O_3 nanostructured catalysts

Regenhardt, SA; Meyer, CI; Sanz, O; Sebastian, V; Ivanova, S; Centeno, MA; Odriozola, JA; Montes, M; Marchi, AJ; Garetto, TF

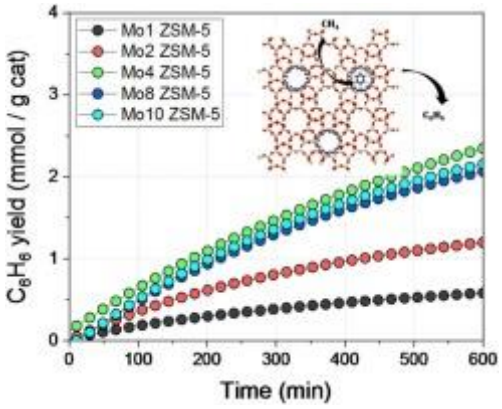
Molecular Catalysis, **481** (2020) 110219
Febrero, 2020 | **DOI:** 10.1016/j.mcat.2018.10.014



The performance of rotating metallic monolith stirrer reactor was studied for selective lactose oxidation in liquid phase at 65 °C, atmospheric pressure and with air as oxidant agent. The Au/Al_2O_3 deposition on metallic substrates was performed by wash-coating, producing catalyst coating thicknesses between 5 and 20 µm. Monoliths with different configuration (channel size between 0.36 and 1.06 mm) were used as stirrer blades in a batch reactor. Internal and external mass transfer limitations were observed during liquid phase lactose oxidation. For stirring rates equal or higher than 600 rpm there were no important external diffusional restrictions and this was also independent of the monolith configuration. Coating with thickness higher than 15 µm presents loss of catalyst effectiveness due to internal diffusional restrictions. Excellent stability in the catalytic tests was obtained after three regeneration-reaction cycles. Regeneration was carried out at 400 °C in air flow. Gold particle size distribution in the monolith washcoat, determined by TEM before and after reaction, was homogeneous with a medium size of around 5 nm. This is in agreement with the very good reproducibility and stability obtained in the catalytic tests. After calcination at 500 °C, some sintering and a heterogeneous distribution of metal particle size was observed, accompanied by a slight loss in catalyst activity. It is concluded that metallic monolith stirrer reactors are a promising application for selective lactose oxidation in liquid phase.

Structural and surface considerations on Mo/ZSM-5 systems for methane dehydroaromatization reaction

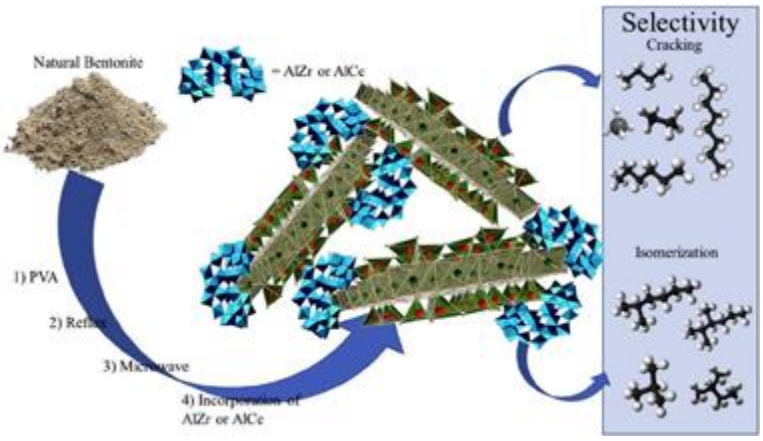
López-Martín, A; Caballero, A; Colon, G
Molecular Catalysis, **486** (2020) 110787
Mayo, 2020 | **DOI:** 10.1016/j.mcat.2020.110787



We have prepared a series of Mo/ZSM-5 systems by impregnation method with different metal loading. The optimum performance has been attained for 4% metal loading, yielding to ca. 2 $mmol_{benzene}/g_{cat}$ at the end of the reaction. The obtained catalysts were widely structural and surface characterized. As Mo content increases, the surface feature of the support is affected specially its mesoporosity. It has been stated the enormous complexity of Mo species present in the studied system. In situ characterization by XPS reveals different reduction and carburization behaviour depending on the Mo content.

Potentialization of bentonite properties as support in acid catalysts

Amaya, J; Bobadilla, L; Azancot, L; Centeno, M; Moreno, S; Molina, R
Materials Research Bulletin, **123** (2020) 110728
Marzo, 2020 | **DOI:** 10.1016/j.materresbull.2019.110728

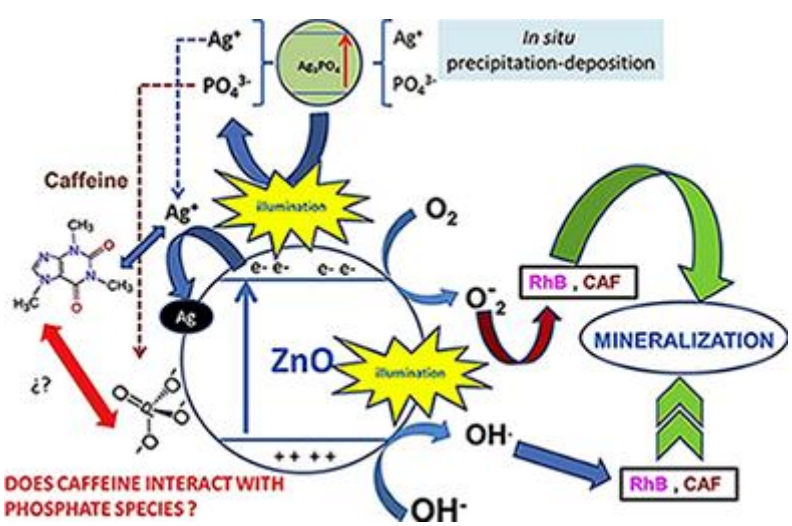


Enhancement of the main physicochemical properties of a natural bentonite was carried out by means of modifications using surfactant, reflux, microwave treatment and, subsequently, the incorporation of AlZr and AlCe species. The evolution of the main changes in each modification stage was evaluated by means of X-ray diffraction, N_2 sortometry, scanning microscopy (SEM), NH_3-TPD, NH_3-DRIFTS and CO adsorption at low temperature. For the evaluation of the catalytic behavior, the dehydration-dehydrogenation reactions of 2-propanol and hydro-conversion of decane were used; both of which generate, in addition, information regarding the acidic properties of the materials. The correlation of the number, type and acid strength with the catalytic behavior, allowed establishing the effect produced by both the delamination method and the nature of the incorporated cation. This generated tools that allow controlling the physicochemical properties,

and more specifically, the enhancement of the acidity of new supports based on this type of natural clay mineral.

Hybrid ZnO/Ag_3PO_4 photocatalysts, with low and high phosphate molar percentages

Martín-Gómez, A.N.; Navío, J.A.; Jaramillo-Páeza, C.; Sánchez-Cid, P.; Hidalgo, M.C.
Journal of Photochemistry and Photobiology A: Chemistry, **388** (2020) 112196
Febrero, 2020 | **DOI:** 10.1016/j.jphotochem.2019.112196

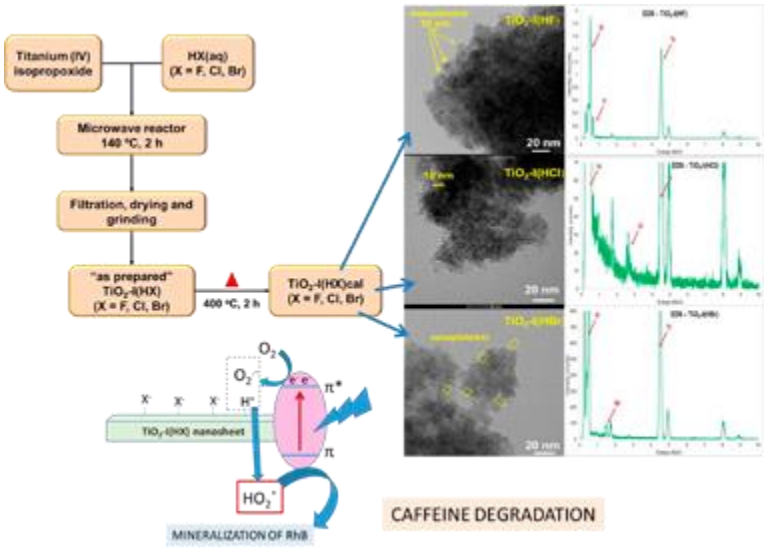


In this work, a previously optimized synthesized ZnO photocatalyst was modified with different molar percentages of Ag_3PO_4 through a facile in situ precipitation–deposition method and then characterized by different techniques (XRD, XRF, BET, UV–vis DRS, SEM, TEM and XPS). The incorporation of Ag_3PO_4produces important changes in the light absorption properties with a significant absorbance in the visible region observed for ZnO modified with different amounts of Ag_3PO_4; the optical absorption intensity in the visible region of the coupled ZnO/Ag_3PO_4 increases as the molar percentages of Ag_3PO_4 increases, evidencing a clear dependence on the content of Ag_3PO_4. However, this work shows that the incorporation of Ag_3PO_4 in almost all cases reduces the photocatalytic capacity of ZnO, except when it is used in a specific percentage of 10 % and only being more active against rhodamine B and not on the Caffeine. SEM images and elemental mapping indicate that Ag_3PO_4 disperses very well in the ZnO particles, exhibiting an almost homogeneous distribution, showing zones with cumulus of Ag_3PO_4 (rich in P-Ag) in contact with ZnO-zones (rich in Zn). All the prepared photocatalysts were tested in the photocatalytic degradation of rhodamine B as a dye, and caffeine as a toxic and persistent emerging compound under UV and visible light illumination. It is reported that not only the ZnO:Ag_3PO_4 ratio is an important factor that influences the photocatalytic process of substrate degradation, but also the nature of the substrate has an important influence on the photocatalytic behavior of the materials under both UV and visible illumination. Thus, pristine Ag_3PO_4 showed high photocatalytic degradation for rhodamine B, while for caffeine negligible photocatalytic degradation was found in both the UV and visible regions. The thermal- and photo-stability of the coupled system was also studied. At least, for rhodamine B no loss of photocatalytic activity has been observed after five recycles although the mineralization degree progressively diminished along the recycles.

Microwave-assisted sol-gel synthesis of TiO_2 in the presence of halogenhydric acids. Characterization and photocatalytic activity

Puga, F; Navío, J.A.; Jaramillo-Páez, C.; Sánchez-Cid, P.; Hidalgo, M.C.

Journal of Photochemistry and Photobiology A: Chemistry, **394** (2020) 112457
Mayo, 2020 | **DOI:** 10.1016/j.jphotochem.2020.112457



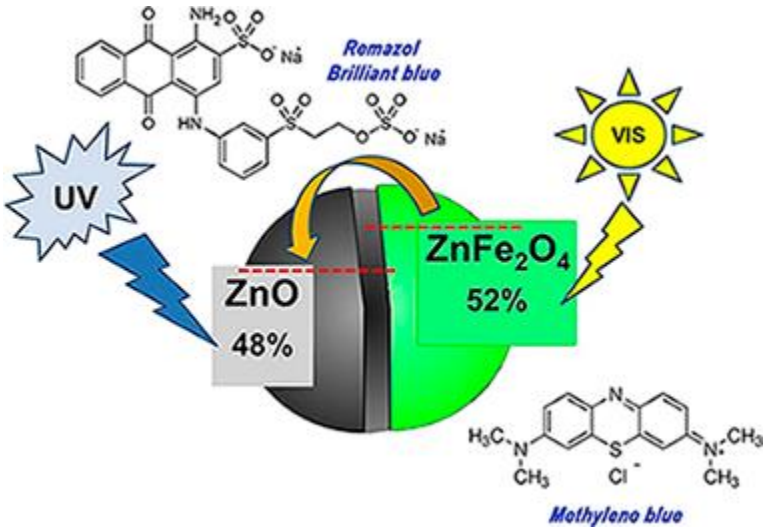
The synthesis of mesoporous TiO_2 nanosheets is reported using Ti(IV) Isopropoxide as Ti(IV) precursor. A sol-gel process combined with microwave activation is used. Three different halogenhydric acids (HX), were used to peptise the sol: HF(ac), HCl (ac) and HBr (ac). The three obtained TiO_2-I(HX) samples were characterized by XRD, XRF, N_2-adsorption, SEM, TEM, DRS and XPS. The three synthesized samples have high values of specific surfaces (between 100 m^2/g and 200 m^2/g) and similar band gap values (3.2–3.3 eV). The analysis of the surface composition by XPS confirms the presence of the halogenated species (F, Cl or Br) on the surface of each ones of the samples. The nanometric size (ca 5 nm) of the particles for each of the three samples was confirmed by XRD and by TEM. On the other hand, the nature of the halogenated acid used plays a role in the composition of the phases. While the TiO_2-I (HF) sample was 100 % anatase, the other samples turned out to be biphasic, showing anatase/rutile in the TiO_2-I(HCl) sample and anatase/brookite in the TiO_2-I(HBr) sample. The samples were tested under two illumination conditions (UV and visible light) using rhodamine B and caffeine. The indirect role of the halide agent on the photocatalytic activities thereof is discussed.

Preparation of $ZnFe_2O_4$/ZnO composite: Effect of operational parameters for photocatalytic degradation of dyes under UV and visible illumination

Zouhier, M.; Tanji, K.; Navio, J.A.; Hidalgo, M.C.; Jaramillo-Páez, C.; Kherbeche, A.

Journal of Photochemistry and Photobiology A: Chemistry, **390** (2020) 112457
Marzo, 2020 | **DOI:** 10.1016/j.jphotochem.2019.112305

An $ZnFe_2O_4$/ZnO composite catalyst was prepared by solution combustion method. In this study, one nominal molar percentage of iron was used in the synthesis, corresponding to 20 % molar relative to ZnO. The samples were characterized by X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), X-ray Fluorescence (XRF), Scanning Electronic Microscopy (SEM), Transmission Electronic Microscopy (TEM) and Ultraviolet-visible (UV–vis) diffuse spectroscopy (DRS). The photocatalytic activities of the catalysts were investigated based on the degradation of two dyes, methylene blue (MB) and remazol brilliant blue (RBB), in aqueous solution under both UV and visible light illumination respectively. It was found that the composite had a good photocatalytic activity at basic pH by using 1 g/L of catalyst under UV illumination for both MB and RBB. Under visible illumination, while pristine ZnO showed no activity, the composite



exhibited an excellent visible efficiency, reaching up to an 80 % conversion of the initial dye concentrations in 2 h. The enhancement of the visible photocatalytic activity of Fe/ZnO sample with respect to pristine ZnO is attributed to the formation of $ZnFe_2O_4$ coupled with ZnO, having a narrow band gap value that contributes to the absorption of visible photons with an improved separation path for the photo-generated carriers.

Modulation of the acidity of a vermiculite and its potential use as a catalytic support

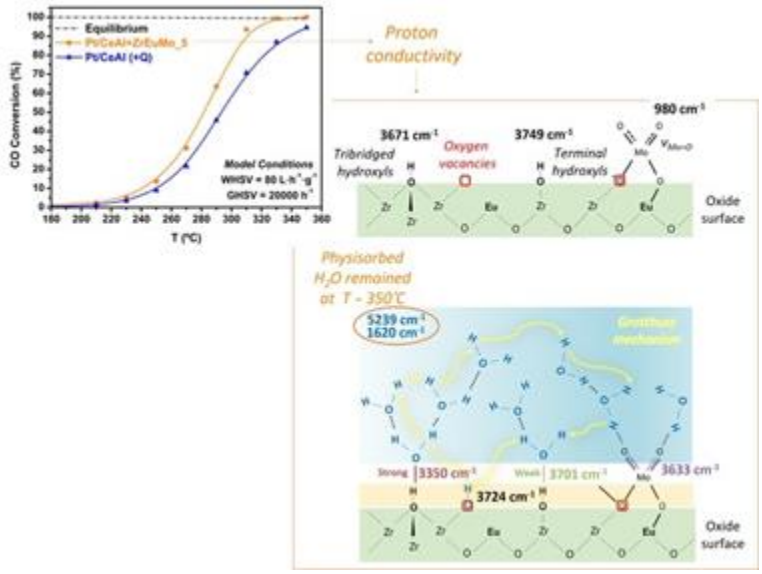
Amaya, J; Bobadilla, L; Azancot, L; Centeno, M; Moreno, S; Molina, R
Journal of Materials Science, **55** (2020) 6482-6501
Febrero, 2020 | **DOI:** 10.1007/s10853-020-04445-5

The modulation and characterization of the acidity of a vermiculite were carried out, which was modified by delamination by means of hydrothermal and acid treatments with the subsequent incorporation of AlZr and AlCe species to modulate the acidity. The effect of these species was evaluated regarding the structural (XRD, XPS and IR), textural (N_2 sortometry) and acidity properties (NH_3-TPD, NH_3-DRIFTS and CO adsorption at low temperature). The catalytic performance was studied in the dehydration-dehydrogenation reactions of 2-propanol and the hydroconversion of decane, which generate important information about the acidity properties such as the type, number and strength of acidic sites. The correlation between the number, type and acid strength with the catalytic behavior allowed to establish the important effect regarding the nature of the mineral, its method of delamination and the nature of the incorporated cation,

thus generating tools for controlled processes for the potentiation of the acidity of new supports from raw vermiculite.

Elucidation of Water Promoter Effect of Proton Conductor in WGS Reaction over Pt-Based Catalyst: An Operando DRIFTS Study

Jurado, L; García-Moncada, N; Bobadilla, LF; Romero-Sarria, F; Odriozola, JA
Catalysts, **10** (2020) 841
Agosto, 2020 | **DOI:** 10.3390/catal10080841



A conventional $Pt/CeO_2/Al_2O_3$ catalyst physically mixed with an ionic conductor (Mo- or Eu-doped ZrO_2) was tested at high space velocity (20,000 h^{-1} and 80 L $h^{-1}g_{cat}^{-1}$) under model conditions (only with CO and H_2O) and industrial conditions, with a realistic feed. The promoted system with the ionic conductor physically mixed showed better catalytic activity associated with better water dissociation and mobility, considered as a rate-determining step. The water activation was assessed by operando diffuse reflectance infrared fourier transformed spectroscopy (DRIFTS) studies under reaction conditions and the Mo-containing ionic conductor exhibited the presence of both dissociated (3724 cm^{-1}) and physisorbed (5239 cm^{-1}) water on the Eu-doped ZrO_2 solid solution, which supports the appearance of proton conductivity by Grotthuss mechanism. Moreover, the band at 3633 cm^{-1}ascribed to hydrated Mo oxide, which increases with the temperature, explains the increase of catalytic activity when the physical mixture was used in a water gas shift (WGS) reaction.

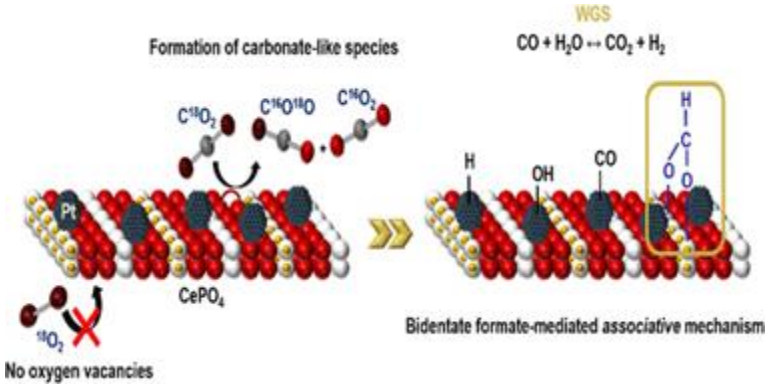
Catalytic Performance of Bulk and Al_2O_3-Supported Molybdenum Oxide for the Production of Biodiesel from Oil with High Free Fatty Acids Content

Navajas, A; Reyero, I; Jiménez-Barrera, E; Romero-Sarria, F; Llorca, J; Gandia, LM
Catalysts, **10** (2020) 158
Febrero, 2020 | **DOI:** 10.3390/catal10020158

Non-edible vegetable oils are characterized by high contents of free fatty acids (FFAs) that prevent from using the conventional basic catalysts for the production of biodiesel. In this work, solid acid catalysts are used for the simultaneous esterification and transesterification with methanol of the FFAs and triglycerides contained in sunflower oil acidified with oleic acid. Molybdenum oxide (MoO_3), which has been seldom considered as a catalyst for the production of biodiesel, was used in bulk and alumina-supported forms. Results showed that bulk MoO_3 is very active for both transesterification and esterification reactions, but it suffered from severe molybdenum leaching in the reaction medium. When supported on Al_2O_3, the MoO_3 performance improved in terms of active phase utilization and stability though molybdenum leaching remained significant. The improvement of catalytic performance was ascribed to the establishment of MoO_3-Al_2O_3 interactions that favored the anchorage of molybdenum to the support and the formation of new strong acidic centers, although this effect was offset by a decrease of specific surface area. It is concluded that the development of stable catalysts based on MoO_3 offers an attractive route for the valorization of oils with high FFAs content.

Evaluation of the Oxygen Mobility in $CePO_4$-Supported Catalysts: Mechanistic Implications on the Water-Gas Shift Reaction

Navarro-Jaen, S; Bobadilla, LF; Romero-Sarria, F; Laguna, OH; Bion, N; Odriozola, JA
Journal of Physical Chemistry C, **124** (2020) 16391-16401
Julio, 2020 | **DOI:** 10.1021/acs.jpcc.0c03649



The hexagonal and monoclinic phases of $CePO_4$ have been demonstrated to be excellent catalytic supports for Pt-based water-gas shift (WGS) catalysts. Consequently, the elucidation of the WGS reaction mechanism in these materials constitutes a fundamental aspect in order to explain their catalytic behavior. Because the observed WGS reaction path is closely related to the absence or presence of oxygen vacancies in the support, the study of the oxygen mobility in these solids constitutes a key factor for the understanding of the structure of the materials and its influence on the reaction mechanism. In this study, the oxygen mobility in $CePO_4$ supports and the corresponding Pt catalysts has been evaluated by means of isotopic exchange experiments using $^{18}O_2$ and $C^{18}O_2$ as probe molecules. Results demonstrate that the evaluated solids present a low exchange activity when $^{18}O_2$ is used, indicating the absence of oxygen vacancies in these solids, thus suggesting a poor influence of the WGS redox mechanism. On the contrary, a high oxygen exchange activity is observed using $C^{18}O_2$, demonstrating that the exchange in these materials takes place through the formation of carbonate-like intermediates,

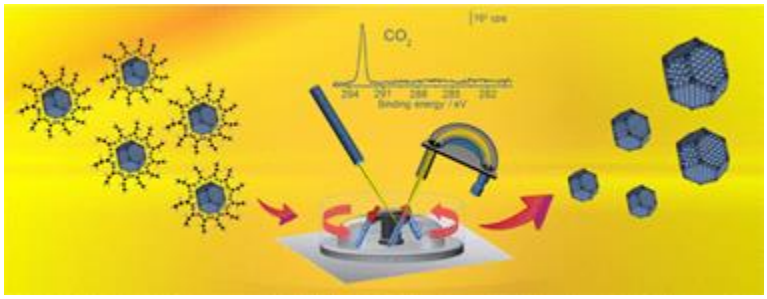
thus suggesting the associative mechanism of the WGS reaction as the preferred path in these solids. Operando diffuse reflectance infrared spectroscopy experiments under WGS reaction conditions confirm these results, proving that the WGS reaction in the studied materials takes place through a formate-mediated associative mechanism.

Free-Carbon Surface for PtCu Nanoparticles: An In Situ Near Ambient Pressure X-ray Photoelectron Spectroscopy Study

Castillo, R; Navarro-Jaen, S; Romero-Sarria, F; Pérez-Dieste, V; Escudero, C; Centeno, MA; Daturi, M; Odriozola, JA

Journal of Physical Chemistry C, **124** (2020) 19046-19056

Septiembre, 2020 | **DOI:** 10.1021/acs.jpcc.0c04713



Usually, nanoparticle synthesis methodologies require the use of organic molecules (capping agent, solvent molecules, etc.), which results in carbon deposits on the nanoparticle surface. These residues modify the surface properties mainly affecting the catalytic behavior. In this work, unsupported poly(vinylpyrrolidone) (PVP)-stabilized PtCu (1:3 molar ratio) bimetallic alloy nanoparticles were synthetized and characterized. An alternative surface cleaning method has been designed, which successfully removes the presence of organic fragments. To address this key issue, we have combined a first nanoparticle washing step with a near ambient pressure X-ray photoelectron spectroscopy (NAPXPS) study in order to obtain a clean active site and the total understanding of the carbon elimination mechanism. The dynamic evolution of the surface organic species composition under different gas mixtures at 750 mTorr and 350 ^{o}C has been studied, and only under CO_2 exposure, NAPXPS analysis revealed a total availability of the active site by the removal of the organic nanoparticle coating.

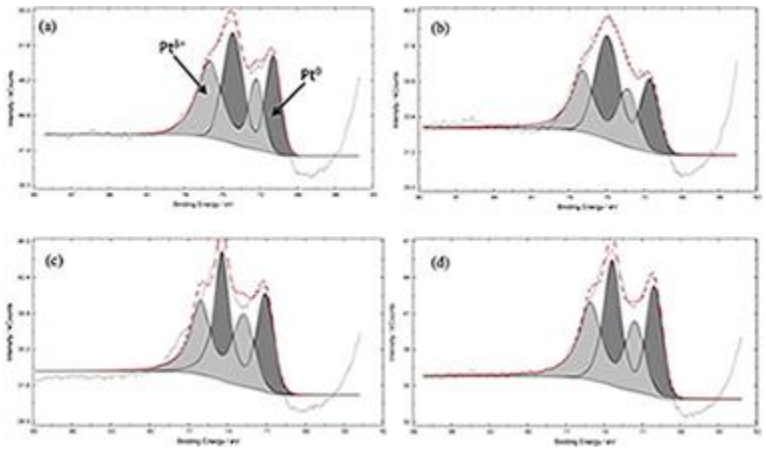
Pt–TiO_2–Nb_2O_5 heterojunction as effective photocatalyst for the degradation of diclofenac and ketoprofen

Sacco, O.l; Murcia, J.J.; Lara, A.E.; Hernández-Laverde, M.; Rojas, H.; Navío, J.A.; Hidalgo, M.C.; Vaiano, V.

Materials Science in Semiconductor Processing, **107** (2020) 104839

Marzo, 2020 | **DOI:** 10.1016/j.mssp.2019.104839

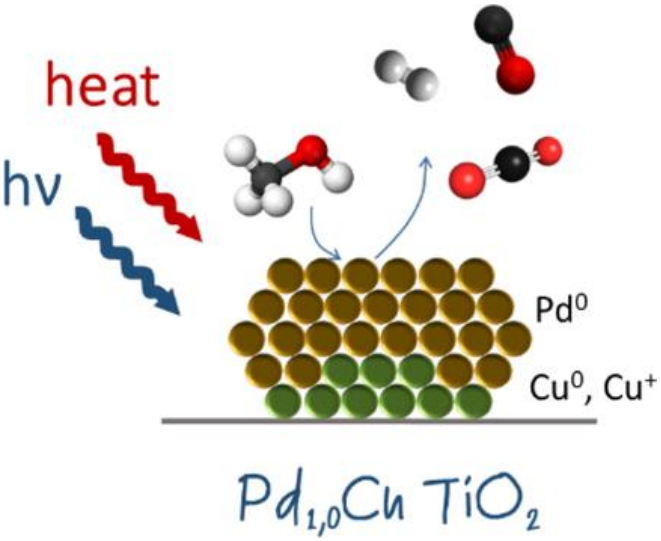
Pt–TiO_2–Nb_2O_5 heterojunction was synthetized and studied for the photocatalytic removal of diclofenac (DCF) and ketoprofen (KTF) under UV light irradiation. The physical-chemical properties of the prepared catalysts were analysed by different characterization techniques revealing that the lowest platinum nanoparticle size and the better metal distribution was



observed in Pt–TiO_2–Nb_2O_5 sample. The Pt–TiO_2–Nb_2O_5 heterojunction possessed the best photocatalytic activity toward both the photodegradation and mineralization of the two selected pollutants. The optimal photocatalyst showed a DCF and KTF mineralization rate of 0.0555 and 0.0746 min−1, respectively, which were higher than those of Pt–TiO_2 (0.0321 min−1 for DCF and 0.0597 min−1 for KTF). The experiments driven to analyse the effects of free radical capture showed that ·OH, $\cdot O_2^-$ and h^+ have a primary role in reactive during the photocatalytic reaction. The improved photocatalytic performances of the Pt– TiO_2–Nb_2O_5 heterojunction could be argue by a direct Z-scheme mechanism in which the Pt^0 nanoparticles could act as a bridge between TiO_2 and Nb_2O_5, improving the electron-hole separation and, ultimately, enhancing the photocatalytic removal rate of both DCF and KTF.

Thermo-Photocatalytic Methanol Reforming for Hydrogen Production over a CuPd-TiO_2 Catalyst

López-Martín, A; Platero, F; Caballero, A; Colon, G
ChemPhotoChem, **4** (2020) 630-637
Mayo, 2020 | **DOI:** 10.1002/cptc.202000010



A bimetallic CuPd/TiO_2 system has been prepared by a two-step synthesis and was used for a methanol steam photoreforming reaction. By sequential deposition, palladium is deposited over copper nanoclusters through a galvanic replacement process. Hydrogen production by steam reforming from methanol was achieved by both thermo-photocatalytic and photocatalytic processes. It appears that H_2 production on the bimetallic system is notably higher than the Pd monometallic reference. Moreover this difference in the catalytic performance could be related to the higher CO evolution observed for the monometallic $Pd_{1.0}$ TiO_2 system which is partially inhibited in the bimetallic catalyst. In addition, an important thermal effect can be envisaged in all cases. Nevertheless, this improved effect in the thermo-photocatalytic process is accompanied by a remarkable CO evolution and SMSI effect (important strong metal-support interactions) that hindered the efficiency as

temperature increases. On this basis, optimal operational conditions for H_2 production are obtained for thermo-photocatalytic reforming at 100 °C, for which the synergetic effect is higher with lower CO production (H_2/CO=4).

Evaluation of Au–ZnO, ZnO/Ag_2CO_3 and Ag–TiO_2 as Photocatalyst for Wastewater Treatment

Murcia, J.J.; Hernández, J.S.;Rojas, H.; Moreno-Cascante, J.; Sánchez-Cid, P.; Hidalgo, M.C.; Navío, J.A.; Jaramillo-Páez, C.
Topics in Catalysis, **4** (2020) 630-637
Febrero, 2020 | **DOI:** 10.1007/s11244-020-01232-z

In this work series of photocatalysts based on ZnO modified by Au and Ag_2CO_3 addition and Ag–TiO_2 materials were synthesized and evaluated in the treatment of handicrafts factories wastewater and water samples taken from a highly polluted river. In general, it was found that ZnO series were more effective in the bacteria elimination than the commonly used TiO_2 semiconductor. It was also observed that the metal (Au, Ag) or silver carbonate addition significantly increases the photocatalytic activity of ZnO and TiO_2. It was determined that the content of the metal or carbonate added is an important factor to take into account in order to obtain suitable efficiency in the photocatalytic process, so, for example in the case of the river water samples the increase of Ag_2CO_3 content from 1 to 5%, had a detrimental effect over the bacteria elimination. The optimal conditions for dyes photodegradation and bacteria elimination were found by using a response surface study and the Au–ZnO (1%) photocatalyst. From this study it was determined that even after recycling this material leads to obtain a removal percentage of these pollutants over than 94%.

LIBROS Y OTRAS PUBLICACIONES / BOOKS AND OTHER PUBLICATIONS

Preface
T.R. Reina, J.A. Odriozola
Heterogeneous Catalysis for Energy Applications, Royal Society of Chemsitry (RSC), VII-VIII
ISBN: 978-1-78801-718-3
DOI: 10.1039/9781788019576

Catalytic Technologies for Clean Hydrogen Production
S. Navarro-Jaén, C. Megías-Sayago, S. Ivanova, J.A. Odriozola
Heterogeneous Catalysis for Energy Applications, Royal Society of Chemsitry (RSC), pp 116-149
ISBN: 978-1-78801-718-3
DOI: 10.1039/9781788019576

Catalytic Technologies for the Production of Liquid Transportation Fuels from Biomass
L. Azancot, L.F. Bobadilla, F. Romero-Sarria, J.A. Odriozola
Heterogeneous Catalysis for Energy Applications, Royal Society of Chemsitry (RSC), pp 202-234
ISBN: 978-1-78801-718-3
DOI: 10.1039/9781788019576

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

4th International Conference on Catalysis and Chemical Engineering (CCE 2020)
24 – 26 febrero [Los Angeles, Estados Unidos de América]

Photoassisted degradation of Rhodamine B and Caffeine by synthesis of ZnO/hydroxyapatite nanoparticles
K. Tanji, J.A. Navío, A. N. Martín-Gómez, M.C. Hidalgo, C. Jaramillo-Páez, J. Naja, A. Chaqroune, A. Kherbeche
Comunicación Oral

XXVII Congreso Iberoamericano de Catálisis CICAT2020
26 – 28 octubre [México]

Novel technique for controlling reactor temperature in exothermic reactions
J.A. Odriozola; Simon Yunes; Patrick Wommack; Carmen Lara
Conferencia Invitada

Estudio comprensivo del efecto promotor del potasio en la reacción de reformado de biogás
Lola Azancot; Luis F. Bobadilla; M.A. Centeno; J.A. Odriozola
Comunicación Oral

Hidrogenación del dióxido de carbono mediante la reacción de Sabatier empleando micromonolitos metálicos
J.C. Navarro; N. García-Moncada; M.A. Centeno; O.H. Laguna; J.A. Odriozola
Comunicación Oral

CONGRESOS Y REUNIONES NACIONALES / NATIONAL CONGRESSES AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

XVI Jornadas Científicas del Instituto Universitario de Materiales
23 – 24 enero [Alicante, España]

Soportes de tipo fosfato en el diseño de catalizadores para la reacción de Water Gas Shift
M.A. Centeno
Conferencia Invitada

FORMACION / TRAINING

TESIS DOCTORALES/ DOCTOR DEGREE THESIS

Título:	**Préparation, caractérisation et réactivité des nanoparticules d'or supportées sur argile et sur supports oxides**
Autor:	Meriem Chenouf
Directores:	Fatima Ammari, Svetlana Ivanova y José Antonio Odriozola Gordón
Centro:	Université Ferhat Abbas - SETIF1 (Argelia) y Universidad de Sevilla
Fecha:	6 de febrero de 2020

FORMACIÓN DE GRADUADOS / MASTER DEGREE THESIS

Título:	**Biopolímeros derivados de productos naturales: Sustratos potenciales para la immobilización de biocatalizadores**
Autor:	Nicolás Sotomayor López
Directores:	Luis Francisco Bobadilla Baladrón y José María Martínez Blanes
Grado:	Trabajo Fin de Grado
Centro:	Universidad de Sevilla
Fecha Defensa:	10 julio 2020

Título:	**Producción de HMF a partir de glucosa sobre catalizadores basados en carbón**
Autor:	Beatriz Lara Bolaños
Directores:	Miguel Ángel Centeno Gallego y María Isabel Domínguez Leal
Grado:	Trabajo Fin de Grado
Centro:	Universidad de Sevilla
Fecha Defensa:	15 julio 2020

Título:	**Zeolitas con aplicaciones catalíticas: intercambio catiónico y sitios activos**
Autor:	Guillermo Torres Semper
Directores:	Luis Francisco Bobadilla Baladrón y Francisca Romero Sarria
Grado:	Trabajo Fin de Grado
Centro:	Universidad de Sevilla
Fecha Defensa:	15 julio 2020

Título:	**Aplicación de métodos high-throughput para el desarrollo de nuevos fármacos**
Autor:	MariLuz Martín Alonso
Directores:	Luis Francisco Bobadilla Baladrón
Grado:	Trabajo Fin de Grado
Centro:	Universidad de Sevilla
Fecha Defensa:	18 septiembre 2020

Título:	**1D and 3D nanomaterial fabrication and surface modification for developing Piezoelectric and Triboelectric Nanogenerators**
Autor:	Xabier García Casas
Directores:	Ángel Barranco Quero, Ana Isabel Borrás Martos y Leidy Marcela Martínez Tejada
Grado:	Trabajo Fin Master
Centro:	Universidad de Sevilla
Fecha Defensa:	9 julio 2020

Título:	**Materiales zeolíticos como catalizadores en reacciones de interés medioambiental**
Autor:	Ligia Amelia Luque Álvarez
Directores:	Luis Francisco Bobadilla Baladrón y Francisca Romero Sarria
Grado:	Trabajo Fin de Master
Centro:	Universidad de Sevilla
Año Académico:	23 julio 2020

Título:	**Catalizadores basados en materiales híbridos C/TiO_2**
Autor:	Beatriz María López Rodríguez
Directores:	Leidy Marcela Martínez Tejada y María Isabel Domínguez Leal
Grado:	Trabajo Fin de Master
Centro:	Universidad de Sevilla
Año Académico:	18 septiembre 2020

Título:	**Propiedades estructurales de catalizadores basados en espinelas tipo ferrita para la reacción de hidrogenación de CO_2**
Autor:	Carlos Hurtado Torres
Directores:	José Antonio Odriozola Gordón y Luis Francisco Bobadilla Baladrón
Grado:	Trabajo Fin de Master
Centro:	Universidad de Sevilla
Año Académico:	18 septiembre 2020

DOCENCIA / TEACHING

Investigadores de esta unidad participan en el Máster en Ciencia y Tecnología de Nuevos Materiales (página 235)

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

GRUPO DE INVESTIGACIÓN

Materiales de Diseño para la Energía y Medioambiente

Designed Materials for the Energy and Environment [642007]

PERSONAL / PERSONNEL

Catedráticos

Dr. Miguel Ángel Castro Arroyo
Dr. Manuel Jiménez Melendo
Dra. Pilar Malet Maenner
Dr. Julián Martínez Fernández

Científicos Titulares

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

Profesores Titulares

Dr. Alfonso Bravo León
Dr. Joaquin Ramírez Rico

Doctores Contratados

Dra. Esperanza Pavón González

PROYECTOS DE INVESTIGACIÓN / RESEARCH PROJECTS

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

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

Código/Code:	MAT2016-76526-R
Periodo/Period:	30-12-2016 / 31-12-2020
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	60.500 €
Investigador responsable/Research head:	Joaquin Ramírez Rico /Ricado Chacartegui
Componentes/Research group:	Manuel Jiménez Melendo, Julián Martínez Fernández

RESUMEN / ABSTRACT

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

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

El proyecto se basa en la innovadora integración de material biocerámico microporoso en las cámaras de combustión de calderas de biomasa de modo que actúen con diferentes funciones: combustor microporoso, filtro de partículas y recuperador de calor. Estas funcionalidades pueden ser simultáneas, en función de la región del flujo en que se encuentren y el rango de temperaturas de esa región. Este material biocerámico es desarrollado a partir de precursores vegetales para obtener elementos de Carburo de Silicio (SiC). Para ello se usan materiales locales sin tratar, produciendo elementos hechos a medida con propiedades microestructurales adecuadas para trabajar con altas temperaturas. Así, productos con geometrías complejas pueden ser obtenidos con relativamente bajo coste comparados con otros materiales con características mecánicas y químicas similares. La integración de componentes basados en estos materiales posibilita nuevos diseños de calderas de biomasa con un alto control de la combustión, las temperaturas y la emisión de partículas. El nuevo diseño

evita la sinterización y fusión de las cenizas, actuando en la formación y evolución de contaminantes, inhibiendo los mecanismos de producción de dioxinas y activando la completa oxidación del monóxido de carbono (CO) y soots. El nuevo concepto permitirá la operación con una importante variedad de mezclas biomasa/ residuos agrícolas con bajas emisiones aun cuando el combustible presente un alto contenido de cenizas, resolviendo el principal reto para el desarrollo del uso residuos agrícolas en calderas de biomasa (especialmente las de menor tamaño). El desarrollo de esta tecnología permitirá ampliar los recursos de la Unión Europea para calefacción de uso doméstico. En la actualidad este uso supone un 30% del consumo energético total en la misma. La propuesta incluye el estudio de los procesos básicos de combustión, flujos, fabricación a medida de las matrices de materiales biocerámicos, así como estudio y desarrollo de prototipos de componentes y del sistema final. Estos serán estudiados a nivel de ensayos de laboratorio con residuos agrícolas, forestales y de la industria olivarera.

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

OTROS PROYECTOS / OTHER PROJECTS

Aspectos genéticos y biofísicos de la formación de la cutícula del fruto de tomate

Código/Code:	RTI2018-094277-B-C22
Periodo/Period:	01-01-2019 / 31-12-2021
Organismo Financiador/Financial source:	Ministerio de Ciencia, Innovacion y Universidades
Importe total/Total amount:	193.600 €
Investigador responsable/Research head:	Eva María Domínguez Carmona (IHSM) y Rafael Fernández Muñoz (IHSM)
Componentes/Research group:	José Jesús Benítez, Luz Divina Gómez Pulido (IHSM), Manuel León Camacho (IG)

Incentivo al Grupo de Investigación TEP-106

Tipo de Proyecto/Ayuda:	Ayudas a Consolidación de Grupos de la Junta de Andalucía
Código/Code:	2019/FQM-342
Periodo/Period:	01-01-2020 / 30-06-2021
Organismo Financiador/Financial source:	Junta de Andalucía
Investigador responsable/Research head:	Joaquín Ramírez Rico

CONVENIOS Y CONTRATOS / CONTRACTS AND AGREEMENTS

Development of a coating for fruits and vegetables derived from aleuritic acid

Periodo/Period:	26-12-2019 / 26-05-2021
Organismo Financiador/Financial source:	DECCO WORLDWIDE POST
Importe total/Total amount:	28.798 €
Investigador responsable/Research head:	José Jesús Benítez

PATENTES / PATENTS

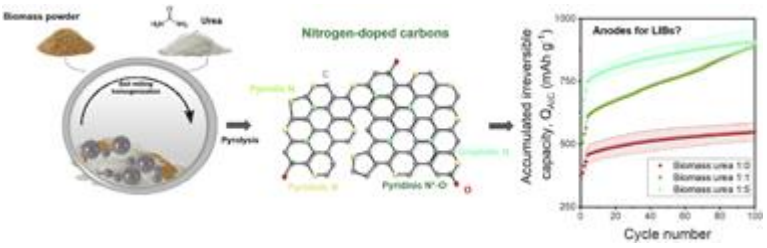
Sensor luminiscente para la monitorización de residuos radioactivos y uso del mismo

Inventores: Esperanza Pavón González, María Dolores Alba Carranza, María José García Jiménez
Tipo de Patente: Nacional
Número de Solicitud: 202020404
Fecha Solicitud: 7 de mayo de 2020
Entidad Titular: Universidad de Cantabria, Consejo Superior de Investigaciones Científicas

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

An electrochemical evaluation of nitrogen-doped carbons as anodes for lithium ion batteries

Gómez-Martín, A; Martínez-Fernández, J; Ruttert, M; Winter, M; Placke, T; Ramírez-Rico, J
Carbon, **164** (2020) 261-271
Agosto, 2020 | **DOI:** 10.1016/j.carbon.2020.04.003



New anode materials beyond graphite are needed to improve the performance of lithium ion batteries (LIBs). Chemical doping with nitrogen has emerged as a simple strategy for enhancing lithium storage in carbon-based anodes. While specific capacity and rate capability are improved by doping, little is known about other key electrochemical properties relevant to practical applications. This work presents a systematic evaluation of electrochemical characteristics of nitrogen-doped carbons derived from a biomass source and urea powder as anodes in LIB half- and full-cells. Results show that doped carbons suffer from a continuous loss in capacity upon cycling that is more severe for higher nitrogen contents. Nitrogen negatively impacts the voltage and energy efficiencies at low charge/discharge current densities. However, as the charge/discharge rate increases, the voltage and energy efficiencies of the doped carbons outperform the non-doped ones. We provide insights towards a fundamental understanding of the requirements needed for practical applications and reveal drawbacks to be overcome by novel doped carbon-based anode materials in LIB applications. With this work, we also want to encourage other researchers to evaluate electrochemical characteristics besides capacity and cycling stability which are mandatory to assess the practicality of novel materials.

Performance trends in wall-flow diesel particulate filters: Comparative analysis of their filtration efficiency and pressure drop

Orihuela, MP; Chacartegui, R; Gómez-Martín, A; Ramírez-Rico, J; Villanueva, JAB
Journal of Cleaner Production, **60** (2020) 12063
Julio, 2020 | **DOI:** 10.1016/j.jclepro.2020.120863

Soot and particulate emissions from the transport sector are a major concern worldwide, given their harmful effects on public health and the environment. On-road vehicles are the main contributing source to this kind of pollution. They are strictly regulated in many countries, with limitations on the number and concentration of released particles, and they must be equipped with particle abatement systems. Wall-flow particulate filters are the most popular and effective devices to reduce particulate emissions from diesel and gasoline vehicles. Diesel Particulate Filters (DPFs) have been a recurrent research topic since the last century. There are different research studies analysing different aspects of these systems, at different levels, using different methodologies and different approaches. Their results are not always comparable. This work

analyses the latest advances and trends in this technology by comparing two relevant performance parameters: their filtration efficiency and pressure drop. The findings of this study suggest that, in order to be competitive, upcoming DPFs should have filtration efficiencies above 80%, and pressure drops below 10 kPa, for space velocities of $1.5 \cdot 10^5\ h^{-1}$ or more at the clean state. They should reach similar to 100% efficiency after a short operation period, before the soot load reaches 0.2 g/L. Later, they should keep a low pressure drop for a longer time, with a reference of no more than 13 kPa for 6 g/L of soot load. Based on this analysis, this work proposes some test criteria and suggestions for the main parameters.

Sustainable, High-Barrier Polyaleuritate/Nanocellulose Biocomposites

Tedeschi, G; Guzman-Puyol, S; Ceseracciu, L; Benitez, JJ; Cataldi, P; Bissett, M; Heredia, A; Athanassiou, A; Heredia-Guerrero, JA

ACS Sustainable Chemistry & Engineering, **8** (2020) 10682-10690

Julio, 2020 | **DOI:** 10.1021/acssuschemeng.0c00909

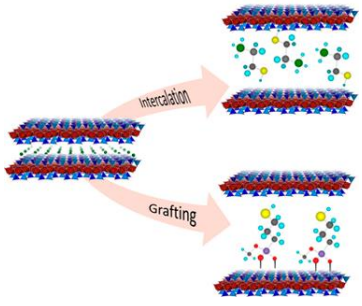
Free-standing and flexible biocomposite films formed by a polyaleuritate matrix and nanocellulose fillers (i.e., cellulose nanofibrils) have been fabricated by a sustainable process. For this, 9,10,16-trihydroxyhexadecanoic (aleuritic) acid from shellac and nanocellulose were blended at different ratios in water through a sonication process. Polymerization of the polyhydroxylated fatty acid into polyaleuritate was induced by a solvent-free, melting polycondensation reaction in the oven. These biocomposites were characterized to evaluate their chemical (by ATR-FTIR spectroscopy) and physical (e.g., density, thermal stability, rigidity, gas permeability, surface energy, etc.) properties. The compatibility between the polyester matrix and the polysaccharide fillers was excellent due to the interaction by H bonds of the polar groups of both components. The addition of nanocellulose increased all determined mechanical parameters as well as the wettability and the barrier properties, while the thermal stability and the water uptake were determined by the polyaleuritate matrix. The physical properties of these biocomposites were compared to those of petroleum-based plastics and bio-based polymers, indicating that the developed materials can represent a sustainable alternative for different applications such as packaging.

An insight on the design of mercapto functionalized swelling brittle micas

Osuna, FJ; Pavón, E; Alba, MD

Journal of Colloid and Interface Science, **561** (2020) 533-541

Marzo, 2020 | **DOI:** 10.1016/j.jcis.2019.11.028



Surface modification of natural clay minerals with reagents containing metal chelating groups has great environmental value. The functionalization by adsorption or grafting guarantees a durable immobilization of the reactive organic groups, preventing their leaching when they are used in liquid media. The aim of this research was the designed mercapto functionalization of swelling brittle micas, Na-Mn, thorough both chemical and physical mechanisms. Na-Mn were functionalized with 2-mercaptoethylammonium (MEA), 2,3-

dimercapto-1-propanol (BAL) and (3-mercaptopropyl)trimethoxysilane (MPTMS). The thiol concentration on swelling brittle micas is higher than the observed value for others adsorbents. The cation exchange reaction with MEA and one-step grafting with MPTMS in acid medium are the most efficient mercapto functionalization mechanism.

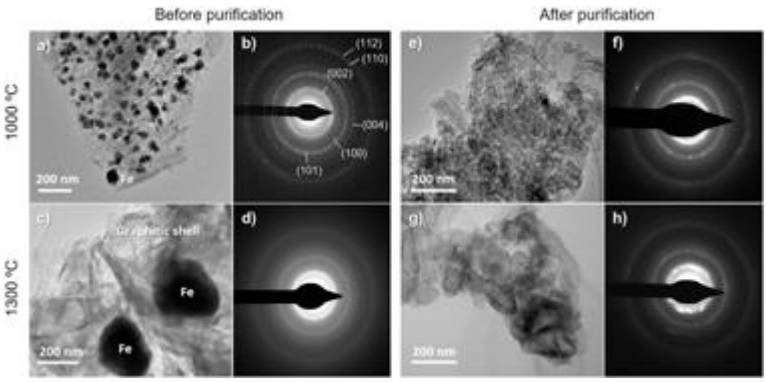
New biomorphic filters to face upcoming particulate emissions policies: A review of the FIL-BIO-DIESEL project

Orihuela, MP; Chacartegui, R; Martínez-Fernández, J
Energy, **201** (2020) 117577
Junio, 2020 | **DOI:** 10.1016/j.energy.2020.117577

With a high number of diesel vehicles worldwide, particulate emission control is an urgent issue with a global impact, from the health of citizens to commercial future of this technology in some transport segments. Particulate filters are widely used in automotive engines to comply emissions regulations, but current technologies have room for improvement as they add additional backpressure in the exhaust system, and efficient on-board regeneration process is challenging.
The Fil-Bio-Diesel Project is a R&D initiative to improve current particle filtration systems, based on the development of novel biomorphic substrates. By replicating the biologic tissue of a wood precursor, a biomorphic silicon carbide with hierarchic orthotropic microstructure can be produced. The porosity, the pore size, and pore orientation of this bioceramic material can be tailored through the selection of a suitable precursor, widening the initially narrow relationship between filtration efficiency and pressure drop that characterizes granular ceramic materials. In this paper the methodology and main results of the Fil-Bio-Diesel Project are presented. This work shows the peculiar advantages of biomorphic silicon carbide through several experimental studies. The results show the potential of this novel filter substrate to be used in future particulate abatement systems.

Binder-free supercapacitor electrodes: Optimization of monolithic graphitized carbons by reflux acid treatment

Gómez-Martín, A; Gutiérrez-Pardo, A; Martínez-Fernández, J; Ramírez-Rico, J
Fuel Processing Technology, **199** (2020) 106279
Marzo, 2020 | **DOI:** 10.1016/j.fuproc.2019.106279



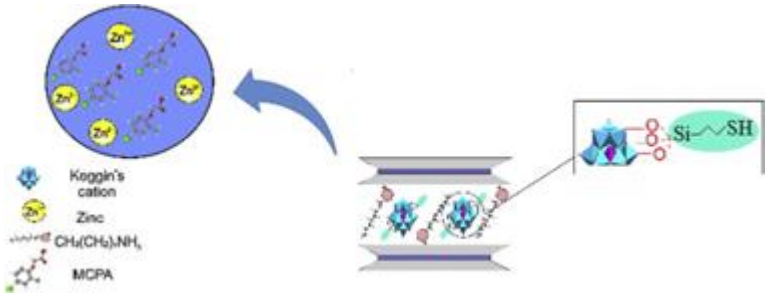
The rational design of electrodes mimicking the cellular structure of natural bio-resources has been a matter of increasing interest for applications in energy storage. Due to their anisotropic and hierarchical porosity, monolithic carbon materials from natural wood precursors are appealing as

electrodes for supercapacitor applications due to their interconnected channels, relatively low cost and environmentally friendly synthesis process. In this work, a liquid-phase oxidative treatment with refluxing nitric acid at 100 ºC for 8 h was performed to enhance the surface properties of beech-derived graphitized carbons treated with an iron catalyst. Microstructural, textural and surface investigations revealed that this strategy was successful in removing amorphous carbon and in functionalizing their surfaces. The crystallinity, accessible surface area, micropore volume and surface functionality of beech-derived carbons were increased upon the reflux treatment. The resulting porous carbon materials were evaluated as binderless monolithic electrodes for supercapacitors applications in aqueous KOH electrolyte. A maximum specific capacitance of 179 $F \cdot g^{-1}$ and a volumetric capacitance of 89 $F \cdot cm^{-3}$ in galvanostatic charge/discharge experiments were reached. Monolithic electrodes exhibited good cycling stability, with a capacitance retention over 95% after 10,000 cycles.

Multiple pollutants removal by functionalized heterostructures based on Na-2-Mica

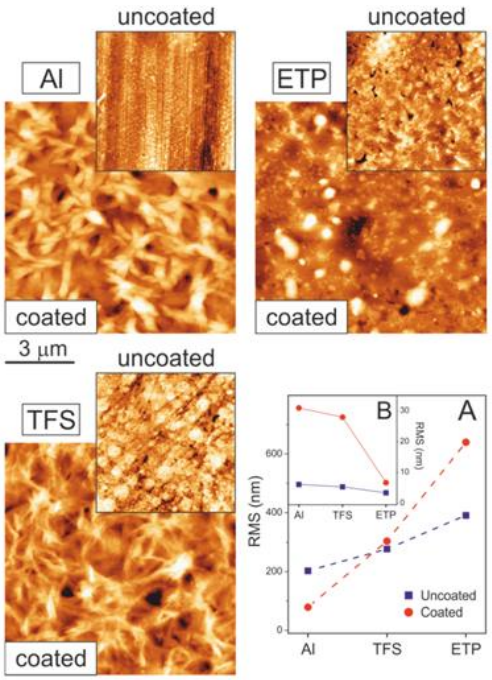
Pazos, MC; Ruiz Bravo, L; Ramos, SE; Osuna, FJ; Pavón, E; Alba, MD
Applied Clay Science, **196** (2020) 105749
Octubre, 2020 | **DOI:** 10.1016/j.clay.2020.105749

Organomica, C8-2-Mica, was obtained from a high charged synthetic mica, Na-2-Mica, by cation exchange reaction with octylammonium cations and these were used to host other bulky guest species such as polyhydroxy aluminium cations, $Al_{13}20$. The hydrolization of 3-mercaptopropyltrimethoxysilane (MPTMS) allowed the covalent attachment with hydroxyl groups of the oligomeric cation, providing thiol groups that create specific adsorption sites, $Al_{13}20$/SH. The structure of the adsorbents was analysed by XRD and Infrared spectroscopy and these were tested as an adsorbent for the removal of zinc and herbicide MCPA from aqueous solutions. C8-2-Mica was the best adsorbent for MCPA and thiol groups favoured the adsorption of Zn^{2+}. Moreover, $Al_{13}20$/SH showed excellent adsorptive properties for the simultaneous adsorption of MCPA and Zn^{2+}.



Bio-Based Coatings for Food Metal Packaging Inspired in Biopolyester Plant Cutin

Benitez, JJ; Osbild, S; Guzman-Puyol, S; Heredia, A; Heredia-Guerrero, JA
Polymers, **12** (2020) 942
Abril, 2020 | **DOI:** 10.3390/polym12040942



Metals used for food canning such as aluminum (Al), chromium-coated tin-free steel (TFS) and electrochemically tin-plated steel (ETP) were coated with a 2-3-µm-thick layer of polyaleuritate, the polyester resulting from the self-esterification of naturally-occurring 9,10,16-trihydroxyhexadecanoic (aleuritic) acid. The kinetic of the esterification was studied by FTIR spectroscopy; additionally, the catalytic activity of the surface layer of chromium oxide on TFS and, in particular, of tin oxide on ETP, was established. The texture, gloss and wettability of coatings were characterized by AFM, UV-Vis total reflectance and static water contact angle (WCA) measurements. The resistance of the coatings to solvents was also determined and related to the fraction of unreacted polyhydroxyacid. The occurrence of an oxidative diol cleavage reaction upon preparation in air induced a structural modification of the polyaleuritate layer and conferred upon it thermal stability and resistance to solvents. The promoting effect of the tin oxide layer in such an oxidative cleavage process fosters the potential of this methodology for the design of effective long-chain polyhydroxyester coatings on ETP.

Preparation and Characterization of Bio-Based PLA/PBAT and Cinnamon Essential Oil Polymer Fibers and Life-Cycle Assessment from Hydrolytic Degradation

Correa-Pacheco, ZN; Black-Solis, JD; Ortega-Gudino, P; Sabino-Gutiérrez, MA; Benitez-Jiménez, JJ; Barajas-Cervantes, A; Bautista-Banos, S; Hurtado-Colmenares, LB

Polymers, **12** (2020) 38
Enero, 2020 | **DOI:** 10.3390/polym12010038

Nowadays, the need to reduce the dependence on fuel products and to achieve a sustainable development is of special importance due to environmental concerns. Therefore, new alternatives must be sought. In this work, extruded fibers from poly (lactic acid) (PLA) and poly (butylene adipate-co-terephthalate) (PBAT) added with cinnamon essential oil (CEO) were prepared and characterized, and the hydrolytic degradation was assessed. A two-phase system was observed with spherical particles of PBAT embedded in the PLA matrix. The

thermal analysis showed partial miscibility between PLA and PBAT. Mechanically, Young's modulus decreased and the elongation at break increased with the incorporation of PBAT and CEO into the blends. The variation in weight loss for the fibers was below 5% during the period of hydrolytic degradation studied with the most important changes at 37 °C and pH 8.50. From microscopy, the formation of cracks in the fiber surface was evidenced, especially for PLA fibers in alkaline medium at 37 °C. This study shows the importance of the variables that influence the performance of polyester-cinnamon essential oil-based fibers in agro-industrial applications for horticultural product preservation.

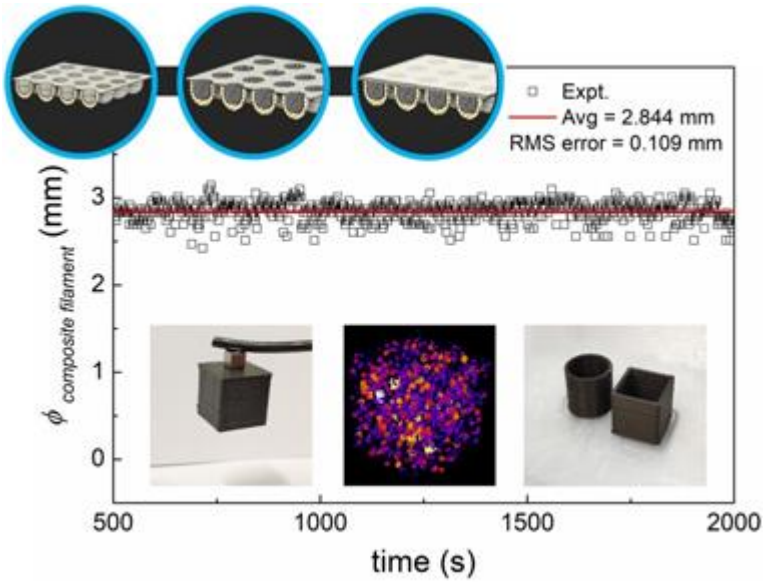
Microstructure and thermal conductivity of Si-Al-C-O fiber bonded ceramics joined to refractory metals

Vera, MC; Martínez-Fernández, J; Singh, M; Casalegno, V; Balagna, C; Ramírez-Rico, J
Materials Letters, **276** (2020) 1282038
Octubre, 2020 | **DOI:** 10.1016/j.matlet.2020.128203

We explore joining Si-Al-C-O fiber-bonded ceramics to Cu-clad-Mo using an Ag-Ti-Cu brazing alloy. A temperature of 900 °C and times in the range of 10-20 min are required to obtain sound joints irrespectively of the fiber orientation. The reaction layer is 1-2 µm thick and free of pores and defects. The thermal conductivity of the joined samples is well described considering that the metal and the ceramic are in series for thermal resistance. This implies that the joint is highly conductive and forms an almost perfect.

Novel procedure for laboratory scale production of composite functional filaments for additive manufacturing

Díaz-García, A; Law, JY; Cota, A; Bellido-Correa, A; Ramírez-Rico, J; Schafer, R; Franco, V
Materials Today Communications, **24** (2020) 101049
Septiembre, 2020 | **DOI:** 10.1016/j.mtcomm.2020.101049



Successful 3D printing by material extrusion of functional parts for new devices requires high quality filaments. Uniform homogeneity and good dispersion of particles embedded in filaments typically takes several cycles of extrusion or well-prepared feedstock by injection molding, industrial kneaders or twin-screw compounding. These methods need specific production devices that are not available in many laboratories non-specialized

in polymer research, such as those working on different material science and technology topics that try to connect with additive manufacturing. Therefore, laboratory studies are usually limited to compositions and filler concentrations provided by commercial companies. Here, we present an original laboratory scale methodology to custom-prepare the feedstock for extruding magnetic composite filaments for fused filament fabrication (FFF), which is attainable by a desktop single-screw extruder. It consists in encapsulating the fillers in custom made capsules that are used as feedstock and reach the melting area of the extruder maintaining the same concentration of fillers. Results have shown that our approach can create smooth and continuous composite filaments with good homogeneity and printability with fine level of dimensional control. We further show the good dispersion of the particles in the composite filament using X-Ray Tomography, which enabled a 3D reconstruction of the spacial distribution of the embedded magnetic particles. The major advantage of this new way of preparing the composite feedstock is that it avoids the hassle of multiple extrusion runs and industrial machinery, yet providing uniform filaments of well controlled filler concentration, which is predictable and reproducible. The proposed methodology is suitable for different polymer matrices and applicable to other functional particle types, not just limited to magnetic ones. This opens an avenue for further laboratory scale development of novel functional composite filaments, useful for any community. This democratization of complex filament preparation, including consumers preparing their own desired uniform novel filaments, will facilitate to unify efforts nearing 3D printing of new functional devices.

Vegetable hierarchical structures as template for bone regeneration: New bio-ceramization process for the development of a bone scaffold applied to an experimental sheep model

Filardo, G; Roffi, A; Fey, T; Fini, M; Giavaresi, G; Marcacci, M; Martínez-Fernández, J; Martini, L; Ramírez-Rico, J; Salamanna, F; Sandri, M; Sprio, S; Tampieri, A; Kon, E

Journal of Biomedical Materials Research Part B-Applied Materials, **108** (2020) 600-611
Abril, 2020 | **DOI:** 10.1002/jbm.b.34414

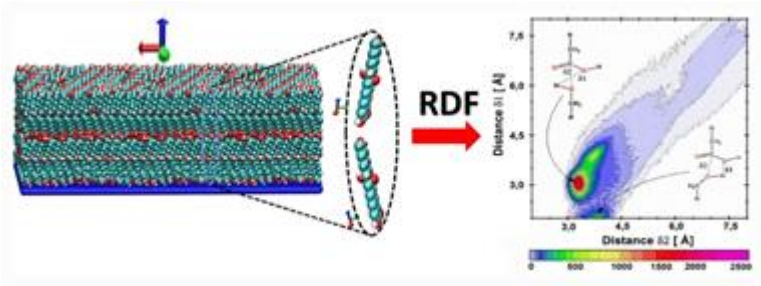
Long bone defects still represent a major clinical challenge in orthopedics, with the inherent loss of function considerably impairing the quality of life of the affected patients. Thus, the purpose of this study was to assess the safety and potential of bone regeneration offered by a load-bearing scaffold characterized by unique hierarchical architecture and high strength, with active surface facilitating new bone penetration and osseointegration in critical size bone defects. The results of this study showed the potential of bio-ceramization processes applied to vegetable hierarchical structures for the production of new wood-derived bone scaffolds, further improved by surface functionalization, with good biological and mechanical properties leading to successful treatment of critical size bone defects in the sheep model. Future studies are needed to evaluate if these scaffolds prototypes, as either biomaterial alone or in combination with augmentation strategies, may represent an optimal solution to enhance bone regeneration in humans.

Elucidating esterification reaction during deposition of cutin monomers from classical molecular dynamics simulations

Bueno, OVM; Benitez, JJ; San-Miguel, MA

Journal of Molecular Modeling, **26** (2020) 280

Septiembre, 2020 | **DOI:** 10.1007/s00894-020-04544-9



The structural behavior of some cutin monomers, when deposited on mica support, was extensively investigated by our research group. However, other events, such as esterification reaction (ER), are still a way to explore. In this paper, we explore possible ER that could occur when these monomers adsorb on support. Although classical molecular dynamics simulations are not able to capture reactive effects, here, we show that they become valuable strategies to analyze the initial structural configurations to predict the most favorable reaction routes. Thus, when depositing aleuritic acid (ALE), it is observed that the loss of capacity to form self-assembled (SA) systems favors different routes to occur ER. In pure ALE bilayers systems, an ER is given exclusively through the -COOH and primary -OH groups. In pure ALE monolayers systems, the ER does not happen when the system is self-assembled. However, for disorganized systems, it is able to occur by two possible routes: -COOH and primary -OH (route 1) and -COOH and secondary -OH (route 2). When palmitic acid (PAL) is added in small quantities, ALE SAMs can now form an ER. In this case, ER occurs mostly through the -COOH and secondary -OH groups. However, when the presence of PAL is dominant, ER can occur with either of both possibilities, that is, routes 1 and 2.

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

Aprovechamiento de Residuos Vegetales para el Diseño de Recubrimientos Inocuos en Envases Metálicos

J. J. Benítez, S. Guzmán-Puyol, E. Domínguez, J. A. Heredia-Guerrero, A. Heredia

Tecnifood. La Revista de la Tecnología Alimentaria. Junio 2020, p.102-103

LIBROS Y OTRAS PUBLICACIONES / BOOKS AND OTHER PUBLICATIONS

Cutin-inspired Polymers and Plant Cuticle-like Composites as sustainable Food Packaging Materials

S. Guzmán-Puyol, A. Heredia, J. A. Heredia-Guerrero, J. J. Benítez

Sustainable Food Packaging Technology. A. Athanassiou (Ed.). Wiley-VCH Verlag. Weinheim, Germany (2020)

ISBN: 978-3-527-34556-4

10.1002/9783527820078.ch6

FORMACION / TRAINING

FORMACIÓN DE GRADUADOS / MASTER DEGREE THESIS

Título:	**Encapsulamiento de residuos radioactivos de alta actividad en matriz arcillosa**
Autor:	Laura Montes Montañez
Directores:	Esperanza Pavón González, Joaquín Ramírez Rico y María Dolores Alba Carranza
Grado:	Trabajo Fin de Master
Centro:	Universidad de Sevilla
Fecha Defensa:	18 septiembre 2020

EQUIPAMIENTO CIENTÍFICO AVAILABLE EQUIPMENT

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

GRUPOS DE INVESTIGACIÓN

Materiales Avanzados
Advanced Materials [642010]

Propiedades Mecánicas, modelización y caracterización de cerámicos avanzados
Mechanical properties, modelling and characterization of advanced ceramics [642016]

Reactividad de Sólidos
Reactivity of Solids [642008]

PERSONAL / PERSONNEL

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Ldo. Nabil Mohamed Amghar

Personal Técnico Contratado

Gdo. Ahmed Taibi

PROYECTOS DE INVESTIGACIÓN / RESEARCH PROJECTS

Integración de la tecnología "calcium looping" en plantas solares para el almacenamiento termoquímico de energía SOlar Calcium-looping integRAtion for Thermo-Chemical Energy Storage

Código/Code:	H2020-ENERGY/0373
Periodo/Period:	01-01-2018 / 30-09-2021
Organismo Financiador/Financial source:	Unión Europea
Importe total/Total amount:	223.500 €
Investigador responsable/Research head:	Luis A. Pérez Maqueda
Componentes/Research group:	María Jesús Diánez Millán, Pedro Enrique Sánchez Jiménez

RESUMEN / ABSTRACT

El almacenamiento de energía es uno de los mayores desafíos para aumentar la viabilidad a corto plazo de las plantas de energía solar concentrada (CSP), que generalmente se caracterizan por su intermitencia en la producción de energía. El proceso Ca-Looping (CaL) basado en la reacción reversible de carbonatación/calcinación del CaO es una de las tecnologías más prometedoras para el almacenamiento termoquímico de energía (TCES). La amplia disponibilidad en la naturaleza de piedra caliza ($CaCO_3$) y su bajo precio (<10 € / ton) son factores clave para el despliegue comercial de la tecnología CaL.

SOCRATCES tiene como objetivo demostrar la viabilidad de la integración CSP-CaL mediante la construcción de una planta a escala piloto que utiliza materiales baratos, abundantes y no tóxicos, así como tecnologías actualmente en uso en la industria, como los reactores de lecho fluidizado y los intercambiadores de calor.

El objetivo global de SOCRATCES es desarrollar un prototipo que reduzca los riesgos a la hora de ampliar la tecnología CaL a escala global y resuelva cuestiones abiertas; comprender mejor y optimizar las eficiencias operativas que podrían obtenerse, con el objeto de habilitar plantas de CSP altamente competitivas y sostenibles.

Energy storage is one of the the greatest challenges for a short-term deeper penetration of Concentrating Solar Power (CSP) plants, which are usually characterized by the intermittency of power production. The Ca-Looping (CaL) process based upon the reversible carbonation/calcination of CaO is one of the most promising technologies for thermochemical energy storage (TCES). The wide availability of natural limestone (almost pure $CaCO_3$) and its low price (<10€/ton) are key factors for the feasibility of the CaL process.

SOCRATCES is aimed at demonstrating the feasibility of the CSP-CaL integration by erecting a pilot-scale plant that uses cheap, abundant and non-toxic materials as well as mature

technologies used in the industry, such as fluidized bed reactor, cyclones or gas-solid heat exchangers.

SOCRATCES global objective is to develop a prototype that will reduce the core risks of scaling up the technology and solve challenges; further understanding and optimise the operating efficiencies that could be obtained; with the longer-term goal of enabling highly competitive and sustainable CSP plants.

Integración del proceso Ca-looping en centrales de energía solar concentrada para el almacenamiento termo-químico de energía Integration of the Ca-looping process in concentrated solar power plants for thermochemical energy storage

Código/Code:	CTQ2017-83602-C2-1-R
Periodo/Period:	01-01-2018 / 31-12-2021
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	145.200 €
Investigador responsable/Research head:	Luis A. Pérez Maqueda, Pedro Enrique Sánchez Jiménez
Componentes/Research group:	María Jesús Diánez Millán

RESUMEN / ABSTRACT

El proyecto solicitado se enmarca dentro del reto general de encontrar nuevas tecnologías de almacenamiento de energía baratas y no contaminantes que permitan superar una de las limitaciones mayores de las fuentes renovables que es la intermitencia en la generación de electricidad. En particular, en este proyecto se propone realizar una integración de la tecnología de Ca-looping en una planta termosolar de concentración. La tecnología de Ca-looping, originariamente propuesta para procesos de captura de CO_2, se basa en procesos de carbonatación-descarbonatación (o calcinación) de óxido de calcio-carbonato cálcico repetidos de forma cíclica. Nuestro grupo de investigación ha trabajado durante varios años en esta tecnología, con el objeto de comprender los mecanismos de desactivación conforme se incrementa el número de ciclos. Así, hemos estudiado los mecanismos cinéticos de estos procesos y los cambios microestructurales que tienen lugar a medida que se ciclan los compuestos. En un proyecto coordinado que concluye a finales de año (SOLARTEQH, Retos 2014) hemos realizado ya una propuesta de integración de Ca-Looping para almacenamiento de energía solar. Este proyecto ha dado lugar a una propuesta H2020 (SOCRATCES) aprobada y que comenzará a comienzos del año próximo. En el proyecto CALSOLAR que ahora presentamos se pretende avanzar más en esta idea de integración para incrementar los valores de eficiencia termoeléctrica. El subproyecto 1 realizará las tareas de coordinación de todo el proyecto. Además, en el subproyecto 1 se realizará la selección, preparación y caracterización de los compuestos a utilizar en el proyecto. En este sentido, se trabajará con empresas mineras que nos facilitarán distintas materias primas (principalmente calizas y dolomitas) con diverso grado

de pureza y cristalinidad. Se prepararán compuestos con sílicas nanoestructuradas obtenidas a partir de cascarilla de arroz (suministradas por arroceras del valle del Guadalquivir). Se investigarán compuestos preparados a partir de escorias de acerías (suministrados por dos empresas del sector afincadas en Andalucía) que son ricos en calcio para su aplicación en ciclos de almacenamiento termoquímico. En el subproyecto, se diseñará y construirá un equipo termogravimétrico que permita realizar experimentos en las condiciones realistas de los ciclos de almacenamiento térmico. Así el equipo permitirá trabajar en condiciones de presión absoluta controlada de CO_2 y en vapor sobrecalentado. En dicho instrumento se realizarán los ciclos de almacenamiento y se estudiarán las condiciones óptimas de dichos ciclos. Se investigarán los mecanismos cinéticos de carbonatación y descarbonatación y se estudiarán los cambios microestructurales durante el ciclado. El equipo de investigación está compuesto por personal con gran experiencia en las tareas propuestas y se cuenta con la participación de investigadores extranjeros con gran experiencia en reacciones sólido-gas y en caracterización microestructural por microscopía de alta resolución. Además, participa una investigadora de la empresa Abengoa con extensa experiencia en almacenamiento termoquímico en plantas solares. Se trabajará en este subproyecto de forma totalmente coordinada con los investigadores del subproyecto 2 con idea de establecer conjuntamente las condiciones de proceso óptimas. Finalmente, los resultados obtenidos del proyecto podrán demostrarse en la planta piloto que se construye en el marco del proyecto H2020 SOCRATCES

The proposal deals with the general social challenge of finding new cheap and environmentally friendly energy storage technologies to overcome the intermittency of energy generation from renewable sources. Particularly, in this project we propose integrating Ca-looping technology within a thermosolar concentration plant. Ca-Looping technology was originally proposed for CO_2 capture and it is based on cycled carbonation-calcination of calcium oxide-calcium carbonate. Our research group has been working on this technology for several years with the objective of understanding the deactivation mechanisms as the number of cycles increases. Thus, we have studied the kinetic mechanisms of these processes and the microstructural changes that takes place during cycling. In a coordinated project that is about to finish this year (SOLARTEQH, Retos 2014) where we already proposed the integration of Ca-Looping for thermosolar energy storage. This project was the basis of a H2020 proposal (SOCRATCES) that has been recently approved and that will start by the beginning of 2018. The project CALSOLAR is a step forward in the integration to increase the efficiency of the plant. Subproject 1 will coordinate the new project. Moreover, subproject 1 will select, prepare and characterize all compounds investigated in the project. We will work with mining companies that will provide the raw materials (mainly limestone and dolomite) with different purities and crystallinity. Composite materials with nanostructured silica obtained from rice husk (provided by rice mills from the Guadalquivir area) will be prepared. Compounds obtained from steel slags (supplied by nearby steel mills) rich in calcium will be prepared. Within subproject 1, a new thermogravimetric instrument to perform thermal storage cycles under realistic conditions will be designed and constructed in our laboratories. This instrument should work under different controlled CO_2 pressures and under superheated steam. The kinetic mechanisms of carbonation and decarbonation and the microstructural changes will be investigated during cycling. The working team is experienced in the tasks of the project while some additional external scientists will participate. Thus, two foreign professors with solid backgrounds in solid-gas reactions and high resolution TEM are collaborating with us. Moreover, an industrial scientist from Abengoa with a very broad experience in thermal storage and thermosolar power plants is also included

in the team. Both subprojects will work in a coordinated way with the aim of setting the optimum conditions for the final application. Finally, the results of the project will be directly applied to the pilot plant constructed within the H2020 SOCRATCES project.

Cerámicas nanoestructuradas a base de carburo de boro y nitruro de titanio para aplicaciones estructurales Boron carbide and titanium nitride-based nanostructured ceramics for structural applications

Código/Code:	MAT2015-71411-R
Periodo/Period:	01-01-2016 / 31-12-2020
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	157.300 €
Investigador responsable/Research head:	Diego Gómez García / Arturo Domínguez Rodríguez
Componentes/Research group:	Francisco L. Cumbreras Hernández, Felipe Gutíerrez Mora, Ana Morales Rodríguez

RESUMEN / ABSTRACT

El proyecto tiene como misión la fabricación de forma controlada científicamente de nanocerámicos de carburo de boro y de nitruro de titanio mediante la técnica de chispa de plasma. Se estudiarán las propiedades mecánicas de ambos cerámicos a temperatura ambiente (dureza y tenacidad), así como su plasticidad a alta temperatura (resistencia a la fluencia, deformación a velocidad constante).

Se pretende estudiar la influencia de la microestructura en la respuesta mecánica, así como dilucidar los mecanismos que controlan la plasticidad (particularmente la interacción de dislocaciones con maclas). Los resultados se modelarán analíticamente o mediante simulación a escala mesoscópica (vía modelos de campos de fases).

Boron carbide and titanium nitride are among the most promising ceramic materials nowadays. In the first case, this is due to the outstanding mechanical properties (it is the third hardest material in nature) and its high resistance to chemical attack. In the case of Titanium nitride, its remarkable optical properties and electrical conductivity makes this a potential material for electronic devices. In both cases, sintering is a challenging issue due to the low diffusitivity. In this project, sintering of these materials by spark plasma sintering will be studied and the conditions for nanostructuration will be determined. Preliminary results show that average grain sizes as low as 100 nm can be achieved. In a second stage, plasticity will be studied. A previous model developed by the authors show that twinning is a key ingredient as a driving force of plasticity of boron carbide. The case of titanium nitride is mostly exciting because the stacking fault energy is the lowest ever known and it can make twinnin very favoured. The

comparison between these two systems can be a clue about the basic mechanism for hardening in these ceramic materials.

Procesado y Caracterización de Composites Cerámicos con Nanomateriales Laminados Bidimensionales Processing and characterization of ceramic composites with two-dimensional laminar nanomaterials

Código/Code:	PGC2018-101377-B-I00
Periodo/Period:	01-01-2019 / 31-12-2022
Organismo Financiador/Financial source:	Ministerio de Ciencia, Innovación y Universidades
Importe total/Total amount:	121.000 €
Investigador responsable/Research head:	Ángela Gallardo López, Rosalía Poyato Galán
Componentes/Research group:	Antonio Muñoz Bernabé, Ana Morales Rodríguez, Felipe Gutiérrez Mora

La incorporación de nanomateriales bidimensionales en materiales compuestos de matriz cerámica está aumentando en un esfuerzo para superar la inherente fragilidad de las cerámicas y para conferirles nuevas funcionalidades. Aún existen cuestiones abiertas en este campo en cuanto a resistencia mecánica, tenacidad a la fractura, cinética de crecimiento de fisuras, comportamiento tribológico, papel de fases interfaciales o idoneidad para el electromecanizado, entre otras. Aunque las nanoláminas de grafeno (NLG) se han mostrado como una segunda fase muy adecuada, los nanomateriales inorgánicos análogos al grafeno podrían extender el rango de aplicabilidad de los materiales con grafeno. El uso de nanoláminas de nitruro de boro (NLNB) en estos compuestos es muy prometedor y está prácticamente inexplorado.

Este proyecto propone un estudio sistemático de materiales compuestos con usos en aplicaciones estructurales y funcionales, con dos matrices de circona estabilizada con itria y dos tipos diferentes de nanomateriales 2D -nanoláminas de grafeno o de nitruro de boro- para profundizar en la comprensión de sus comportamientos mecánico y eléctrico. Con este fin, se fabricarán materiales con matrices de circona tetragonal dopada con 3 %mol de itria y circona cúbica dopada con itria, persiguiendo una microestuctura óptima con una distribución homogénea de los nanomateriales 2D en ambas matrices cerámicas. Se investigarán en profundidad materiales con NLG para dar respuesta a cuestiones abiertas en el conocimiento de estos materiales. La distribución, tamaño e integridad estructural de las NLG se caracterizarán mediante difracción de rayos X, microscopía electrónica de barrido y espectroscopía Raman, y las intercaras entre las NLG y la matriz se caracterizarán mediante microscopía electrónica de transmisión. La resistencia mecánica, resistencia a la rotura, mecanismos de refuerzo y cinética de crecimiento de grano en estos materiales se examinará en profundidad, y se establecerá la mejor combinación de ruta de procesado y contenido de NLG en términos de refuerzo a la matriz. Se realizarán medidas de conductividad eléctrica en materiales con diferentes

contenidos de NLG y se evaluará la respuesta al electromecanizado de los materiales eléctricamente conductores. Se realizarán también medidas de conductividad eléctrica en función de la temperatura para describir las posibles variaciones en el tipo de conducción al aumentar el contenido en NLG. Por otra parte, se investigarán materiales con NLNB con el objetivo de obtener una primera aproximación a la comprensión de este sistema. Con este fin, tras la síntesis de las NLNB usando una estrategia de mezcla de disolventes para la exfoliación en fase líquida de nanoláminas a partir de polvo de BN hexagonal, se prepararán polvos con diferentes contenidos de NLNB usando técnicas de procesado de polvo en medio húmedo. La caracterización microestructural de los materiales sinterizados mediante “Spark Plasma Sintering" se realizará mediante microscopía electrónica de barrido y de transmisión, difracción de rayos X y espectroscopía Raman. Se estudiarán propiedades como dureza, resistencia a la flexión y resistencia al desgaste, y se realizarán ensayos mecánicos a alta temperatura. Se analizará la conductividad eléctrica en función de la temperatura para esclarecer el efecto de la incorporación de una segunda fase aislante en las fronteras de grano sobre el comportamiento eléctrico de un conductor iónico.

Two-dimensional nanomaterials are being increasingly used as fillers in ceramic composites in an effort to overcome the inherent fragility of ceramics and to provide them with new functionalities. There are open issues in the field of these composites regarding their strength and fracture toughness mechanisms, crack growth kinetics, tribological behavior, role of interfacial phases or suitability for electrical discharge machining, among others. Although graphene nanosheets (GNS) are excellent fillers, inorganic graphene analogues could extend the range of applicability of graphene ceramic composites. The use of boron nitride nanosheets (BNNS) as fillers in ceramic composites is promising and practically unexplored.

This proposal outlines a systematic study of composites intended for use in structural and functional applications, with two different ceramic matrices from the yttria-stabilized zirconia system incorporating two different 2D laminar nanomaterials -graphene or boron nitride nanosheets-, to deepen in the understanding of their mechanical and electrical behavior. To that end, composites with 3 mol% yttria tetragonal zirconia and 8 mol% yttria cubic zirconia matrices will be fabricated, pursuing an optimum microstructure with a homogeneous distribution of the 2D nanomaterials throughout both ceramic matrices. On the one hand, ceramic composites with graphene nanosheets will be investigated in depth to complete the gaps in the current knowledge of these materials. The distribution, size and structural integrity of the GNS will be characterized by X-ray diffraction, scanning electron microscopy and Raman spectroscopy while the interfaces between the GNS and the matrix will be characterized by transmission electron microscopy. The strength, failure resistance, reinforcement mechanisms and crack growth kinetics of these composites will be thoroughly examined, and the best combination of processing route and GNS content in terms of reinforcement will be established. Electrical conductivity measurements of composites with different GNS contents will be carried out at room temperature and the response to electrical discharge machining of the electrically conductive composites will be evaluated. Conductivity measurements will be carried out also as a function of temperature in order to describe the possible variations of conduction type when increasing the GNS content. On the other hand, ceramic composites with boron nitride nanosheets will be investigated in order to get a first approach to the understanding of this system. For this purpose, after the synthesis of the BN nanosheets using a mixed-solvent strategy for liquid exfoliation of BNNS from h-BN powder, composites with different contents of BNNS will be prepared using wet powder processing techniques. The microstructural

characterization of the spark plasma sintered composites will be carried out by scanning and transmission electron microscopy, X-ray diffraction and Raman spectroscopy. Mechanical properties as hardness, flexural strength and wear resistance will be studied at room temperature, whereas deformation tests at high temperatures will be also performed. The electrical conductivity as a function of temperature will be analyzed in order to clarify the effect of incorporating an insulating second phase at the grain boundaries on the electrical performance of an ionic conductor.

Nuevos materiales para el almacenamiento de Energía Solar Concentrada mediante Calcium-Looping (SOLACAL) New materials for energy storage of Concentrated Solar Power using Calcium-Looping (SOLACAL)

Código/Code:	US-1262507
Periodo/Period:	01-02-2020 / 30-04-2022
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	76.700 €
Investigador responsable/Research head:	Antonio Perejón Pazo y José Manuel Valverde Millán (US)
Componentes/Research group:	María Jesús Diánez Millán, Luis A. Pérez Maqueda, Virginia Moreno García

RESUMEN / ABSTRACT

En este proyecto se estudia el comportamiento de nuevos materiales basados en CaO durante ciclos de calcinación/carbonatación (Ca-Looping) en condiciones realistas de almacenamiento de energía en centrales de energía solar concentrada (CSP).

Para simular las condiciones realistas se utilizan equipos termogravimétricos capaces de emplear elevadas velocidades de calentamiento y enfriamiento y diferentes atmósferas de gases. De esta forma, los resultados obtenidos son realmente representativos y podrán ser extrapolados a las condiciones prácticas de operación en plantas CSP.

Se estudia la reactividad multicícilica de muestras de caliza y dolomita, a las que se les realizan tratamientos mecánicos y con ácido acético que pueden mejorar su actividad. Además, se ha demostrado que la presencia de MgO en la dolomita calcinada estabiliza térmicamente el CaO, por lo que se preparan dolomitas sintéticas con distinto contenido en MgO mediante tratamientos mecánicos y coprecipitación con el objeto de encontrar la cantidad óptima de MgO que mejore la actividad multicíclica del CaO. Se estudian asimismo otros materiales con los que se pueda aumentar la temperatura de carbonatación, como el $SrCO_3$ y el $BaCO_3$, lo que permitiría incrementar aún más la eficiencia termoeléctrica de las plantas CSP con almacenamiento termoquímico.

Un aspecto relevante de SOLACAL es que los resultados obtenidos serán transferidos de manera directa a la planta CSP-CaL de demostración que se está construyendo en Sevilla dentro del proyecto H2020 SOCRATCES iniciado en 2018 y coordinado por la Universidad de Sevilla.

This project is focused on the performance of new CaO-based materials during calcination/carbonation cycles (Ca-Looping) under realistic energy storage conditions in concentrated solar power plants (CSP).

In order to simulate realistic conditions, thermogravimetric instruments are used, which are able of employing high heating and cooling rates and different atmospheres of gases. In this way, the results obtained are truly representative and can be extrapolated to practical operating conditions in CSP plants. The multicycle reactivity of limestone and dolomite samples is studied. These samples are modified by mechanical and acetic acid treatments that can improve their reactivity. Moreover, it has been shown that the presence of MgO in calcined dolomite thermally stabilizes CaO. Synthetic dolomites with different MgO content are prepared by mechanical treatments and co-precipitation in order to find the optimal amount of MgO that improves the multicycle activity of CaO. Other materials in which the carbonation temperature can be increased, such as $SrCO_3$ and $BaCO_3$, are also studied, which would further increase the thermoelectric efficiency of CSP plants with thermochemical energy storage.

A relevant aspect of SOLACAL is that the results obtained will be transferred directly to the CSP-CaL demonstration plant that is being built in Seville within the H2020 SOCRATCES project, started in 2018 and coordinated by the University of Seville.

OTROS PROYECTOS / OTHER PROJECTS

Diseño y selección de materiales novedosos para fabricar pilas de combustible de óxido sólido de alto rendimiento

Código/Code:	US-15382
Periodo/Period:	01-02-2020 / 31-01-2022
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	100.000 €
Investigador responsable/Research head:	Francisco J. García García (US)
Participante del ICMS como investigador:	Francisco J. Gotor Martínez

Desarrollo y caracterización de nuevos composites geopoliméricos basados en residuos de la industria del olivar. Hacia una construcción sostenible

Código/Code:	MAT2017-88097-R
Periodo/Period:	01-01-2018 / 31-12-2020
Organismo Financiador/Financial source:	MINECO, proyecto I+D+I RETOS
Importe total/Total amount:	108.900 €
Investigador responsable/Research head:	D. Eliche-Quesada (UJaén)
Participante del ICMS como investigador:	Pedro José Sánchez Soto

Sinterización FLASH Reactiva para la preparación de compuestos inestables y resistentes a la densificación

Código/Code:	201960E092
Periodo/Period:	01-10-2019 / 30-09-2022

Organismo Financiador/Financial source: CSIC (Intramural)
Importe total/Total amount: 350.000 €
Investigador responsable/Research head: Luis Allan Pérez Maqueda

Síntesis y procesado de cerámicas avanzadas mediante Sinterizado FLASH

Código/Code: 202060I004
Periodo/Period: 02-10-2020 / 01-10-2021
Organismo Financiador/Financial source: CSIC (Ayudas para la incorporación de personal investigador a las escalas científicas del CSIC)
Importe total/Total amount: 5.000 €
Investigador responsable/Research head: Pedro Enrique Sánchez Jiménez

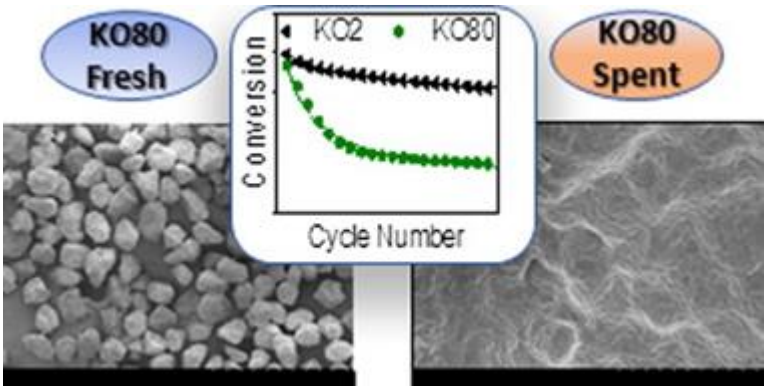
AYUDAS PARA LA ADQUISICIÓN DE EQUIPOS

Equipo de Análisis Térmico de Altas Prestaciones (EQC2019-005791-P)
Financia: Ministerio de Ciencia e Innovación y Consejo Superior de Investigaciones Científicas
Importe Concedido: 112.379,78 €
Periodo: 1-1-2019 / 31-12-2021
Cofinanciado por el Grupo de Investigación "Reactividad de Sólidos"

Equipo para estudios de reacciones térmicas sólido-gas (EQC2018-004339-P)
Financia: Ministerio de Ciencia e Innovación y Consejo Superior de Investigaciones Científicas
Importe Concedido: 131.533,77 €
Periodo: 1-1-2018 / 31-12-2020
Cofinanciado por el Grupo de Investigación "Reactividad de Sólidos"

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

Role of particle size on the multicycle calcium looping activity of limestone for thermochemical energy storage
Duran-Martín, JD; Jiménez, PES; Valverde, JM; Perejón, A; Arcenegui-Troya, J; Trinanes, PG; Maqueda, LAP
Journal of Advanced Research, **22** (2020) 67-76
Marzo, 2020 | **DOI:** 10.1016/j.jare.2019.10.008

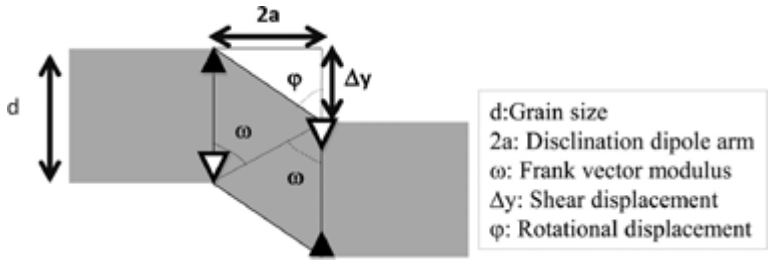


The calcium looping process, based on the reversible reaction between $CaCO_3$ and CaO, is recently attracting a great deal of interest as a promising thermochemical energy storage system to be integrated in Concentrated Solar Power plants (CaL-CSP). The main drawbacks of the system are the incomplete conversion of CaO and its sintering-induced deactivation. In this work, the influence of particle size in these deactivation mechanisms has been assessed by performing experimental multicycle tests using standard limestone particles of well-defined and narrow particle size distributions. The results indicate that CaO multicycle conversion benefits from the use of small particles mainly when the calcination is carried out in helium at low temperature. Yet, the enhancement is only significant for particles below 15 µm. On the other hand, the strong sintering induced by calcining in CO_2 at high temperatures makes particle size much less relevant for the multicycle performance. Finally, SEM imaging reveals that the mechanism responsible for the loss of activity is mainly pore-plugging when calcination is performed in helium, whereas extensive loss of surface area due to sintering is responsible for the deactivation when calcination is carried out in CO_2 at high temperature.

Disclination dipoles are the Holy Grail for high temperature superplasticity in ceramics

Moshtaghioun, BM; Bejarano-Palma, JA; García, DG

Scripta Materialia, **185** (2020) 21-24
Agosto, 2020 | **DOI:** 10.1016/j.scriptamat.2020.03.049



A model for high-temperature plasticity of polycrystals controlled by disclination dipoles is proposed that predict a parabolic dependence of the strain rate versus the applied stress. The presence of a precise stationary disclination density explains the origin of plasticity without microstructural invariance, commonly known as superplasticity. The disclination mechanism is universal, although other processes, such as dislocation glide, are superposed to this one in many systems such as metals or metallic alloys. While, in ceramics it is likely to be the only operative mechanism. Activation of disclination dipoles is a necessary condition for plasticity and sufficient one for superplastic yielding.

Dust filter of secondary aluminium industry as raw material of geopolymer foams

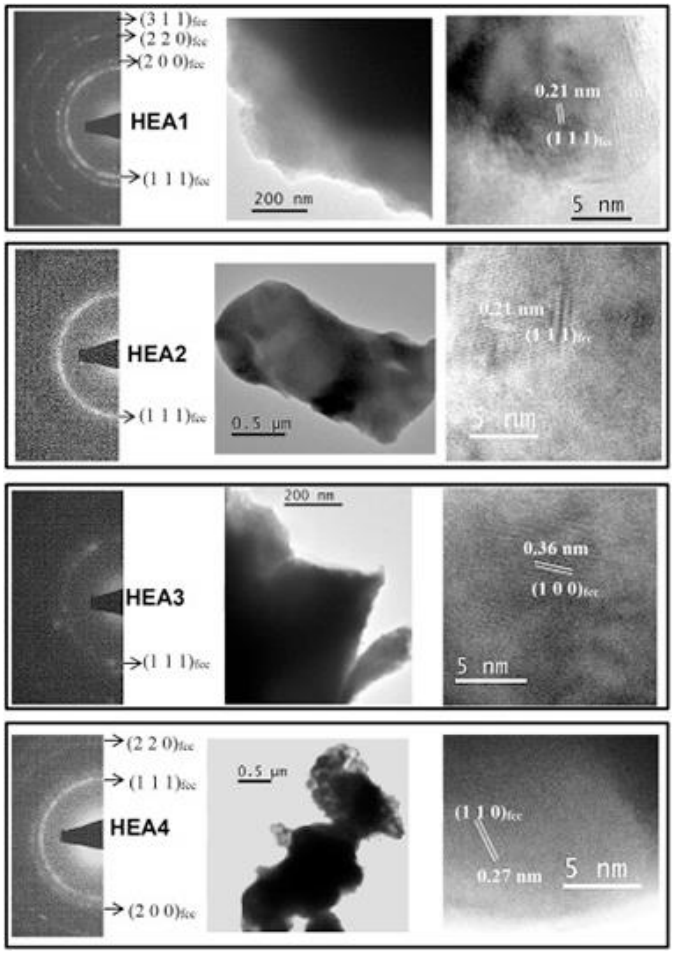
Eliche-Quesada, D; Ruiz-Molina, S; Pérez-Villarejo, L; Castro, E; Sánchez-Soto, PJ
Journal of Building Engineering, **32** (2020) 101656
Noviembre, 2020 | **DOI:** 10.1016/j.jobe.2020.101656

In this work, the use of waste dust filter of secondary aluminum industry (DFA) to obtain geopolymer foams has been studied. The waste was used as source of alumina and foaming agent. As precursor and principal reactive silica supplier rice husk ash was used. Precursors were chemically activated by means of a sodium hydroxide aqueous solution and a commercial sodium silicate solution. The influence of the DFA content or Si/Al molar ratio (4-7) were determined by keeping the Si/Na molar ratio of 0.7 M constant and the concentration of sodium hydroxide in the activating solution equal to 8.5 M. The geopolymer foams obtained were studied by X-ray Diffraction (XRD), adsorption/desorption of nitrogen, infrared spectroscopy (FTIR), and scanning electron microscope (SEM) techniques. The results indicated that geopolymer foams presented low values of bulk density (643-737 kg/m^3) high values of apparent porosity (62-70%), low, but sufficient values of compressive strength (0.5-1.7 MPa) and good values of thermal conductivity (0.131-0.157 W/mK). Lower values of thermal conductivity were obtained for Si/Al = 4 and 5 M ratios, due to the highest apparent porosity and the highest total pore volume. These geopolymer foam materials have similar properties to other construction materials sector such as gypsum boards, foamed concrete, or insulating materials. In addition, its use in other applications of interest such as catalyst support or gas filtration materials could be investigated.

Development of Ti(C,N)-based cermets with (Co,Fe,Ni)-based high entropy alloys as binder phase

de la Obra, AG; Sayagues, MJ; Chicardi, E; Gotor, FJ
Journal of Alloys and Compounds, **814** (2020) 152218
Enero, 2020 | **DOI:** 10.1016/j.jallcom.2019.152218

High entropy alloys have been proposed as novel binder phases in cemented carbides and cermets. Many aspects related to the stability of these alloys during the liquid phase sintering process are still unclear and were addressed in this work. Consolidated Ti(C,N)-based cermets using four different (Co,Fe,Ni)based high entropy alloys as the binder phase were obtained. The



chosen alloys - CoCrCuFeNi, CoCrFeNiV, CoCrFeMnNi and CoFeMnNiV - were previously synthesized through mechanical alloying and a single alloyed solid solution phase with fcc structure and nanometric character was always obtained. The powdered alloys and the consolidated cermets were analyzed by X-ray diffraction, scanning electron microscopy, X-ray energy dispersive spectrometry and transmission electron microscopy. Differential thermal analysis was employed to determine the melting point of the four high entropy alloys that ranged between 1310 °C and 1375 °C. Although a high temperature of 1575 °C was required to obtain the highest cermet densification by pressureless sintering, porosity still remained in most of the cermets. Best densification was achieved when CoCrFeNiV was used as the binder phase. During liquid phase sintering, different compositional changes were observed in the ceramic and binder phases. A core-rim microstructure was observed in cermets containing V in the alloys (CoCrFeNiV and CoFeMnNiV), since this element was incorporated to the carbonitride structure during sintering. A slight Cr segregation was detected in cermets containing Cr, leading to CrTi-rich alloys in small binder regions. However, a great Cu segregation was produced when CoCrCuFeNi was used, and the formation of two different fcc alloys -a Cu-rich and a Cu-depleted- was observed. Finally, a loss of Mn was also evidenced in CoCrFeMnNi and CoFeMnNiV, probably due to its sublimation at the sintering temperature.

Mg_2SiO_4-$MgAl_2O_4$ directionally solidified eutectics: Hardness dependence modelled through an array of screw dislocations

Moshtaghioun, BM; Gómez-García, D; Pena, JI

Journal of the European Ceramic Society, **40** (2020) 4171-4176

Septiembre, 2020 | **DOI:** 10.1016/j.jeurceramsoc.2020.05.015

Mg_2SiO_4-$MgAl_2O_4$ eutectic ceramics have been fabricated by means of the laser floating zone (LFZ) technique. The microstructure has revealed as an unusual one at lower growth rate, composed of broken lamellae of $MgAl_2O_4$ distributed randomly along one matrix, composed of Mg_2SiO_4. At higher growth rates, a cell structure with intra-cell lamella structure is dominant. Contrary to most eutectic systems, hardness is not dependent upon the inter-spacing, but it

does depend on one characteristic length of lamellae: their perimeter. One simple model based upon the dislocation is proposed, which successfully accounts for such extraordinary hardness law. Accordingly, Mg_2SiO_4-$MgAl_2O_4$ eutectic ceramics fabricated at 50 mm/h growth rate with the smallest $MgAl_2O_4$ lamella perimeter favorably showed more elevated hardness (13.4 GPa from Vickers indentation and 15.3 GPa from nanoindentation) and strength (430 MPa) than those found in the monolithic Mg_2SiO_4 matrix.

Obituary: Prof. José Manuel Criado

Pérez-Maqueda, LA; Real, C; Gotor, FJ; Alcala, MD; Malek, J; Koga, N
Journal of Thermal Analysis and Calorimetry, **139** (2020) 2941-2942 (4,626)
Marzo, 2020 | **DOI:** 10.1007/s10973-019-09207-3

Development of a high-pressure thermobalance working under constant rate thermal analysis

Perejón, Antonio; Sánchez-Jiménez, Pedro E.; Criado, José Manuel; Pérez-Maqueda, Luis A.
Journal of Thermal Analysis and Calorimetry, **142** (2020) 1329-1334
Abril, 2020 | **DOI:** 10.1007/s10973-020-09644-5

A thermogravimetric instrument that works at high pressure of different gases has been designed and assembled. The instrument has been devised to work in a temperature range from room temperature to 1000 ^{o}C in various controlled pressures of selected gas up to 15 bar, and under conventional rising temperature and constant rate thermal analysis (CRTA) modes. CRTA method allows an intelligent control of the reaction temperature using a feedback system that monitors the mass gain or mass loss of the sample in such a way that the reaction rate is maintained constant all over the process at a preselected value. CRTA method provides a significant advantage for studying processes under high pressure as it reduces heat and mass transfer phenomena that are very relevant under these high-pressure experimental conditions. The thermal oxidation of Ni at 8 bar of pure oxygen has been used for testing the performance of the instrument under both linear heating rate and CRTA conditions.

Characterization, thermal and ceramic properties of phyllite clays from southeast Spain

Garzon, E; Pérez-Villarejo, L; Sánchez-Soto, Pedro J
Journal of Thermal Analysis and Calorimetry, **142** (2020) 1659-1670
Diciembre, 2020 | **DOI:** 10.1007/s10973-020-10160-9

The present research studied a set of phyllite clays from several deposits in southeast Spain. These phyllite clays have traditionally been used as sealing material to impermeabilize roofs, embankments, ponds, construction and waste landfill, with recent applications in the preparation of new mortars. However, studies on thermal behaviour and ceramic properties of phyllite clays have been scarce. The present research showed a summary of previous characterization studies on representative phyllite clays from these deposits with additional results. Mineralogical, by X-ray diffraction, and chemical, by X-ray fluorescence characterization of these samples were summarized. Thermal analysis methods (DTA-TG and thermal

diffractometry) were applied to achieve a more complete mineralogical characterization. Several phyllite clay samples were selected for a ceramic study by firing pressed powdered samples up to 1300 °C. Sintered or vitrified materials, with porosities almost zero, were obtained from these phyllite clays after firing at 1100-1200 °C, with apparent densities between 2.1 and 2.4 g cm^{-3}. Higher firing temperatures (> 1250 °C) produced deformation and expansion of the ceramic bodies. These results allowed obtain the vitrification temperature T_v and the temperature of the maximum bulk density (T_d). According to the previous mineralogical and chemical characterization and the values of these parameters, the phyllite clay samples were classified in three varieties, as follows: (1) Micaceous, characterized by predominant layer silicates, mainly muscovite or illite, alkaline elements (mainly K_2O higher than 3.5 mass%) and lower values of both T_v and T_d, (2) Quartzitic, with predominant quartz and SiO(2)and intermediate values of T_v and T_d, and (3) Carbonaceous, characterized by predominant dolomite, medium contents of CaO and MgO and higher values of both T_v and T_d. These results are interesting for the application of these phyllite clays as ceramic raw materials.

Control of experimental conditions in reaction flash-sintering of complex stoichiometry ceramics

Gil-González, E; Perejón, A; Sánchez-Jiménez, PE; Román-González, D; Pérez-Maqueda, LA
Ceramics International, **46** (2020) 29413-29420
Diciembre, 2020 | **DOI:** 10.1016/j.ceramint.2020.05.091

The inherent potential of reaction flash-sintering for the preparation of complex oxides is evidenced by the one-step synthesis and densification of a ceramic of complex stoichiometry. The system $Bi_{0.93}La_{0.07}FeO_3$, a multi-ferroic ceramic with promising technological applications, has been chosen. This system presents three different metals in its composition and it is extremely challenging to prepare by conventional procedures. Non-stoichiometric materials with unwilling secondary phases are usually obtained by conventional methods, due to the high volatility of bismuth oxide at the temperatures required for inducing the solid-solid reactions. Here, it is demonstrated that a careful control of the experimental flash conditions (applied electric field and selected current density limit) is required to obtain a high quality ceramic. Small deviations from the optimum conditions result in either non-stoichometric or poorly densified samples.

Synthesis of all equiatomic five-transition metals High Entropy Carbides of the IVB (Ti, Zr, Hf) and VB (V, Nb, Ta) groups by a low temperature route

Chicardi, E; García-Garrido, C; Hernandez-Saz, J; Gotor, FJ
Ceramics International, **46** (2020) 21421-21430
Septiembre, 2020 | **DOI:** 10.1016/j.ceramint.2020.05.240

The six possible equiatomic five-transition metal High Entropy Carbides (HECs) of the IVB (Ti, Zr, Hf) and VB (V, Nb, Ta) groups of the periodic table, i.e., $TiZrHfVNbC_5$, $TiZrHfVTaC_5$, $TiZrHfNbTaC_5$, $TiZrVNbTaC_5$, $TiHfVNbTaC_5$ and $ZrHfVNbTaC_5$, were successfully obtained via a powder metallurgy route at room temperature, specifically, by one-step diffusion mechanosynthesis starting from the elemental constituents (using graphite as the carbon source). Three of those HECs, $TiZrHfVTaC_5$, $TiZrVNbTaC_5$ and $ZrHfVNbTaC_5$, were developed for the first time. Their

development was possible without any subsequent thermal treatment, in contrast to the usual way (reactive sintering at 1800-2200 °C), and in a powder form, make them potential advanced raw ceramics for hard, refractory and oxidation resistance coatings or matrix phase composites.

Synthesis of Mn^{2+}-doped ZnS by a mechanically induced self-sustaining reaction

Aviles, MA; Córdoba, JM; Sayagues, MJ; Gotor, FJ
Journal of Materials Science, **55** (2020) 1603-1613
Febrero, 2020 | **DOI:** 10.1007/s10853-019-04138-8

The mechanochemical process denoted as a mechanically induced self-sustaining reaction was successfully applied in obtaining Mn-doped ZnS samples with Mn content between 0 and 5 mol%. The process consists in milling Zn/Mn/S powder elemental mixtures with the appropriate stoichiometry, which promotes after approximately 80 min the induction of a combustion reaction. The doping level was properly adjusted by controlling the atomic ratio of the starting mixture. A complete characterization of samples was carried out, including X-ray diffraction, high-resolution transmission electron microscopy, selected area electron diffraction, energy-dispersive X-ray spectroscopy, Raman spectroscopy, diffuse reflectance UV-Vis spectroscopy and emission and excitation photoluminescence measurements. A wurtzite structure, in which Mn^{2+} replaces Zn^{2+}, was obtained with a nanometric character. The photoluminescence of samples showed the characteristic $Mn^{2+4}T_1$-$^6A_1$ emission that was highly dependent on the doping level. The maximum luminescence efficiency through the ZnS excitation was found for a doping value of 1 mol%. The photoluminescence showed virtually no contribution from the host emission, which confirmed that samples were properly doped.

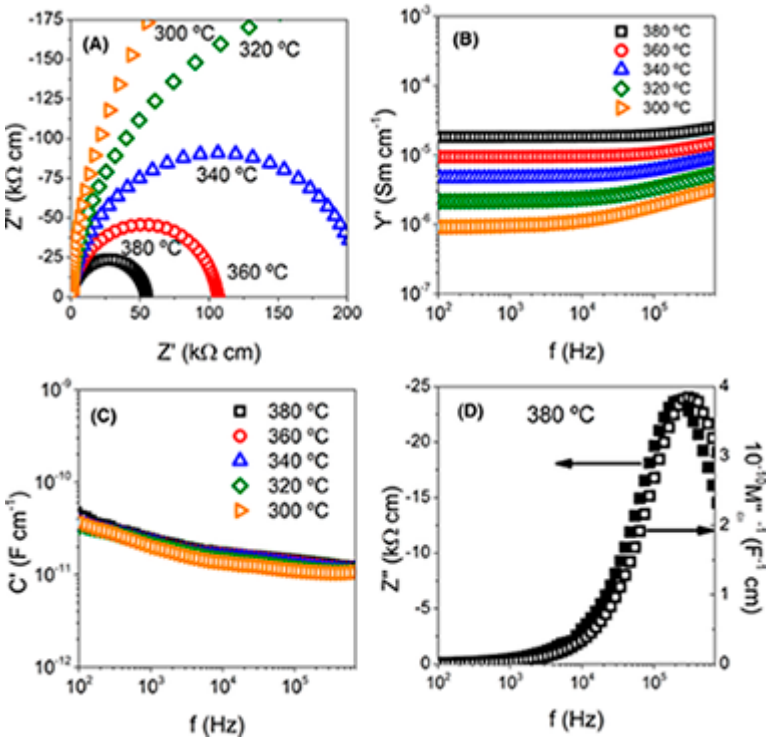
Graphene-coated Ti-Nb-Ta-Mn foams: A promising approach towards a suitable biomaterial for bone replacement

Lascano, S; Chavez-Vasconez, R; Munoz-Rojas, D; Aristizabal, J; Arce, B; Parra, C; Acevedo, C; Orellana, N; Reyes-Valenzuela, M; Gotor, FJ; Arevalo, C; Torres, Y
Surface & Coatings Technology, **401** (2020) 126250
Noviembre, 2020 | **DOI:** 10.1016/j.surfcoat.2020.126250

The design of bone implants with proper biological and mechanical properties remains a challenge in medical implantology. The use of bioactive coatings has been shown to improve the biocompatibility of the implant surface. In this study, a new approach including porous scaffolds, beta-Ti alloys and nanocoatings to design new bone implants is presented. Porous Ti-Nb-Ta-xMn alloys (x: 2, 4, and 6 wt%) substrates were obtained by powder metallurgy and the effect of the porosity and Mn content on mechanical properties was studied. CVD single-layer graphene was transferred onto the porous substrates that presented the best mechanical response (x: 4 wt%) for further evaluation of in vitro cell behavior (biocompatibility and cell adhesion). Cytotoxicity and biocompatibility tests confirmed that cell adhesion and proliferation were successfully achieved on graphene-coated porous substrates, confirming these systems are potential candidates for using in partial bone tissue replacement.

Processing and properties of $Bi_{0.98}R_{0.02}FeO_3$ (R = La, Sm, Y) ceramics flash sintered at similar to 650 °C in <5 s

Gil-González, E; Perejón, A; Sánchez-Jiménez, PE; Raj, R; Pérez-Maqueda, LA
Journal of the American Ceramic Society, **103** (2020) 136-144
Enero, 2020 | **DOI:** 10.1111/jace.16718



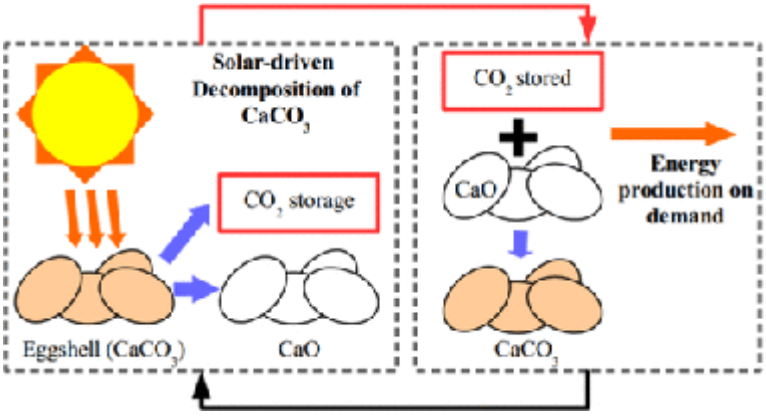
We show that flash sintering produces single-phase, nanograin-sized polycrystals of isovalent-substituted multiferroic ceramics of complex compositions. Single-phase polycrystals of $Bi_{0.98}R_{0.02}FeO_3$ (R = La, Sm, Y) were produced at a furnace temperature of ~650°C in a few seconds by the application of an electric field of 50 V cm^{-1}, with the current limit set to 40 mA mm^{-2}. The dielectric and insulating properties compared favorably with expected values. Impedance spectroscopy suggests electrically homogenous microstructure, except for the sample $Bi_{0.98}R_{0.02}FeO_3$ that shows a small grain boundary contribution to the impedance. These results reinforce the enabling nature of flash sintering for ceramics which pose difficulties in conventional sintering because they contain low melting constituents or develop secondary phases during the sintering protocol.

Calcium-Looping Performance of Biomineralized $CaCO_3$ for CO_2 Capture and Thermochemical Energy Storage

Arcenegui-Troya, J; Sánchez-Jiménez, PE; Perejón, A; Valverde, JM; Chacartegui, R; Pérez-Maqueda, LA
Industrial & Engineering Chemistry Research, **59** (2020) 12924-12933
Julio, 2020 | **DOI:** 10.1021/acs.iecr.9b05997

The commercial deployment of calcium-looping (CaL)-based technologies relies on the availability of nontoxic, widely available and cheap $CaCO_3$ rich materials. Biomineralized $CaCO_3$ from waste amply fulfills the aforementioned requirements. In the present work, we study the performance of eggshell and snail shell from food waste as CaO precursors for CaL applications. The results obtained suggest the feasible use of these waste materials. The multicyclic conversion exhibited by biomineralized $CaCO_3$ was comparable to that demonstrated by limestone, which is a commonly proposed material for CaL applications. In addition, the

temperature needed to completely calcine biomineralized $CaCO_3$ in short residence times is lower than that required to fully calcine limestone. This would mitigate the energy cost of the technology.



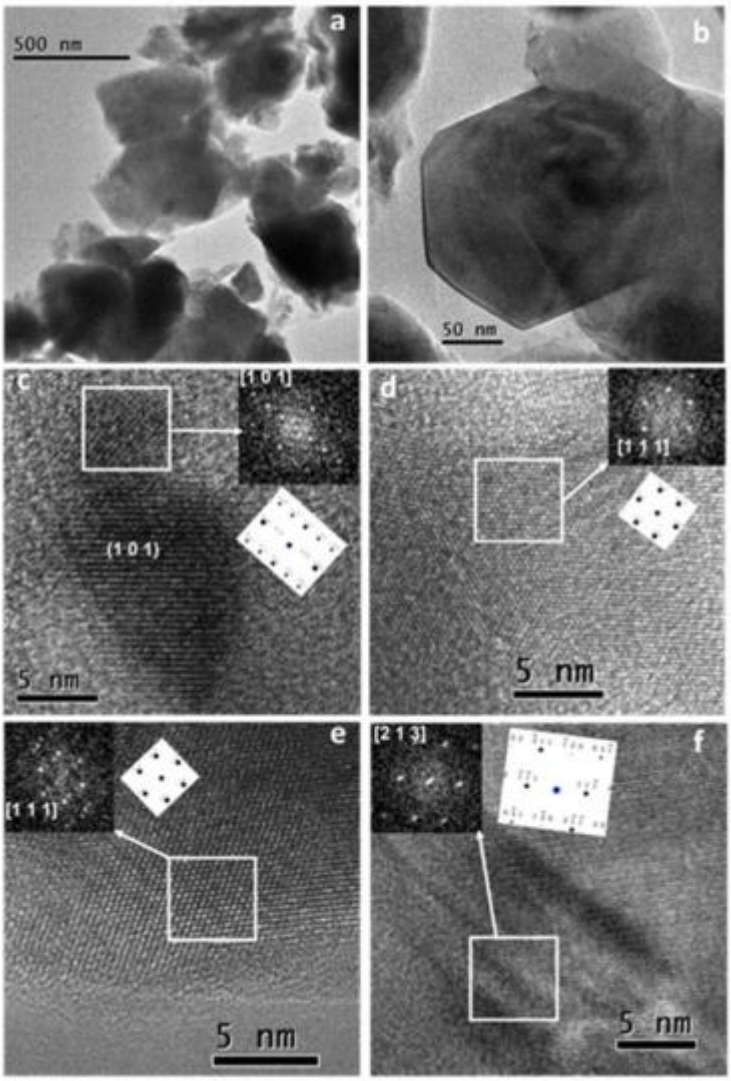
Electrochemically Exfoliated Graphene-Like Nanosheets for Use in Ceramic Nanocomposites

Poyato, R; Verdugo, R; Munoz-Ferreiro, C; Gallardo-López, A
Materials, **13** (2020) 11
Junio, 2020 | **DOI:** 10.3390/ma13112656

In this work, the synthesis of graphene-like nanosheets (GNS) by an electrochemical exfoliation method, their microstructural characterization and their performance as fillers in a ceramic matrix composite have been assessed. To fabricate the composites, 3 mol % yttria tetragonal zirconia (3YTZP) powders with 1 vol % GNS were processed by planetary ball milling in tert-butanol to enhance the GNS distribution throughout the matrix, and densified by spark plasma sintering (SPS). According to a thorough Raman analysis and SEM observations, the electrochemically exfoliated GNS possessed less than 10 graphene layers and a lateral size lower than 1 µm. However, they contained amorphous carbon and vacancy-like defects. In contrast the GNS in the sintered composite exhibited enhanced quality with a lower number of defects, and they were wavy, semi-transparent and with very low thickness. The obtained nanocomposite was fully dense with a homogeneous distribution of GNS into the matrix. The Vickers hardness of the nanocomposite showed similar values to those of a monolithic 3YTZP ceramic sintered in the same conditions, and to the reported ones for a 3YTZP composite with the same content of commercial graphene nanosheets.

Development by Mechanochemistry of $La_{0.8}Sr_{0.2}Ga_{0.8}Mg_{0.2}O_{2.8}$ Electrolyte for SOFCs

García-García, FJ; Tang, YQ; Gotor, FJ; Sayagues, MJ
Materials, **13** (2020) 1366
Marzo, 2020 | **DOI:** 10.3390/ma13061366



In this work, a mechanochemical process using high-energy milling conditions was employed to synthesize $La_{0.8}Sr_{0.2}Ga_{0.8}Mg_{0.2}O_{3-\delta}$ (LSGM) powders from the corresponding stoichiometric amounts of La_2O_3, SrO, Ga_2O_3, and MgO in a short time. After 60 min of milling, the desired final product was obtained without the need for any subsequent annealing treatment. A half solid oxide fuel cell (SOFC) was then developed using LSGM as an electrolyte and $La_{0.8}Sr_{0.2}MnO_3$ (LSM) as an electrode, both obtained by mechanochemistry. The characterization by X-ray diffraction of as-prepared powders showed that LSGM and LSM present a perovskite structure and pseudo-cubic symmetry. The thermal and chemical stability between the electrolyte (LSGM) and the electrode (LSM) were analyzed by dynamic X-ray diffraction as a function of temperature. The electrolyte (LSGM) is thermally stable up to 800 and from 900 °C, where the secondary phases of $LaSrGa_3O_7$ and $LaSrGaO_4$ appear. The best sintering temperature for the electrolyte is 1400 °C, since at this temperature, $LaSrGaO_4$ disappears and the percentage of $LaSrGa_3O_7$ is minimized. The electrolyte is chemically compatible with the electrode up to 800 °C. The powder sample of the electrolyte (LSGM) at 1400 °C observed by HRTEM indicates that the cubic symmetry Pm-3m is preserved. The SOFC was constructed using the brush-painting technique; the electrode–electrolyte interface characterized by SEM presented good adhesion at 800 °C.

The electrical properties of the electrolyte and the half-cell were analyzed by complex impedance spectroscopy. It was found that LSGM is a good candidate to be used as an electrolyte in SOFC, with an Ea value of 0.9 eV, and the LSM sample is a good candidate to be used as cathode.

Influence of DSC thermal lag on evaluation of crystallization kinetics

Svoboda, R; Maqueda, LP; Podzemna, V; Perejón, A; Svoboda, O
Journal of Non-Crystalline Solids, **528** (2020) 119738
Enero, 2020 | **DOI:** 10.1016/j.jnoncrysol.2019.119738

Influence of added thermal resistance on crystallization kinetics, as measured by differential scanning calorimetry (DSC), of the Se70Te30 glass was studied. The increase of thermal resistance was achieved by adding polytetrafluorethylene discs of different thicknesses (up to 0.5 mm) in-between the DSC platform and the pan with sample. Increase of the thermal resistance led to an apparent decrease (by more than 30%) in the crystallization enthalpy. Significant change of model-free kinetics occurred: apparent activation energy E of the crystallization process decreased (by more than 20%) due to the DSC data being progressively shifted to higher temperatures with increasing heating rate. The model-based kinetics was changed only slightly; the DSC peaks retained their asymmetry and the choice of the appropriate model was not influenced by the added thermal resistance. The temperature shift caused by added thermal lag was modeled for the low-to-moderate heating rates.

ICTAC Kinetics Committee recommendations for analysis of multi-step kinetics

Vyazovkin, S; Burnham, AK; Favergeon, L; Koga, N; Moukhina, E; Pérez-Maqueda, LA; Sbirrazzuoli, N
Thermochimica Acta, **689** (2020) 178597
Julio, 2020 | **DOI:** 10.1016/j.tca.2020.178597

The present recommendations have been developed by the Kinetics Committee of the International Confederation for Thermal Analysis and Calorimetry (ICTAC). The recommendations provide guidance on kinetic analysis of multi-step processes as measured by thermal analysis methods such as thermogravimetry (TGA) and differential scanning calorimetry (DSC). Ways of detecting the multi-step kinetics are discussed first. Then, four different approaches to evaluation of kinetic parameters (the activation energy, the pre-exponential factor, and the reaction model) for individual steps are considered. The approaches considered include multi-step model-fitting as well as distributed reactivity, isoconversional, and deconvolution analyses. For each approach practical advice is offered on its effective usage. Due attention is also paid to the typical problems encountered and to the ways of resolving them. The objective of these recommendations is to help a non-expert with efficiently performing multi-step kinetic analysis and interpreting its results.

Influence of the Test Configuration and Temperature on the Mechanical Behaviour of WC-Co

González, LM; Chicardi, E; Gotor, FJ; Bermejo, R; Llanes, L; Torres, Y
Metals, **10** (2020) 322
Marzo, 2020 | **DOI:** 10.3390/met10030322

In this work, the effect of the test configuration and temperature on the mechanical behaviour of cemented carbides (WC-Co) with different carbide grain sizes (d_{WC}) and cobalt volume fractions (V_{Co}), implying different binder mean free paths (λ_{Co}), was studied. The mechanical strength was measured at 600 °C with bar-shaped specimens subjected to uniaxial four-point bending (4PB) tests and with disc specimens subjected to biaxial ball-on-three-balls (B3B) tests. The results were analysed within the frame of the Weibull theory and compared with strength measurements performed at room temperature under the same loading conditions. A mechanical degradation greater than 30% was observed when the samples were tested at 600 ^{o}C due to oxidation phenomena, but higher Weibull moduli were obtained as a result of narrower defect size distributions. A fractographic analysis was conducted with broken specimens from each test configuration. The number of fragments (N_f) and the macroscopic fracture surface were related to the flexural strength and fracture toughness of WC-Co. For a given number of fragments, higher mechanical strength values were always obtained for WC-Co grades with higher K_{Ic}. The observed differences were discussed based on a linear elastic fracture mechanics (LEFM) model, taking into account the effect of the temperature and microstructure of the cemented carbides on the mechanical strength.

New waste-based clinkers for the preparation of low-energy cements. A step forward toward circular economy

Martínez-Martínez, S; Pérez-Villarejo, L; Eliche-Quesada, D; Sánchez-Soto, PJ; Christogerou, A; Kanellopoulou, DG; Angelopoulos, GN
International Journal of Applied Ceramic Technology, **17** (2020) 12-21
Enero, 2020 | **DOI:** 10.1111/ijac.13390

This paper describes the use of industrial wastes arising from different production processes of the ceramic and marble industries as raw materials for the design and formulation of new cement clinkers with a high content of dicalcium silicate (Belite). The aim was to reintroduce these wastes in the industrial sector and take advantage of them for a greater environmental benefit, as indicated by the principles of the circular economy. Formulations containing 2.5, 5 and 10 wt% of chamotte and marble sludge, respectively, and a waste-free formulation have been designed to obtain clinkers with a content of dicalcium silicate higher than 60 wt%. The different blends have been studied up to a maximum temperature of 1390 °C by Thermal Analysis. Other techniques such as XRD, XRF, Modified Bogue Equation, Quality Indexes (LSF, AM, SM) and Optical Microscopy have been used for the study and characterization of industrial

wastes, the raw materials and the high belite-type cement dosages. The results indicate that this type of cements can be designed using different types of wastes and in this way reduce the environmental impacts caused by the extraction of raw materials and the deposition of the wastes in landfills, improving the circular economy of the construction industry.

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

Flexure strength and fracture propagation in zirconia ceramic composites with exfoliated graphene nanoplatelets
Á. Gallardo-López, J. Castillo-Seoane, C. Muñoz-Ferreiro, C. López-Pernía, A. Morales-Rodríguez, R. Poyato
Ceramics, 3 (2020) 78-91

La química protagonista de los VII Premios Joven a la Cultura Científica
P.J. Sánchez-Soto
Revista QUÍMICOS DEL SUR, 112 (2020) 74-75

Investigación. Víctor Manuel Pérez Puyana
P.J. Sánchez-Soto
Revista QUÍMICOS DEL SUR, 112 (2020) 76-77

LIBROS Y OTRAS PUBLICACIONES / BOOKS AND OTHER PUBLICATIONS

An overview of clay brick manufacturing from the raw clay materials to the firing products: the case of SW Spain
L. Pérez-Villarejo, S. Martínez-Martínez, B. Carrasco-Hurtado, D. Eliche-Quesada, J.S. Bueno-Rodríguez, A. Christogerou, P.J. Sánchez-Soto
Manufacturing Technology Research: The Fundamentals of Brick Manufacturing, Editorial Nova Science Publishers, New York. Md. Faruque Hossain (Editor), Chapter 3, págs. 51-86.
ISBN: 978-1-53617-224-9

Valorization of Olive Biomass Fly Ash for Production Eco Friendly Ceramic Bricks
L. Pérez-Villarejo, D. Eliche-Quesada, B. Carrasco-Hurtado, P.J. Sánchez-Soto
Encyclopedia of Renewable and Sustainable Materials, Editorial Elsevier, Oxford. Saleem Hashmi and Imtiaz Ahmed (Editores), Vol. 5, págs. 285-294
ISBN: 978-0-12-803581-8

Eco-cement Clinker design valorizing wastes from different industries
S. Martínez-Martínez, L. Pérez-Villarejo, P.J. Sánchez-Soto, D. Eliche-Quesada
New Trends in Green Construction, Editorial Universidad de Córdoba UCO Press, Córdoba. A. Rodero Serrano, J.R. Jiménez Romero, J.Mª. Fernández Rodríguez, E. Fernández-Ledesma (Editores), págs. 137-138.
ISBN: 978-84-9927-554-3

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

4th European Conference on Unsaturated Soils, E-UNSAT 2020,
19-21 octubre [Lisboa, Portugal]

Physical and geotechnical properties of a silty sand soil treated with calcium carbonate fixing bacteria
E. Garzón, L. Morales, J. Reca, E. Romero, P.J. Sánchez-Soto
Comunicación Oral
Publicación como OPEN ACCESS ARTICLE, E3S Web of Conferences, E-UNSAT 2020, R. Cardoso, C. Jommi y E. Romero (Editores), Vol. 195, 05002 (2020)
https://doi.org/10.1051/e3sconf/202019505002

Stabilization to prevent soil erosion using vetiver (Chrysopogon zizanioides L.) in slopes: a field case study of selected grounds at Guatemala

E. Garzón, F. M. González-Miranda, J. Reca, P.J. Sánchez-Soto
Comunicación Oral
Publicación como OPEN ACCESS ARTICLE, E3S Web of Conferences, E-UNSAT 2020, R. Cardoso, C. Jommi y E. Romero (Editores), Vol. 195, 01014 (2020)
https://doi.org/10.1051/e3sconf/202019501014

CONGRESOS Y REUNIONES NACIONALES / NATIONAL CONGRESS AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

LVII Congreso Nacional de la Sociedad Española de Cerámica y Vidrio (SECV)
26-29 octubre [Castellón, España]

Diseño y estudio de clínker de cemento innovador de baja energía con alto contenido en subproductos industriales
S. Martínez-Martínez, L. Pérez-Villarejo, D. Eliche-Quesada, P.J. Sánchez-Soto
Comunicación Póster

FORMACION / TRAINING

FORMACIÓN DE GRADUADOS / MASTER DEGREE THESIS

Título:	**Diseño de electrolitos para pilas de combustible de óxido sólido mejorando la sinterización para promover la densificación**
Autor:	José Juan Galera Rodríguez
Directores:	Francisco José Gotor Martínez
Grado:	Trabajo Fin de Grado
Fecha Defensa:	23 julio 2020

Título:	**Caracterización de compuestos de matriz cerámica y óxido de grafeno reducido**
Autor:	Paloma Luna Garrido
Directores:	Rosalía Poyato Galán y Ana Morales Rodríguez
Grado:	Trabajo Fin de Máster
Fecha Defensa:	9 julio 2020

Título:	**Estado del arte y síntesis mecanoquímica de High Entropy-Ultra High Temperature Ceramics (HE-UHTC)**
Autor:	María Jiménez Fuentes
Directores:	Francisco José Gotor Martínez
Grado:	Trabajo Fin de Master
Fecha Defensa:	9 julio 2020

DOCENCIA / TEACHING

Investigadores de esta unidad participan en el Máster en Ciencia y Tecnología de Nuevos Materiales (página 235)

EQUIPAMIENTO CIENTÍFICO AVAILABLE EQUIPMENT

- Microdurómetro FM700 Future-Tech
- Máquina de ensayo universal AGS-J, Shimadzu
- 3 Termobalanzas CI Electronics
- 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)
- Horno horizontal alta temperatura 1800 ^{o}C AGNI
- Horno horizontal alta temperatura 1600 ^{o}C Carbolite
- Horno horizontal 1200 ^{o}C 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)
- Horno horizontal 1150 ^{o}C Hobersal
- Impedancímetro Agilent 4294ª
- Multímetro Keysight B2901A
- Equipo de Hot-Press TERMOLAB

MATERIALES FUNCIONALES NANOESTRUCTURADOS
NANOSTRUCTURED FUNCTIONAL MATERIALS

GRUPOS DE INVESTIGACIÓN

Materiales Nanoestructurados y Microestructura
Nanostructured Materials and Microstructure [642015]
http://nanomatmicro.icmse.csic.

Materiales para Bioingeniería y Regeneración Tisular
Materials for Bioengineering and Tissue Regeneration [642014]

Nanotecnología en Superficies y Plasma
Nanotechnologies on Surfaces and Plasma [642012]
http://sincaf.icms.us-csic.es/

Tribología y Protección de Superficies
Tribology and Protection of Surfaces [861494]

PERSONAL / PERSONNEL

Profesores de Investigación

Dr. Juan Pedro Espinós Manzorro
Dra. Asunción Fernández Camacho
Dr. Agustín Rodríguez González-Elipe

Catedráticos

Dr. José Cotrino Bautista

Investigadores Científicos

Dr. Ángel Barranco Quero
Dr. Juan Carlos Sánchez López
Dr. Francisco Yubero Valencia

Científicos Titulares

Dra. Ana Isabel Borrás Martos
Dra. María Aránzazu Díaz Cuenca
Dr. Alberto Palmero Acebedo
Dra. T. Cristina Rojas Ruiz

Contratados Ramón y Cajal

Dr. Juan Ramón Sánchez Valencia

Contratados Plan Propio US

Dr. Rafael Álvarez Molina
Dra. Carmen López Santos

Profesores Contratados Doctor

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Dr. Francisco J. Aparicio Rebollo
Dr. Jorge A. Budagosky Marcilla
Dra. Lidia Contreras Bernal
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Personal Investigador en Formación

Ldo. Javier Castillo Seoane
Ldo. Xabier García Casas
Lda. Paula de Navascues Garvin
Ldo. José Manuel Obrero Pérez
Ldo. Noel Orozco Corrales
Ldo. Alvaro Perea Brenes
Lda. Xiaozhe Song

Personal Técnico Contratado

Ldo. Dirk Hufschmidt
Lda. Beatriz Medrán Barranco

PROYECTOS DE INVESTIGACIÓN / RESEARCH PROJECTS

Descongelación inteligente y sostenible mediante ingeniería de ondas acústicas aplicadas a superficies | SOUNDOFICE Sustainable Smart De-Icing by Surface Engineering of Acoustic Waves | SOUNDOFICE

Código/Code:	H2020-FET-OPEN/0717
Periodo/Period:	01-11-2020 / 31-10-2024
Organismo Financiador/Financial source:	Unión Europea
Importe total/Total amount:	690.602 €
Investigador responsable/Research head:	Ana Isabel Borrás Martos
Componentes/Research group:	Agustín R. González-Elipe, Juan Pedro Espinós, Francisco Yubero, Ángel Barranco, Víctor Rico, María del Carmen López Santos

RESUMEN / ABSTRACT

Icing on surfaces is commonplace in nature and industry and too often causes catastrophic events. SOUNDofICE ultimate goal is to overcome costly and environmentally harmful de-icing methods with a pioneering strategy based on the surface engineering of MHz Acoustic Waves for a smart and sustainable removal of ice. This technology encompasses the autonomous detection and low-energy-consuming removal of accreted ice on any material and geometry. For the first time, both detection and de-icing will share the same operating principle. The visionary research program covers the modeling of surface wave atom excitation of ice aggregates, integration of acoustic transducers on large areas, and the development of surface engineering solutions to stack micron-size interdigitated electrodes together with different layers providing efficient wave propagation, anti-icing capacity, and aging resistance. We will demonstrate that this de-icing strategy surpasses existing methods in performance, multifunctionality, and capacity of integration on industrially relevant substrates as validated with proof of concept devices suited for the aeronautic and wind power industries. SOUNDofICE high-risks will be confronted by a strongly interdisciplinary team from five academic centers covering both the fundamental and applied aspects. Two SMEs with first-hand experience in icing will be in charge of testing this technology and its future transfer to key EU players in aeronautics, renewable energy, and household appliances. An Advisory Board incorporating relevant companies will contribute to effective dissemination and benchmarking. The flexibility of the R&D plan, multidisciplinarity, and assistance of the AdB guarantee the success of this proposal, bringing up a unique opportunity for young academia leaders and SMEs from five different countries to strengthen the EU position on a high fundamental and technological impact field, just on the moment when the climate issues are of maxima importance.

Diseño de nanomateriales tridimensionales para la solución todo en uno a la recolección de energía ambiental de fuentes múltiples | 3DSCAVENGERS Three-dimensional nanoscale design for the all-in-one solution to environmental multisource energy scavenging | 3DSCAVENGERS

Código/Code: H2020-ERC-STG/0655
Periodo/Period: 01-03-2020 / 28-02-2025
Organismo Financiador/Financial source: Unión Europea
Importe total/Total amount: 1.498.414 €
Investigador responsable/Research head: Ana Isabel Borrás Martos

RESUMEN / ABSTRACT

Thermal and solar energy as well as body movement are all sources of energy. They can be exploited by advanced technology, obviating the need for battery recharging. These local ambient sources of energy can be captured and stored. However, their low intensity and intermittent nature reduces the recovery of energy by microscale instruments, highlighting the need for an integrated multisource energy harvester. Existing methods combine different single source scavengers in one instrument or use multifunctional materials to concurrently convert various energy sources into electricity.

The EU-funded 3DScavengers project proposes a compact solution based on the nanoscale architecture of multifunctional three-dimensional materials to fill the gap between the two existing methods. These nanoarchitectures will be able to simultaneous and individual harvesting from light, movement and temperature fluctuations. 3DScavengers ultimate goal is to apply a scalable and environmental friendly one-reactor plasma and vacuum approach for the synthesis of this advanced generation of nanomaterials.

Tecnología de plasma para la fabricación de celdas solares de perovskita eficientes y duraderas a prueba de agua Plasma technology for efficient and DURAble waterproof perovskite SOLar cells

Código/Code: PID2019-109603RA-I00
Periodo/Period: 01-06-2020 / 31-05-2023
Organismo Financiador/Financial source: Ministerio de Ciencia, Innovación y Universidades
Importe total/Total amount: 96.800 €

Investigador responsable/Research head: Juan Ramón Sánchez Valencia y María del Carmen López Santos

Componentes/Research group: Juan Pedro Espinós Manzorro, Javier Castillo Seoane, Xabier García Casas, Víctor López Flores

RESUMEN / ABSTRACT

Las celdas solares –dispositivos que transforman directamente la luz solar en electricidad- son de vital interés para el futuro sostenible del planeta. Durante los últimos años y conscientes de este hecho, la comunidad científica ha realizado un gran esfuerzo por mejorar la eficiencia de estos dispositivos. Un ejemplo particular de celda solar que contiene una perovskita de haluro organometálico como absorbedor de luz han centrado la atención de la comunidad científica durante la última década debido, sobre todo, a su alta eficiencia y bajo coste. Esta tecnología de celda solar supone una alternativa prometedora a las celdas actuales (basadas en Si y en calcogenuros), aunque se enfrentan a un reto científico y tecnológico que no ha sido resuelto en 10 años desde su descubrimiento: para que la realización final y comercial de las celdas de perovskita sea posible, necesitan alcanzar una mayor estabilidad, durabilidad y reproducibilidad. El principal problema radica en la alta sensibilidad que presentan estas perovskitas al oxígeno y humedad ambiental, que producen una rápida degradación del comportamiento de la celda en un tiempo extremadamente corto, haciendo inviable su comercialización.

DuraSol persigue abordar este gran reto científico y tecnológico mediante la fabricación de componentes de la celda mediante tecnología de vacío y plasma. Estas metodologías son escalables industrialmente y presentan grandes ventajas con respecto a las metodologías en disolución (las más usadas), entre las que destacan: su alta versatilidad, control de composición y microestructura, bajo coste, que son respetuosas con el medio ambiente ya que no precisan disolventes, no producen emisiones contaminantes y son compatibles con la tecnología actual de semiconductores.

El objetivo principal de DuraSol es la fabricación de celdas solares de perovskita “a prueba de agua” mediante integración de componentes fabricados por metodologías de vacío y plasma en forma de películas delgadas y nanoestructuras, que actúan como sellantes hidrofóbicos. La viabilidad de DuraSol se basa en resultados recientes que demuestran que la fabricación asistida por plasma de distintos componentes de la celda solar puede ser una de las vías más prometedoras para aumentar su estabilidad y durabilidad, que es hoy en día el cuello de botella que impide su comercialización. Cabe señalar que no hay ningún ejemplo en la literatura de este enfoque sintético, y se espera que esta oportunidad demuestre las ventajas y la versatilidad de esta metodología innovadora en un campo de muy alto impacto. La investigación propuesta en DuraSol se enmarca dentro de las áreas prioritarias del programa Horizon 2021-2027 de la Unión Europea y responden a varios de los retos propuestos en la presente convocatoria de “Energía segura, eficiente y limpia” (Reto 3) y de “Cambio climático y utilización de recursos y materias primas” (Reto 5).

Solar cells – devices that transform sunlight into electricity – are of vital interest for the sustainable future of the planet. During the last years and aware of this fact, the scientific community has made a great effort to improve the efficiency of these devices. A particular example of a solar cell that contains an organometallic halide perovskite as light absorber has focused the attention of the scientific community during the last decade due, above all, to its

high efficiency and low cost. This solar cell technology is a promising alternative to currently existing ones (based on Si and chalcogenides), although they face a scientific and technological challenge that has not been solved in 10 years since its discovery: for the commercial realization of the perovskite cells possible, they need to achieve higher stability, durability and reproducibility. The main problem lies in the high sensitivity of these perovskites to oxygen and environmental humidity, which produce a rapid degradation of the cell's behaviour in an extremely short time, making commercialization unfeasible.

DuraSol seeks to address this great scientific and technological challenge by manufacturing cell components using vacuum and plasma technology. These methodologies are industrially scalable and present great advantages over solution methods (the most used), among which are: their high versatility, control of composition and microstructure, low cost, environmentally friendly since they do not require solvents, do not produce pollutant emissions and are compatible with current semiconductor technology.

The main objective of DuraSol is the fabrication of waterproof perovskite solar cells by integrating components manufactured by vacuum and plasma methodologies in the form of thin films and nanostructures, which act as hydrophobic sealants. The viability of DuraSol is based on recent results that demonstrate that plasma-assisted synthesis of different components of the solar cell can be one of the most promising ways to increase its stability and durability, which is today the bottleneck that prevents their commercialization. It is worth to highlight that there is no example in the literature about this synthetic approach, and this opportunity is expected to demonstrate the advantages and versatility of this innovative methodology in a field of very high impact. The research proposed in DuraSol falls within the priority areas of the European Union Horizon 2021-2027 program and responds to several of the challenges proposed in this call for "Energía segura, eficiente y limpia" (Challenge 3) and "Cambio climático y utilización de recursos y materias primas" (Challenge 5).

Estructuras adaptativas multiresponsivas para fotónica integrada, piezo/tribotronica y monitorización optofluídica | AdFunc

Adaptive multiresponsive nanostructures for integrated photonics, piezo/tribotronics and optofluidic monitoring | AdFunc

Código/Code:	PID2019-110430GB-C21
Periodo/Period:	01-06-2020 / 31-05-2023
Organismo Financiador/Financial source:	Ministerio de Ciencia, Innovación y Universidades
Importe total/Total amount:	211.750 €
Investigador responsable/Research head:	Ángel Barranco Quero y Ana Isabel Borrás Martos
Componentes/Research group:	José Cotrino Bautista, Víctor J. Rico Gavira, Francisco Yubero Valencia, Juan Pedro Espinós Manzorro, Agustín R. González-Elipe

RESUMEN / ABSTRACT

AdFUNC es un proyecto muy interdisciplinar que tiene como principal objetivo conseguir un progreso significativo en dos temáticas en la frontera de la Ciencia de Materiales: el desarrollo de sensores con capacidad multirespuesta y de sistemas de energía activados por luz. Los denominadores comunes de AdFUNC son el diseño inteligente de arquitecturas complejas en la nanoescala y el desarrollo de demostradores a escala de laboratorio.

Estamos convencidos de que el proyecto nos abre una ventana de oportunidad para realizar investigaciones que podemos clasificar en cuatro áreas: i) Aplicaciones y dispositivos: Desarrollaremos los recientemente descubiertos efectos tribotrónicos y piezotrónicos para fabricar dispositivos sensores autoalimentados. Con estos materiales, en combinación con varias tecnologías avanzadas de sensado fotónico y espectro-electroquímico, expandiremos la eficiencia, multiactuación y multirespuesta de sistemas adaptativos optofluídicos. Estos sistemas, manteniendo una arquitectura común, presentarán una respuesta diferenciada frente a escenarios reales diversos y complejos, que se simularán en el proyecto (alteraciones medioambientales como vertidos, accidentes, amenazas químicas o de explosivos). También se plantean dispositivos captadores de energía solar en condiciones de baja iluminación, captadores de energía mecánica y dispositivos que sean capaces de acoplar luz y movimiento a la activación de procesos de descomposición electroquímica del agua. Ii) Nanomateriales: Adfunc es un proyecto donde concurren un equipo de especialistas en el desarrollo de nanoestructuras soportadas por distintas tecnologías. Esto nos permitirá, por primera vez, implementar un conjunto de nanoarquitecturas 3D (nanohilos, nanotubos, core@shell) y el diseño de materiales con estructuras nanoporosas controladas (capas esculturales, nanocanales, porosidad asociada en varias escalas, multicapas ópticas porosas, desarrollos pioneros de redes metaloorgánicas (MOFs) en estructuras fotónicas porosas) directamente a la mejora de los componentes activos de los dispositivos del proyecto. Iii) Estrategia. El proyecto nos da la oportunidad de trabajar simultáneamente en rutas sintéticas nuevas, caracterización avanzada de materiales y propiedades, integración de materiales en dispositivos, y esto a la vez que se tiene información de modelado y simulación. Iv) Perspectiva de escalabilidad: En todos los casos se utilizarán métodos y técnicas compatibles con procesos industriales establecidos, como el plasma y el vacío típicos de la industria optoelectrónica y microelectrónica, y procesos de síntesis en disolución. Otro aspecto interesante, es la posibilidad de introducir plásticos y polímeros para fabricar dispositivos, lo que puede permitir revalorizar residuos de la industria del plástico, en un esfuerzo de economía circular en el que investigadores del proyecto están comprometidos.

AdFunc sólo es posible gracias al esfuerzo conjunto de un gran número de investigadores, en su mayoría del ICMS-CSIC y la Universidad Pablo de Olavide, que se completa con un grupo de investigadores de otros centros y colaboradores internacionales con experiencia e interés complementarios. Es precisamente la coordinación de un número tan elevado de especialistas (25 doctores en los dos subproyectos) lo que nos permite plantear el desarrollo de un conjunto de actividades tan completo y ambicioso. --

AdFUNC is a highly interdisciplinary project whose main objective is to achieve significant progress in two areas at the frontier of Materials Science: the development of multi-response sensors and light-activated energy systems. The common denominators of AdFUNC are the intelligent design of complex architectures at the nanoscale and the development of laboratory scale demonstrators.

We are convinced that the project opens a window of opportunity for us to carry out research that can be classified into four areas: i) Applications and devices: We will develop the recently discovered tribotronic and piezotronic effects to manufacture self-powered sensor devices. With these materials, in combination with several advanced photonic sensing and spectro-electrochemical technologies, we will expand the efficiency, multiactuation and multiresponse of optofluidic adaptive systems. These systems, maintaining a common architecture, will present a differentiated response to diverse and complex real scenarios, which will be simulated in the project (environmental alterations such as spills, accidents, chemical or explosive threats).

Another fundamental aspect of the project are the photovoltaic devices, which will be optimized to be able to work in low light conditions, and mechanical energy collectors and devices that are capable of coupling light and movement to the activation of the water electrochemical decomposition. ii) Nanomaterials: Adfunc is a project where a team of specialists in the development of supported nanostructures by different technologies come together. This will allow us, for the first time, to implement a set of 3D nanoarchitectures (nanowires, nanotubes, core@shell) and the design of materials with controlled nanoporous structures (sculptural layers, nanochannels, porosity associated in several scales, porous optical multilayers, pioneering developments of metalloorganic networks (MOFs) in porous photonic structures) directly to the improvement of the active components of the project devices. Iii) Strategy: The project gives us the opportunity to work simultaneously on new synthetic routes, advanced characterization of materials and properties, integration of materials into devices, and this while simultaneously obtaining modeling and simulation information. iv) Perspective of scalability: In all cases, methods and techniques compatible with established industrial processes will be used, such as plasma and vacuum, typical of the optoelectronic and microelectronic industry, and synthesis processes in solution. Another interesting aspect is the possibility of introducing plastics and polymers to manufacture devices, which may allow the valorization of waste from the plastic industry, in an effort of circular economy in which researchers of the project are committed.

AdFunc is only possible thanks to the joint effort of a large number of researchers, mostly from ICMS-CSIC and the Pablo de Olavide University, which is completed by a group of researchers from other national and international institutions with complementary experience and interest. It is precisely the coordination of such a large number of specialists (25 doctors in the two subprojects) that allows us to propose the development of such a complete and ambitious set of activities.

Recubrimientos innovadores preparados por magnetron sputtering para absorción solar selectiva | MAGICOS2 Magnetron sputtered innovative coatings for solar selective absorption | MAGICOS2

Código/Code:	PID2019-104256RB-I00
Periodo/Period:	01-06-2020 / 31-05-2024
Organismo Financiador/Financial source:	Financiador: Ministerio de Ciencia, Innovación y Universidades
Importe total/Total amount:	121.000 €
Investigador responsable/Research head:	Juan Carlos Sánchez López y Ramón Escobar Galindo (US)
Componentes/Research group:	Cristina Rojas Ruiz, Belinda Sigüenza Carballo

RESUMEN / ABSTRACT

El cambio climático ocasionado por las emisiones de gases con efecto invernadero y el agotamiento de los combustibles fósiles a corto-medio plazo hacen necesaria la búsqueda de nuevas fuentes de energía alternativas, limpias y renovables. De entre ellas, la energía solar es una de las mejores opciones por su gran disponibilidad para la generación de calor y electricidad.

El objetivo de este proyecto va encaminado al desarrollo de nuevos recubrimientos absorbedores solares selectivos crecidos en forma de multicapas basados en nitruros metálicos de cromo y aluminio (CrAlN). Las propiedades de resistencia a la oxidación y estabilidad térmica del CrAlN unidas a un diseño nanoestructurado adecuado permitirán mantener unas buenas prestaciones ópticas (alta absorbancia y baja emitancia) y mejorar su durabilidad a alta temperatura. El incremento de la temperatura de trabajo (T>550 ^{o}C) conllevará una mejora de la eficiencia y una reducción de costes de las plantas de concentración de solar térmica, haciéndolas más competitivas. Para su preparación se utilizará la técnica de pulverización catódica mediante impulsos de alta intensidad (HiPIMS), una variante reciente de la pulverización catódica convencional que permite mejorar la densidad y compacidad de las capas gracias a un mayor grado de ionización del plasma. Estas propiedades son de interés para mejorar la adhesión al sustrato y ralentizar los procesos de degradación térmica. Además de los nitruros se ensayarían otras configuraciones cambiando el tipo de material absorbedor (oxinitruros y nanocomposites de carburos metálicos).

El proyecto comprenderá todas las etapas, desde la síntesis de los materiales componentes de las estructuras solares selectivas, diseño y simulación de su comportamiento óptico, a su validación en condiciones similares a la aplicación final (a nivel de laboratorio y ensayos de campo). La caracterización estructural, química y de estabilidad térmica y resistencia a la oxidación discurrirá en paralelo con el fin de optimizar los recubrimientos solares selectivos con mejores prestaciones y durabilidad.

The climatic change produced by the gas pollutants emissions and the greenhouse effect along to the short mid-term depletion of the energy fosil fuels make necessary the search of

alternative energy sources, clean and renewable. Among them, the solar energy is one the best options due to the major availability to generate heat and electricity.

The goal of the present project is the development of new solar multilayered absorber coatings based on chromium and aluminium nitride (CrAlN). The good oxidation resistance and thermal stability of CrAlN, together with a nanostructured design will ensure a good optical performance (high absorbance and low emissivity) and increase their durability at high temperature. The increment of the working temperature (T>550°C) will improve the efficiency and reduce the costs of the solar thermal power plants, make them more competitive. The High Power Impulse Magnetron Sputtering technique (HiPIMS) will be used for the preparation of the coatings. This recent innovation of the conventional magnetron sputtering technology allows increasing the film density and compactness thanks to an increased ionization of the plasma. These properties are interesting for the improvement of the adhesion to the substrate and decrease the thermal degradation. In addition to abovementioned strategy, other alternative configurations changing the nature of the material absorber (metal oxynitrides and carbides nanocomposites) would be tried.

The project will comprise all the stages, from the synthesis of the material components of the solar selective structures, design and simulation of the optical behaviour, to the validation in conditions similar to the final application (both in lab and field tests). The structural and chemical characterization, the evaluation of the thermal stability and oxidation resistance will run simultaneously with the aim of optimizing the solar absorber selective coatings with the best performance and durability.

MINISTERIO DE CIENCIA, INNOVACIÓN Y UNIVERSIDADES

FEDER Fondo Europeo de Desarrollo Regional

UNIÓN EUROPEA "Una manera de hacer Europa"

Desarrollo de catalizadores y soportes para procesos de almacenamiento químico de energía neutros en CO_2 basados en líquidos orgánicos portadores de hidrógeno Development of catalysts and supports for CO_2 neutral chemical energy storage processes based on liquid organic hydrogen carriers

Código/Code:	RTI2018-093871-B-I00
Periodo/Period:	01-01-2019 / 31-12-2021
Organismo Financiador/Financial source:	Ministerio de Ciencia, Innovación y Universidades
Importe total/Total amount:	176.176 €
Investigador responsable/Research head:	Asunción Fernández Camacho
Componentes/Research group:	Mª Carmen Jiménez de Haro

RESUMEN / ABSTRACT

El agotamiento de los combustibles fósiles (a corto y largo plazo) y el calentamiento global derivado del efecto invernadero son consecuencias del uso extensivo de estos

combustibles. Por lo tanto, es muy conveniente utilizar y desarrollar energías renovables y así eliminar nuestra dependencia de los combustibles fósiles. Esto hace que el almacenamiento de energía producida por fuentes renovables (que son intermitentes) sea un objetivo importante de investigación. En proyectos anteriores, hemos trabajado en el estudio de nanomateriales y catalizadores para el almacenamiento de hidrógeno como vector de transporte y almacenamiento de energía (ciclo del H_2). En este nuevo proyecto, el grupo de investigación propone avanzar en la Implementación de líquidos orgánicos como portadores de hidrógeno (LOHC) como una forma prometedora de combinar los ciclos del CO_2 y del H_2 que conduzca a un almacenamiento de energía sostenible en un ciclo neutro en carbono. Pequeñas moléculas orgánicas, como el ácido fórmico o el metanol, se pueden usar para almacenar el H_2 (y la energía) proveniente de fuentes renovables. Estos combustibles alternativos se pueden quemar o usarse para generar H_2 que alimente directamente a una pila de combustible.

En este proyecto se llevarán a cabo investigaciones para la implementación de dos procesos relacionados con las tecnologías LOHC:

i) La descomposición térmica selectiva del ácido fórmico por catálisis heterogénea para la producción bajo demanda de hidrógeno exento de monóxido de carbono.

ii) La producción de hidrógeno por reformado de alcoholes (biometanol) en procesos fotocatalíticos heterogéneos. La catálisis desempeña un papel clave en la implementación de estos dos procesos. Por lo tanto, los principales objetivos y actividades del proyecto son el diseño racional y la preparación de catalizadores y soportes para estudiar las relaciones composición-estructura desempeño en los dos procesos mencionados anteriormente. El enfoque innovador es la aplicación de técnicas asistidas por plasma, como la pulverización catódica para el crecimiento de películas delgadas, y los tratamientos con plasmas de oxidación, reducción y grabado, para el desarrollo de recubrimientos catalíticos nanoestructurados y nanopartículas soportadas. Se desarrollarán espumas de carbono poroso y catalizadores basados en Pd que incluyen Pd, Pd-C, Pd-B o Pd-Cu para el estudio de la reacción de descomposición de ácido fórmico. Se investigarán películas fotocatalíticas de TiO_2-TiO_x con Pt (y/o Au) como co-catalizadores para el foto-reformado de metanol.

The depletion of fossil fuels (in short and long term) and the global warming derived from greenhouse eHect are consequences of the extensive use of these fuels. It is therefore highly desirable to use and develop renewable energies and so eliminate our dependence on fossil tuels. This makes the storage of energy produced by renewable sources (which are ¡ntermittent) an important target. In previous projects we have been working in the study of nanomaterials and catalysts for the storage of hydrogen as a vector of energy transport and storage (H_2 cycle). In this new project the research group propose to move into the implementatlon of the liquid organic hydrogen carriers (LOHC) as a promising way of comblning the CO_2 and de H_2 cycles leading to a sustainable energy storage in a carbon neutral cycle.

Small organic molecules, IIke formic acid or methanol, can be used to store the H_2 (and energy) coming from renewable sources. These alternative fuels can be combusted themselves or be used to generate H_2 directly feeding a fuel cell.

Research will be conducted in this project to the implementation of two processes related to the LOHC technologies:

i) The selective low temperature decomposition of formic acid by heterogeneous catalysis to the on-demand production of carbon monoxide free hydrogen.

ii) The hydrogen production by reforming of alcohols (i.e. biomethanol) in heterogeneous photocatalytic processes.

Catalysis is playing the key role in the implementation of these Iwo processes. Therefore the main objectives and activities in the project are the rational design and preparation of catalysts and supports to study composition-structure-performance relationships for the two aboye mentioned processes. The innovative approach is the application of plasma assisted techniques, like the magnetron sputtering for

thin film growth, as well as plasma treatments of oxidation, reduction and etchlng for the development of nanostructured catalytic coatings and supported nanoparticles. Porous carbon foams supports and Pd based catalysts including Pd, Pd-C, Pd-B or Pd-Cu will be developed for the study of the formic acid decomposition reaction. TiO_2-TiO_x photocatalytic films with Pt (and/or gold) as co-catalysts will be investlgated for the photo-reforming 01 methanol.

Arquitecturas de multicapas nanostructuradas para el desarrollo de dispositivos optofluídicos sensores y procesos de funcionalización superficial avanzada (NANOFLOW)

Nanostructured multilayered architectures for the development of optofluidic responsive devices, smart labers, and advanced Surface functionalization (NANOFLOW)

Código/Code:	MAT2016-79866-R
Periodo/Period:	30-12-2016 / 29-06- 2020
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	332.750 €
Investigador responsable/Research head:	Ángel Barranco Quero / Francisco Yubero Valencia
Componentes/Research group:	Ana Isabel Borrás Martos, Juan Pedro Espinós Manzorro, Fabián Frutos Rayego (US), Germán de la Fuente Leis (ICMA), Fernando Lahoz Zamarro (IPNA), Ricardo Molina Mansilla (IQAC), Alberto Palmero Acebedo, Víctor Joaquin Rico Gavira, Agustín R. González-Elipe

RESUMEN / ABSTRACT

NANOFlow es un projecto multidisciplinar que persigue el desarrollo de nuevos dispositivos optofluídicos mediante la integración de materiales nanostructurados multifuncionales. El proyecto está sólidamente fundamentado en la experiencia de los componentes del grupo de investigación en campos como la síntesis de películas multifuncionales, procesos avanzados de modificación de superficies y en el desarrollo de dispositivos fotónicos multicapa. El objetivo principal de este proyecto de investigación es combinar e integrar los distintos métodos avanzados de síntesis y procesado disponibles en la

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

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

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

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

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

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

Junta de Andalucía

Unión Europea
Fondo Europeo de Desarrollo Regional

Andalucía se mueve con Europa

Modelado e implementación de la técnica Freeze-Casting: gradientes de porosidad con un equilibrio tribo-mecánico y comportamiento celular electro-estimulado Modeling and implementation of the freeze casting technique: gradients of porosity with a tribo-mechanical equilibrium and electro-stimulated cellular behavior

Código/Code:	US-1259771
Periodo/Period:	01-02-2020 / 31-01-2022
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	90.000 €
Investigador responsable/Research head:	Yadir Torres Hernández (US) y Juan Carlos Sánchez López
Componentes/Research group:	Ana María Beltrán Custodio, Alberto Olmo Fernández, Paloma Trueba Muñoz, María de los Ángeles Vázquez Gámez

RESUMEN / ABSTRACT

El titanio comercialmente puro (Ti c.p.) y la aleación Ti6Al4V, son los biomateriales metálicos con el mejor pronóstico para la reparación clínica del tejido óseo. Sin embargo, a pesar de sus ventajas, 5-10% de los implantes fallan durante los cinco años post-implantación. Éstos se asocian fundamentalmente al apantallamiento de tensiones (diferencias de rigidez entre el implante-hueso), el empleo de criterios de diseño (fractura y fatiga) no adecuados para biomateriales, a los fenómenos de tribo-corrosión en condiciones de servicio y a los problemas que ocurren en la intercara (micromovimientos y/o presencia de bacterias) que limitan la

capacidad de oseintegración. En este proyecto se propone fabricar e implementar un dispositivo sencillo y económico para obtener cilindros con porosidad controlada (gradiente) y alargada mediante la técnica de congelación dirigida. Se desarrollaran modelos de elementos finitos para estimar el crecimiento geométrico de las dendritas de hielo y el comportamiento mecánico de los cilindros porosos (distribución de esfuerzos y deformaciones), usando radiografías en tiempo real del proceso de congelación dirigida, así como los parámetros que caracterizan la microestructura (proporción, tamaño, morfología de la porosidad) y el comportamiento a compresión (rigidez y límite de fluencia). Además, se plantea la generación de patrones de rugosidad superficial mediante el bombardeo de iones, encaminados a mejorar la unión intima entre el implante y el tejido óseo. Por otra parte, se plantean protocolos in-vitro adecuados para evaluar la citotoxicidad, la adhesión, diferenciación y proliferación celular. Finalmente, se desarrollará un sistema de medida de bio-impedancia que permita racionalizar la influencia de la porosidad, el acabado superficial y los estímulos eléctricos en el comportamiento in-situ de osteoblastos. En este contexto, el objetivo principal es fabricar cilindros con una porosidad controlada y su superficie modificada, que permita garantizar un mejor equilibrio biomecánico, tribo-corrosivo y biofuncional (in-growth y oseointegración del tejido óseo y el implante).

Commercial pure Titanium (c.p. Ti) and Ti6Al4V alloy are metal biomaterials with the best properties for clinical repair bone tissue. However, despite their advantages, 5-10 % of implants fail during the five years post-implantation. They are mainly associated with stress shielding (difference stiffness between bone and implant), the use of design criteria (fracture and fatigue) not suitable for biomaterials, the tribo-corrosion phenomena in service conditions and the interface problems (micro-movements and / or the presence of bacteria) that limit the capacity of osseointegration. This project proposes the manufacture and implementation of a simple and economical device to obtain cylinders with controlled (gradient) and elongated porosity by the freeze casting technique. Finite element models will be developed to estimate the geometric growth of the ice dendrites and the mechanical behaviour of the porous cylinders (distribution of stresses and deformations), using real-time radiographs of the directed freezing process, as well as the parameters that characterize the microstructure (amount, size and morphology of porosity) and compression behaviour (stiffness and yield strength). In addition, the generation of surface roughness patterns by ion sputtering is proposed, with the aim to improve the close bond between the implant and the bone tissue. Furthermore, suitable in-vitro protocols are proposed to evaluate cytotoxicity, adhesion, differentiation and proliferation cell. Finally, a bio-impedance measuring system will be developed in order to rationalize the influence of porosity, finished surface and electrical stimulus on the in-situ behaviour of osteoblasts. In this context, the main objective is to manufacture cylinders with a controlled porosity and modified surface, with enhanced biomechanical, tribo-corrosive and biofunctional balance (in-growth and osseointegration of the bone tissue and the implant).

Tecnología de plasma para el desarrollo de una nueva generación de conductores de huecos en celdas solares de perovskita. PlasmaCells

Plasma technology for the development of a new generation of hole transport layers in perovskite solar cells

Código/Code:	US-1263142
Periodo/Period:	01-02-2020 / 31-01-2022
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	30.000 €
Investigador responsable/Research head:	Juan Ramón Sánchez Valencia
Componentes/Research group:	Ángel Barranco Quero, Juan Pedro Espinós Manzorro, Cristina Rojas Ruiz, José Cotrino Bautista

RESUMEN / ABSTRACT

Las celdas solares (CSs) de tercera generación son dispositivos nanotecnológicos que convierten directamente la luz solar en electricidad y suponen el paradigma de la investigación en energías renovables de cuyo aprovechamiento dependerá el futuro energético del planeta. Recientemente, un ejemplo particular de CSs que contienen una perovskita de haluro organometálico como absorbedor de luz han centrado la atención de la comunidad científica debido, ante todo, a su alta eficiencia y bajo coste. Estas características las convierten en una alternativa prometedora a las celdas actuales (de Si y calcogenuros). Sin embargo, para que la realización final y comercial de las celdas de perovskita sea posible es necesario que alcancen una mayor estabilidad, durabilidad y reproducibilidad. Los avances más importantes alcanzados se han debido a la intensa investigación sobre los elementos que integran esta CS: conductor de electrones, perovskita y conductor de huecos. En concreto, este último elemento ha tenido una importancia crucial en su evolución tras la implementación de los conductores de huecos en estado sólido.

PlasmaCells persigue abordar por primera vez la síntesis de una nueva familia de conductores de huecos por técnicas de vacío y plasma. Estas metodologías son escalables industrialmente y presentan grandes ventajas con respecto a las metodologías en disolución (las más usadas), entre las que destacan: su alta versatilidad, control de composición y microestructura, bajo coste, que son respetuosas con el medio ambiente ya que no precisan disolventes, no producen emisiones contaminantes y son compatibles con la tecnología actual de semiconductores.

El objetivo principal de PlasmaCells es la integración de estos nuevos conductores de huecos procesados por plasma en CSs de perovskita. La importancia del proyecto se basa en resultados recientes obtenidos por el Investigador Principal (IP) que demuestran que la aproximación propuesta puede ser una de las vías más prometedoras para el aumento de la estabilidad, durabilidad y reproducibilidad de estas CSs, que actualmente suponen el cuello de botella que impide su industrialización. Cabe destacar que no existe en la bibliografía ningún ejemplo sobre esta aproximación sintética para el desarrollo de conductores de huecos. Se

espera que esta oportunidad permita demostrar las ventajas y versatilidad de esta metodología innovadora en un campo de alto impacto, que se enmarca dentro de las áreas prioritarias RIS3 Andalucía y en el PAIDI 2020 de crecimiento sostenible, eficiencia energética y energías renovables.

Third generation solar cells (SCs) are nanotechnological devices that directly convert sunlight into electricity and represent the paradigm of research in renewable energies, the use of which will depend on the energy future of the planet. Recently, a particular example of SCs containing an organometallic halide perovskite as a light absorber have attracted the attention of the scientific community due, above all, to their high efficiency and low cost. These characteristics make them a promising alternative to current cells (Si and chalcogenides). However, for the commercial realization of perovskite cells, it is necessary to achieve greater stability, durability and reproducibility. The most important advances have been achieved due to the intense research on the elements that integrate a SC: electron transport layer, perovskite and hole transport layer. Specifically, this latter element has been crucial for its evolution after the implementation of solid state hole conductors.

PlasmaCells pursuits to address for the first time the synthesis of a new family of hole transporters by vacuum and plasma techniques. These methodologies are industrially scalable and have great advantages over solution methodologies (the most used), among which stand out: their high versatility, composition and microstructural control, low cost, are environmental friendly since they do not require solvents, do not produce polluting emissions and are compatible with current semiconductor technology.

The main objective of PlasmaCells is the integration of these new plasma-processed hole transport layers into perovskite SCs. The importance of the project is based on recent results obtained by the Principal Investigator (PI) that demonstrate that the proposed approach may be one of the most promising ways to increase the stability, durability and reproducibility of these SCs, which currently represent the bottleneck that prevents their industrialization. It should be noted that there is no example in the literature of this synthetic approach for the development of hole transporters. It is expected that this opportunity will allow to demonstrate the advantages and versatility of this innovative methodology in a high-impact field, which is framed within the priority areas RIS3 Andalucia and in the PAIDI 2020 of sustainable growth, energy efficiency and renewable energies.

Nuevos recubrimientos nanoestructurados para absorción eficiente de la radiación solar en dispositivos de concentración

New nanostructured coatings for efficient absorption of solar radiation in concentrated devices

Código/Code:	P18-RT-2641
Periodo/Period:	01-02-2020 / 31-01-2022
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	102.268 €
Investigador responsable/Research head:	Juan Carlos Sánchez López
Componentes/Research group:	Cristina Rojas Ruiz, Belinda Sigüenza Carballo

RESUMEN / ABSTRACT

La mejora de los materiales empleados en los dispositivos usados en el campo de las energías renovables permitirá incrementar la eficiencia de los mismos haciéndolos más competitivos y rentables. El presente proyecto pretende desarrollar nuevos recubrimientos absorbedores selectivos de la energía solar aptos para trabajar a temperaturas superiores a las posibles con los materiales actualmente en uso en dispositivos de concentración solar térmica (500 ^{o}C en vacío – media concentración; 800 ^{o}C al aire – alta concentración). Los sistemas serán preparados en forma de multicapas por la novedosa tecnología de pulverización catódica donde los materiales son evaporados mediante impulsos de alta energía (HiPIMS - High Power Impulse Magnetron Sputtering). Los materiales preparados deberán cumplir los requisitos ópticos y de estabilidad química para soportar las condiciones de alta irradiación solar y temperaturas de trabajo. Este ambicioso proyecto se llevará a cabo mediante la colaboración de dos grupos de investigación pertenecientes al Instituto de Ciencia de Materiales de Sevilla CSIC-ICMS (grupo TEP958) y a la plataforma solar de Almería CIEMAT-PSA (Grupo TEP247). El grupo CSIC-ICMS se encargará del diseño, preparación y caracterización de los recubrimientos. Por su parte CIEMAT-PSA, diseñará y desarrollará los ensayos de campo, validando los recubrimientos en condiciones de trabajo similares a las de la aplicación final en términos de flujo solar concentrado incidente y temperaturas de operación. Dichos ensayos incluirán tanto determinación de parámetros térmicos y ópticos en condiciones nominales de operación, así como ciclado térmico de alta frecuencia (tratamiento térmico y envejecimiento).

The improvement of the materials employed in the devices used in the renewable energy sector will enable to increase the efficiency of these systems to become more competitive and profitable. The current project aims to develop new solar selective coatings able to operate at temperatures beyond the working temperature limits of the materials currently being used in concentrated solar systems (500 ^{o}C in vacuum- mid concentration; 800 ^{o}C in air –high concentration). The systems will be prepared in the form of multilayers using the novel technology of magnetron sputtering where the materials are evaporated by means of high energy pulses (HiPIMS - High Power Impulse Magnetron Sputtering). The developed materials should fulfill the optical requirements and thermal stability to withstand the high solar irradiance flux and working temperatures. This project will be carried out through the collaboration of two

research groups belonging to the "Instituto de Ciencia de Materiales de Sevilla", CSIC-ICMS (TEP958 group) and the "Plataforma Solar de Almería", CIEMAT-PSA (TEP247 group). The ICMS-CSIC group will perform the design, preparation and characterization of the coatings. Meanwhile, the CIEMAT-PSA group will be in charge of designing the bench tests, validating the coatings in working conditions similar to the final application in terms of high incident solar flux and operation temperatures. Such tests will include both the determination of thermal and optical parameters in nominal operating conditions, as well as the thermal cycling at high frequency (thermal treatment and aging).

Recubrimientos termocrómicos inteligentes para la climatización eficiente y el control ambiental (TOLERANCE) Smart thermochromic coatings for smart windows and environmental control (TOLERANCE)

Código/Code:	P18-RT-2641
Periodo/Period:	01-01-2020 / 31-12-2022
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	119.800 €
Investigador responsable/Research head:	Ángel Barranco Quero y Alberto Palmero Acebedo
Componentes/Research group:	Ana María Gómez Ramírez, Juan Ramón Sánchez Valencia, Víctor J. Rico Gavira, Rafael Álvarez Molina, Francisco Yubero Valencia, Juan Pedro Espinós Manzorro, Ana Isabel Borrás Martos, Agustín R. González-Elipe

RESUMEN / ABSTRACT

La Agencia Internacional de la Energía considera que el uso sistemático de procedimientos autónomos de control ambiental representa una de las mejores apuestas tecnológicas para reducir el consumo energético asociado a la climatización de edificios (más del 40% del consumo global en países desarrollados, muy superior al porcentaje debido al transporte), reduciendo el impacto ambiental y mejorando además el confort habitacional. TOLERANCE persigue introducir y desarrollar en Andalucía la tecnología de los recubrimientos termocrómicos como elemento inteligente y autónomo de control de la irradiación solar en edificios. El interés de la propuesta se centra en nichos de aplicación como el cerramiento de edificios, el mobiliario urbano, la mejora de sistemas de producción de agua caliente sanitaria o la mejora de invernaderos. Un recubrimiento termocrómico se caracteriza por transmitir todo el espectro solar a bajas temperaturas y reflejar selectivamente parte de éste (el infrarrojo) a altas temperaturas. En esta línea, el proyecto propone diversas acciones de I+D para el desarrollo de capas delgadas con composición VO_2, óxido termocrómico caracterizado con una temperatura de transición cercana a la temperatura ambiente, sobre vidrio y plásticos mediante

técnicas escalables industrialmente, así como su nanoestructuración, dopado e integración en sistemas multicapas a fin de mejorar sus características y prestaciones multifuncionales.

The International Energy Agency considers that the systematic use of autonomous procedures for environmental control is one of the best technological approaches to minimize the energy employed to cool down buildings and other urban structures (it represents more than 40% of the global energy use in developed countries, much above the use in transportation, for instance), thus reducing the environmental impact and improving human comfort. TOLERANCE aims at introducing and developing a technology based on thermochromic materials in Andalusia as a smart and autonomous element to control the penetration of solar radiation in buildings. This project focusses on various applications such as smart windows in buildings and urban furniture, improvement of sanitary water systems or environmental control in greenhouses. While at low temperatures, a thermochromic coating transmits most solar spectrum, it selectively filters out the infrared region of this spectrum at high temperatures. In this research, TOLERANCE proposes several R+D actions to grow thin films with composition VO_2, a thermochromic oxide with transition temperature near room temperature, on glass and plastic by means of industrial scalable techniques, as well as its nanostructuration, doping and integration in multilayer systems to improve its features and multifunctional properties.

OTROS PROYECTOS / OTHER PROJECTS

Desarrollo de electrolizadores más eficientes y competitivos basados en electrodos preparados mediante pulverización catódica

Periodo/Period:	2020 / 2021
Entidad Financiadora:	Ayuda a la Investigación “Fundación Domingo Martínez”
Importe total/Total amount:	50.000 €
Investigador responsable/Research head:	Antonio de Lucas Consuegra (Universidad Castilla La Mancha)
Componentes/Research group:	Agustín R. González-Elipe, Francisco Yubero Valencia

Sistema de generación de hidrógeno a partir de gasoil

Periodo/Period:	09-04-2020 / 1-11-2022
Entidad Financiadora:	Proyecto contratado con Inyecciones plásticas mecacontrol, S.L.
Importe total/Total amount:	45.000 €
Investigador responsable/Research head:	Ana María Gómez Ramírez
Componentes/Research group:	Agustín R. González-Elipe, José Cotrino Bautista, Manuel Oliva Ramírez

Recubrimientos nanoestructurados antiCOVID-19

Código/Code:	2020-URL-Proj 020
Periodo/Period:	01-01-2020 / 31-12-2020
Organismo Financiador/Financial source:	Universitat Ramón Llull (URL)
Importe total/Total amount:	13.000 €
Investigador responsable/Research head:	Manuel David Abad Roldán (URL)
Componentes/Research group:	Juan Carlos Sánchez López

Prototipo para la detección por imagen de contaminantes emergentes en aguas residuales mediante nanotecnología y deep learning (nanovision)

Código/Code:	6079
Periodo/Period:	31-10-2019 / 30-10-2020
Organismo Financiador/Financial source:	Junta de Andalucia
Importe total/Total amount:	70.117 €
Investigador responsable/Research head:	Ángel Barranco Quero

Red de Terapia Cellular (TERCEL)

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

Sensores optofluídicos y electroquímicos 1D preparados mediante técnicas de PVD a ángulo rasante

Código/Code:	201860E050
Periodo/Period:	01-06-2018 / 31-05-2021
Organismo Financiador/Financial source:	CSIC (Intramural)
Importe total/Total amount:	208.029 €
Investigador responsable/Research head:	Agustín R. González-Elipe

Ayudas extraordinarias para la preparación de proyectos 2019 (MAT2016-79866-R)

Código/Code:	2019AEP161
Periodo/Period:	01-01-2020 / 31-05-2020
Organismo Financiador/Financial source:	CSIC
Importe total/Total amount:	34.139 €
Investigador responsable/Research head:	Ángel Barranco Quero

AYUDAS PARA LA ADQUISICIÓN DE EQUIPOS

Goniómetro para medidas de ángulo de contacto de superficies en ambiente controlado (IE_19_142 USE)
Financia: Junta de Andalucía
Importe Concedido: 82.000,00 €
Periodo: 29-12-2020 / 28-12-2022

CONVENIOS Y CONTRATOS / CONTRACTS AND AGREEMENTS

Nueva matriz multisensora óptica reconfigurable para detección de contaminantes en el agua

Periodo/Period:	02-12-2019 / 02-12-2021
Organismo Financiador/Financial source:	EXPACE ON BOARD SYSTEMS, S.L.
Importe total/Total amount:	181.500 €
Investigador responsable/Research head:	Ángel Barranco Quero

Desarrollo de nuevos plasmas fríos para aceleración de germinación en condiciones de sequía

Periodo/Period:	02-12-2019 / 01-12-2021
Organismo Financiador/Financial source:	ARQUIMEA INGENIERIA, S.L.
Importe total/Total amount:	227.480 €
Investigador responsable/Research head:	Agustin R. González-Elipe

Nuevo sistema de propulsión espacial basado en el principio de Mach

Periodo/Period:	02-12-2019 / 30-12-2022
Organismo Financiador/Financial source:	ARQUIMEA INGENIERIA, S.L.
Importe total/Total amount:	100.000 €
Investigador responsable/Research head:	Agustin R. González-Elipe

Caracterización superficial de membranas de ósmosis inversa

Periodo/Period:	06-04-2018 / 01-04-2020
Organismo Financiador/Financial source:	ACCIONA AGUA, S.A.U.
Importe total/Total amount:	13.646,65 €
Investigador responsable/Research head:	Juan Pedro Espinós Manzorro

Caracterización superficial de membranas de ósmosis inversa

Periodo/Period:	01-05-2020 / 06-10-2022
Organismo Financiador/Financial source:	ACCIONA AGUA, S.A.U.

Importe total/Total amount: 5.971,35 €
Investigador responsable/Research head: Juan Pedro Espinós Manzorro

Caracterización microestructural y química de materiales para avisadores sonoros

Periodo/Period: 16-06-2019 / 15-01-2021
Organismo Financiador/Financial source: Clarton Horn
Importe total/Total amount: 7.260 €
Investigador responsable/Research head: Asunción Fernández Camacho

Optimización del proceso de oxidación de anillos de Kovar para la fabricación de uniones vidrio-metal

Periodo/Period: 19-01-2010 / 31-12-2020
Organismo Financiador/Financial source: Clarton Horn
Importe total/Total amount: 45.575 €
Investigador responsable/Research head: Asunción Fernández Camacho

PATENTES / PATENTS

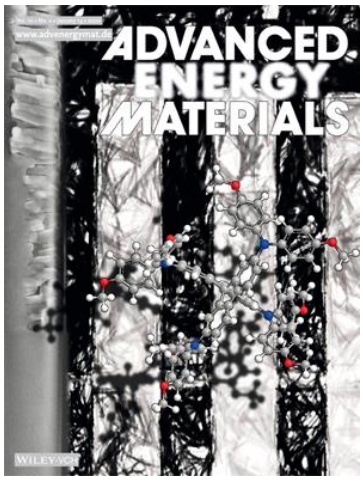
Sistema de Identificación In situ de productos petrolíferos en yacimientos

Inventores: Víctor J. Rico Gavira, Jorge Gil Rostra, Agustín R. González-Elipe, Francisco Yubero Valencia
Tipo de Patente: Nacional
Número de Solicitud: 202031026
Fecha Solicitud: 9 de septiembre de 2020
Entidad Titular: Consejo Superior de Investigaciones Científicas, Repsol, S.A., Indra Sistemas, S.A.

Vectores no virales a partir de nanopartículas mesoporosas para su aplicación en terapia génica para el tratamiento de patologías degenerativas de la retina

Inventores: Lourdes Valdes Sánchez (CABIMER), María Aránzazu Díaz Cuenca, Adoración Montero Sánchez (CABIMER), Sara Borrego González, Berta de la Cerda Haynes (CABIMER)
Tipo de Patente: Nacional
Número de Solicitud: 202031290
Fecha Solicitud: 23 de diciembre de 2020
Entidad Titular: Consejo Superior de Investigaciones Científicas

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

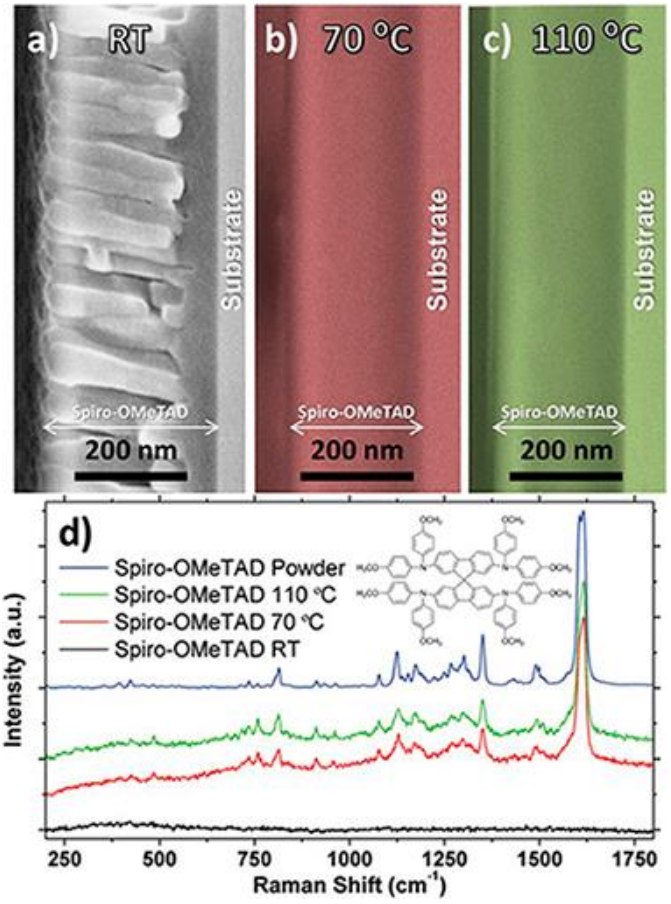


Enhanced Stability of Perovskite Solar Cells Incorporating Dopant-Free Crystalline Spiro-OMeTAD Layers by Vacuum Sublimation

Barranco, A; López-Santos, MC; Idigoras, J; Aparicio, FJ; Obrero-Pérez, J; López-Flores, V; Contreras-Bernal, L; Rico, V; Ferrer, J; Espinós, JP; Borrás, A; Anta, JA; Sánchez-Valencia, JR

Advanced Energy Materials, **58** (2020) 1901524
Enero, 2020 | **DOI:** 10.1002/aenm.201901524

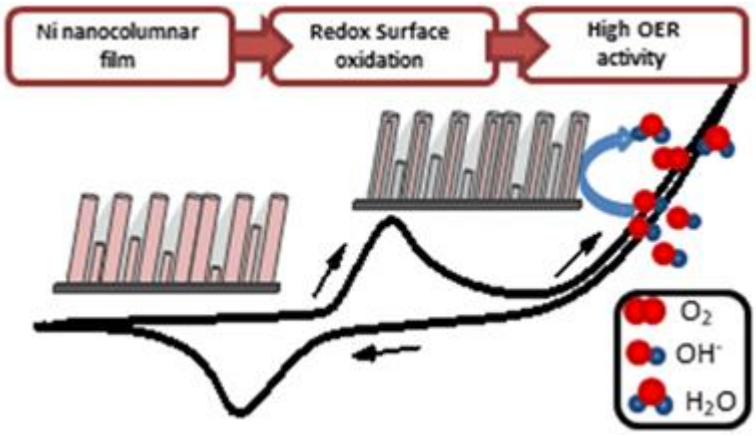
The main handicap still hindering the eventual exploitation of organometal halide perovskite-based solar cells is their poor stability under prolonged illumination, ambient conditions, and increased temperatures. This article shows for the first time the vacuum processing of the most widely used solid-state hole conductor (SSHC), i.e., the Spiro-OMeTAD [2,2 ',7,7 '-tetrakis (N,N-di-p-methoxyphenyl-amine) 9,9 '-spirobifluorene], and how its dopant-free crystalline formation unprecedently improves perovskite solar cell (PSC) stability under continuous illumination by about two orders of magnitude with respect to the solution-processed reference and after annealing in air up to 200 °C. It is demonstrated that the control over the temperature of the samples during the vacuum deposition enhances the crystallinity of the SSHC, obtaining a preferential orientation along the pi-pi stacking direction. These results may represent a milestone toward the full vacuum processing of hybrid organic halide PSCs as well as light-emitting diodes, with promising impacts on the development of durable devices. The microstructure, purity, and crystallinity of the vacuum sublimated Spiro-OMeTAD layers are fully elucidated by applying an unparalleled set of complementary characterization techniques, including scanning electron microscopy, X-ray diffraction, grazing-incidence small-angle X-ray scattering and grazing-incidence



wide-angle X-ray scattering, X-ray photoelectron spectroscopy, and Rutherford backscattering spectroscopy.

Chemistry and Electrocatalytic Activity of Nanostructured Nickel Electrodes for Water Electrolysis

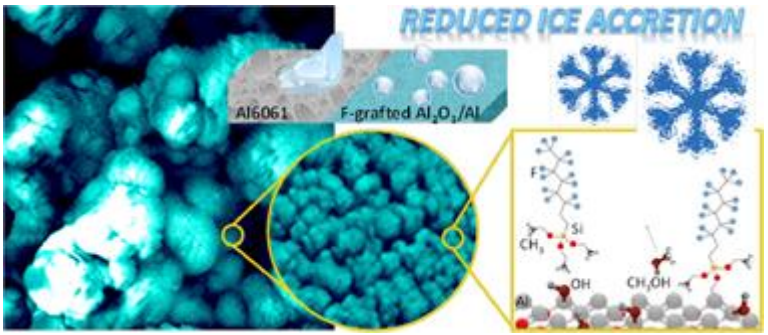
López-Fernández, E; Gil-Rostra, J; Espinós, JP; González-Elipe, AR; Consuegra, AD; Yubero, F
ACS Catalysis, **10** (2020) 6159-6170
Junio, 2020 | **DOI:** 10.1021/acscatal.0c00856



Herein we have developed nanostructured nickel-based electrode films for anion exchange membrane water electrolysis (AEMWE). The electrodes were prepared by magnetron sputtering (MS) in an oblique angle configuration and under various conditions aimed at preparing metallic, oxide, or oxyhydroxide films. Their electrochemical analysis has been complemented with a thorough physicochemical characterization to determine the effect of microstructure, chemical state, bilayer structure, and film thickness on the oxygen evolution reaction (OER). The maximum electrocatalytic activity was found for the metallic electrode, where analysis by X-ray photoelectron spectroscopy (XPS) and X-ray absorption spectroscopy (XAS) demonstrated that the active catalytic phase at the surface after its electrochemical conditioning is a kind of oxidized nickel oxide/hydroxide layer with the thickness of a few nanometers. Electrochemical impedance spectroscopy analysis of these steady-state working electrodes supports that the enhanced performance of the metallic nickel anode vs other chemical states resides in the easier electron transfer through the electrode films and the various interlayers built up during their fabrication and activation. The long-term steady-state operation of the anodes and their efficiency for water splitting was proved in a full-cell AEMWE setup incorporating magnetron-sputtered metallic nickel as the cathode. This work proves that MS is a suitable technique to prepare active, stable, and low-cost electrodes for AEMWE and the capacity of this technique to control the chemical state of the electrocatalytically active layers involved in the OER.

Robust anti-icing superhydrophobic aluminum alloy surfaces by grafting fluorocarbon molecular chains

Rico, V; Mora, J; García, P; Aguero, A; Borrás, A; González-Elipe, AR; López-Santos, C
Applied Materials Today, **21** (2020) 100815
Diciembre, 2020 | **DOI:** 10.1016/j.apmt.2020.100815



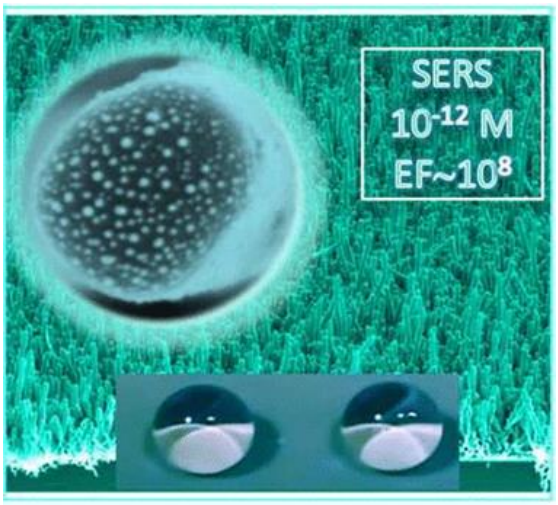
Infusion of low surface tension liquids in nanostructured surfaces is currently used to promote an anti icing response, although the long term stability of these systems is often jeopardized by losses of the infused liquid. In this work, we propose an alternative to the infusion procedure to induce a more effective and long lasting anti-icing capacity. The method consists of a combination of surface nanostructuration with the chemical grafting of fluorocarbon molecules. Al6061 substrates have been subjected to laser roughening and further modified with a nanostructured Al_2O_3 thin film to achieve a dual roughness and porous surface state. These surfaces have been subjected to a grafting treatment with perfluorooctyltriethoxysilane (PFOTES) vapor or, for comparative purposes, infused with a low surface tension liquid. A comparative analysis of the wetting, water condensation and anti-icing properties of these two systems showed an outstandingly better performance for the grafted surfaces with respect to the infused ones. Grafted surfaces were markedly superhydrophobic and required higher water vapor pressures to induce condensation. When looking for their anti-icing capacity, they presented quite long freezing delay times for supercooled water droplets (i.e. almost four hours) and exhibited a notably low ice accretion in a wind tunnel test. The high aging resistance and durability of these grafted surfaces and the reproducibility of the results obtained when subjected to successive ice accretion cycles show that molecular grafting is an efficient anti-icing methodology that, in aggressive media, may outperform the classical infusion procedures. The role of the fluorocarbon chains anchored on the surface in inducing an anti-icing functionality is discussed.

Plasma-Enabled Amorphous TiO_2 Nanotubes as Hydrophobic Support for Molecular Sensing by SERS

Filippin, N; Castillo-Seoane, J; López-Santos, MC; Rojas, CT; Ostrikov, K; Barranco, A; Sánchez-Valencia, JR; Borrás, A

ACS Applied Materials & Interfaces, **12** (2020) 50721-50733
Noviembre, 2020 | **DOI:** 10.1021/acsami.0c14087

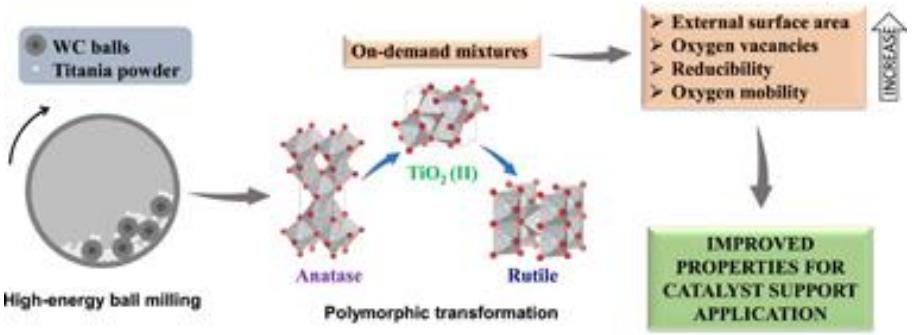
We devise a unique heteronanostructure array to overcome a persistent issue of simultaneously utilizing the surface-enhanced Raman scattering, inexpensive, Earth-abundant materials, large surface areas, and multifunctionality to demonstrate near single-molecule detection. Room-temperature plasma-enhanced chemical vapor deposition and thermal evaporation provide high-density arrays of vertical TiO_2 nanotubes decorated with Ag nanoparticles. The role of the TiO_2 nanotubes is 3-fold: (i) providing a high surface area for the homogeneous distribution of supported Ag nanoparticles, (ii) increasing the water contact angle to achieve superhydrophobic



limits, and (iii) enhancing the Raman signal by synergizing the localized electromagnetic field enhancement (Ag plasmons) and charge transfer chemical enhancement mechanisms (amorphous TiO_2) and by increasing the light scattering because of the formation of vertically aligned nanoarchitectures. As a result, we reach a Raman enhancement factor of up to 9.4 × 107, satisfying the key practical device requirements. The enhancement mechanism is optimized through the interplay of the optimum microstructure, nanotube/shell thickness, Ag nanoparticles size distribution, and density. Vertically aligned amorphous TiO_2 nanotubes decorated with Ag nanoparticles with a mean diameter of 10–12 nm provide enough sensitivity for near-instant concentration analysis with an ultralow few-molecule detection limit of 10–12 M (Rh6G in water) and the possibility to scale up device fabrication.

Tailoring materials by high-energy ball milling: TiO_2 mixtures for catalyst support application

Rinaudo, MG; Beltran, AM; Fernández, MA; Cadus, LE; Morales, MR
Materials Today Chemistry, **17** (2020) 100340
Septiembre, 2020 | **DOI:** 10.1016/j.mtchem.2020.100340



We carried out a rational design of catalyst supports by high-energy ball milling. Tailored mixtures of TiO_2 crystalline phases were obtained using rotational speed and milling time as variable parameters. Polymorphic transformation from anatase to rutile through high-pressure TiO_2 (II) as intermediate was confirmed by X-ray Diffraction (XRD), Raman Spectroscopy and Transmission Electron Microscopy (TEM). Also, starting material doubled its specific surface area due to particle fragmentation, as confirmed by surface area of Brunauer-Emmet-Teller (S-BET) and Scanning Electron Microscopy (SEM). Defects introduced during milling process generated oxygen vacancies in the surface and bulk of supports, as evidenced by X-ray Photoelectron Spectroscopy (XPS) and Electron Paramagnetic Resonance (EPR). Furthermore, longer milling time increased reducibility and oxygen mobility of supports, as observed by H_2 Temperature Programmed Reduction (H_2-TPR) and O_2 Temperature Programmed Desorption (O_2-TPD). Phase composition remained unchanged even under extreme conditions, highlighting the stability of unusual TiO_2 (II) phase. Properties achieved in

present materials could benefit metal-support interactions and play a major role in supported catalysts.

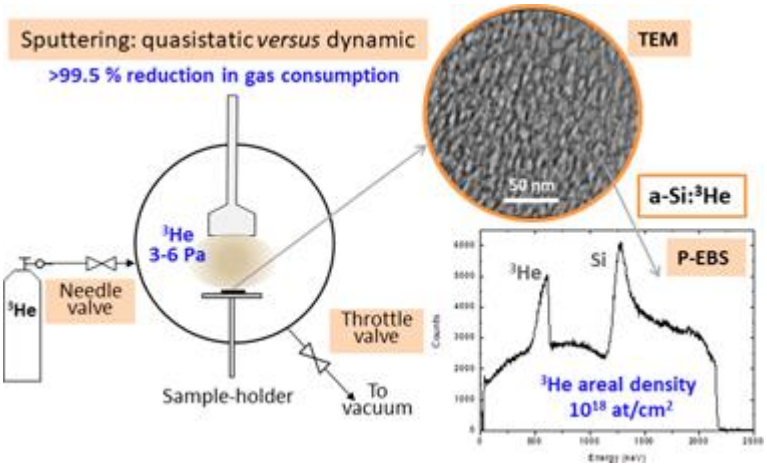
Unraveling Discharge and Surface Mechanisms in Plasma-Assisted Ammonia Reactions

Navascues, P; Obrero-Pérez, JM; Cotrino, J; González-Elipe, AR; Gómez-Ramírez, A
ACS Sustainable Chemistry & Engineering, **8** (2020) 14855-14866
Octubre, 2020 | **DOI:** 10.1021/acssuschemeng.0c04461

Current studies on ammonia synthesis by means of atmospheric pressure plasmas respond to the urgent need of developing less environmentally aggressive processes than the conventional Haber-Bosch catalytic reaction. Herein, we systematically study the plasma synthesis of ammonia and the much less investigated reverse reaction (decomposition of ammonia into nitrogen and hydrogen). Besides analyzing the efficiency of both processes in a packed-bed plasma reactor, we apply an isotope-exchange approach (using D_2 instead of H_2) to study the reaction mechanisms. Isotope labeling has been rarely applied to investigate atmospheric plasma reactions, and we demonstrate that this methodology may provide unique information about intermediate reactions that, consuming energy and diminishing the process efficiency, do not effectively contribute to the overall synthesis/decomposition of ammonia. In addition, the same methodology has demonstrated the active participation of the interelectrode material surface in the plasma-activated synthesis/decomposition of ammonia. These results about the involvement of surface reactions in packed-bed plasma processes, complemented with data obtained by optical emission spectroscopy analysis of the plasma phase, have evidenced the occurrence of inefficient intermediate reaction mechanisms that limit the efficiency and shown that the rate-limiting step for the ammonia synthesis and decomposition reactions are the formation of NH* species in the plasma phase and the electron impact dissociation of the molecule, respectively.

Low gas consumption fabrication of He-3 solid targets for nuclear reactions

Fernández, A; Hufschmidt, D; Colaux, JL; Valiente-Dobon, JJ; Godinho, V; de Haro, MCJ; Feria, D; Gadea, A; Lucas, S
Materials & Design, **186** (2020) 108337
Enero, 2020 | **DOI:** 10.1016/j.matdes.2019.108337

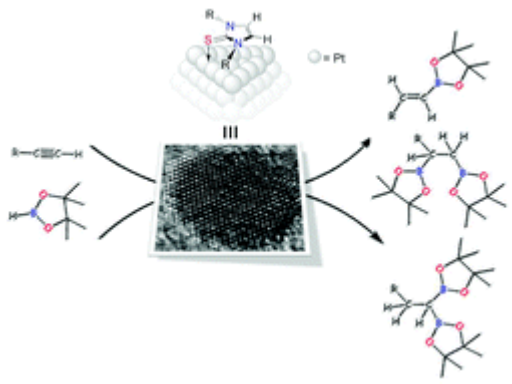


Nanoporous solids that stabilize trapped gas nanobubbles open new possibilities to fabricate solid targets for nuclear reactions. A methodology is described based on the magnetron sputtering (MS) technique operated under quasistatic flux conditions to produce such nanocomposites films with He-3 contents of up to 16 at.% in an amorphous-silicon matrix. In addition to the characteristic low pressure (3-6 Pa) needed for the gas discharge, the method ensures almost complete reduction of the process gas flow during film fabrication. The method could produce similar materials to those obtained under classical dynamic flux conditions for MS. The drastic reduction (>99.5%) of the gas consumption is fundamental for the fabrication of targets with scarce and expensive gases. Si:He-3 and W:He-3 targets are presented together with their microstructural (scanning and transmission electron microscopy, SEM and TEM respectively) and compositional (Ion Beam Analysis, IBA) characterization. The He-3 content achieved was over 1 x 10^{18} at/cm^2 for film thicknesses between 1.5 and 3 µm for both Si and W matrices. First experiments to probe the stability of the targets for nuclear reaction studies in inverse kinematics configurations are presented.

Platinum nanoparticles stabilized by N-heterocyclic thiones. Synthesis and catalytic activity in mono- and di-hydroboration of alkynes

Moraes, LCC; Figueiredo, RCC; Espinós, JPP; Vattier, F; Franconetti, A; Jaime, C; Lacroix, B; Rojo, J; Lara, P; Conejero, S

Nanoscale, **12** (2020) 6821-6831
Marzo, 2020 | **DOI:** 10.1039/d0nr00251h



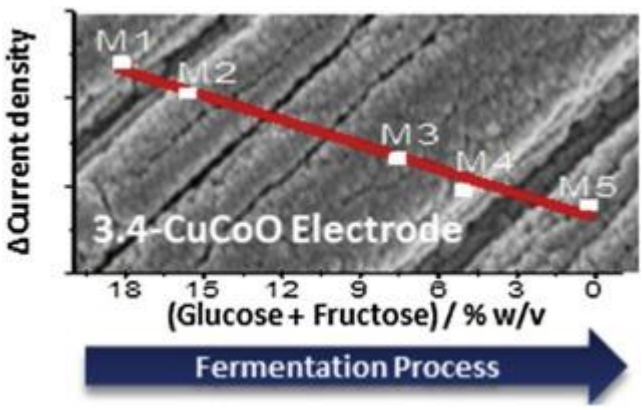
N-Heterocyclic Thiones (NHT) proved to be efficient ligands for the stabilization of small platinum nanoparticles (1.3-1.7 nm), synthesized by decomposition of [Pt(dba)$_2$], under a H_2 atmosphere, in the presence of variable sub-stoichiometric amounts of the NHT. Full characterization by means of TEM, HR-TEM, NMR, ICP, TGA and XPS have been carried out, providing information about the nature of the metal nanoparticles and the interaction of the NHT ligands to the metal surface. Importantly, DFT calculations indicate that some NHT ligands interact with the metal through the C=C double bond of the imidazole fragment in addition to the sulfur atom, thus providing additional stabilization to the nanoparticles. According to XPS, TGA and ICP techniques, the surface

coverage by the ligand increases by decreasing the size of the substituents on the nitrogen atom. The platinum nanoparticles have been used as catalyst in the hydroboration of alkynes. The most active system is that with a less covered surface area lacking an interaction of the ligand by means of the C═C double bond. This catalyst hydroborates alkynes with excellent selectivities towards the monoborylated anti-Markovnikov product (vinyl-boronate) when one equiv. of borane is used. Very interestingly, aliphatic alkynes undergo a second hydroborylation process leading to the corresponding 1,1- and 1,2-diboroylated species with good selectivities towards the former.

Robust label-free CuxCoyOz electrochemical sensors for hexose detection during fermentation process monitoring

López-Fernández, E; Gil-Rostra, J; Espinós, JP; González, R; Yubero, F; de Lucas-Consuegra, A; González-Elipe, AR

Sensors and Actuators B-Chemical, **304** (2020) 127360
Febrero, 2020 | **DOI:** 10.1016/j.snb.2019.127360

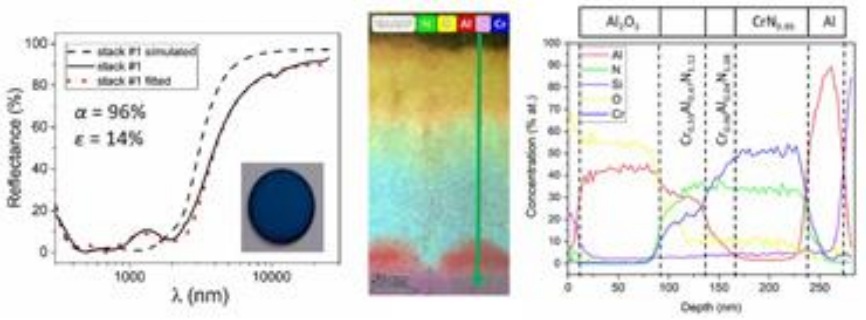


Label free electrochemical sensors of glucose are used whenever long-term operation and stable response are required. For this purpose, various metals and oxides of the first transition series have been proposed as alternative to more expensive noble metal electrochemical sensors. In this work we propose a new formulation consisting of copper-cobalt mixed oxides which, in the form of porous and nanostructured thin films with well controlled Co/Cu ratio, are prepared at room temperature in one step by a modification of the magnetron sputtering oblique angle deposition procedure. Films with various compositions were electrochemically characterized by cyclic voltammetry to determine their amperometric response to glucose as a function of voltage and NaOH electrolyte concentration. This analysis showed that films with a Co/Cu atomic ratio equal 3.4 presented a maximum sensitivity (0.710 A M^{-1} cm^{-2}), a small limit of detection (0.105 μM) and a resilient behaviour upon cycling operation and long storage periods that clearly overpassed the performance of copper and cobalt single oxides. The Cu_xCo_yO electrocatalysts also presented a good selectivity towards glucose and fructose in the presence of common interference compounds found in biological fluids (e.g., ascorbic acid, acetaminophen and uric acid), sucrose and ethanol, this latter present in many agrofood liquids. The possibilities of this sensor electrocatalyst have been tested for the analysis of a wine synthetic fermentation process. The comparison of the electrochemical results with conventional analytical methods showed a lineal amperometric response with respect hexose contents in a must at different stages of its transformation into wine.

High-temperature solar-selective coatings based on Cr(Al)N. Part 2: Design, spectral properties and thermal stability of multilayer stacks

Rojas, TC; Caro, A; Escobar-Galindo, R; Sánchez-López, JC
Solar Energy Materials and Solar Cells, **218** (2020) 110812
Diciembre, 2020 | **DOI:** 10.1016/j.solmat.2020.110812

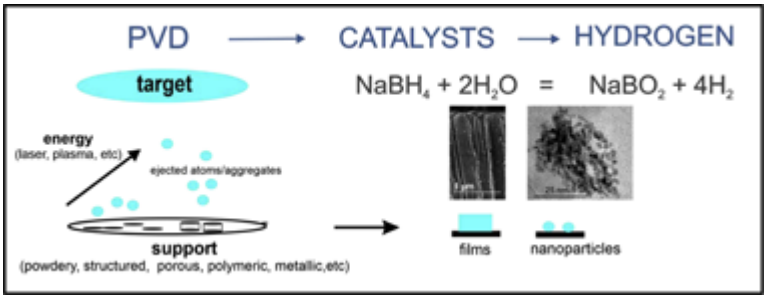
Two multilayer solar selective absorber coatings [Al/$CrN_{0.95}$/$Cr_{0.96}Al_{0.04}N_{1.08}$/$Cr_{0.53}Al_{0.47}N_{1.12}$/$Al_2O_3$ (stack #1) and $Cr_{0.96}Al_{0.04}N_{0.89}$/$Cr_{0.62}Al_{0.38}N_{1.00}$/$Cr_{0.53}Al_{0.47}N_{1.12}$/$Al_2O_3$ (stack #2)] were deposited on 316L steel by combining direct current (DC) and high power impulse magnetron sputtering (HiPIMS) technologies with the aim of increasing the working limit temperature. The composition and thickness of the constituent layers were optimized using CODE software to achieve a high solar absorptance (α) and low values of thermal emittance (ε) in the infrared region. The deposited multilayered stacks were heated during 2 h in air at 600, 700 and 800 °C to study their thermal stability and optical performance. Compositional, structural and optical characterization of the stacks (as-prepared and after thermal treatment) was performed. Both stacks presented a good solar selectivity with α > 95% and ε < 15%, were stable up to 600 °C and fulfilled the performance criterion PC < 5% after 600 and 700 °C treatments. Despite the stacks suffered chemical transformations above 600 °C, partial oxidation (stack #1) and Cr_2N formation (stack #1 and #2), the optical properties were optimum up to 700 °C for stack #1 (α = 94%, $\varepsilon_{(25\ ºC)}$ = 12%) and 600 °C for stack #2 (α = 93%, , $\varepsilon_{(25\ ºC)}$ = 13%). The solar-to-mechanical energy conversion efficiencies (h) of the as-deposited and annealed (600 and 700 °C) samples were up to 20% points higher than the absorber paint commercially used (Pyromark). At 800 °C, they underwent a further structural transformation, provoked by the oxidation of the inner layers, and they consequently lost their solar selectivity.



Advances in the implementation of PVD-based techniques for the preparation of metal catalysts for the hydrolysis of sodium borohydride

Arzac, GM; Fernández, A
International Journal of Hydrogen Energy, **58** (2020) 33288-33309
Noviembre, 2020 | **DOI:** 10.1016/j.ijhydene.2020.09.041

Sodium borohydride constitutes a safe alternative for the storage of hydrogen with a high gravimetric content. Catalytic hydrolysis of sodium borohydride permits on-demand hydrogen generation for multiple applications. In this field, the rational design of efficient metal catalysts deposited on structured supports is highly desirable. For most reactions, chemical methods are the most commonly used methods for the preparation of supported metal catalysts. Physical vapour deposition techniques are emerging as an alternative for the preparation of catalytic materials because of their multiple advantages. They permit the one-step deposition of catalysts



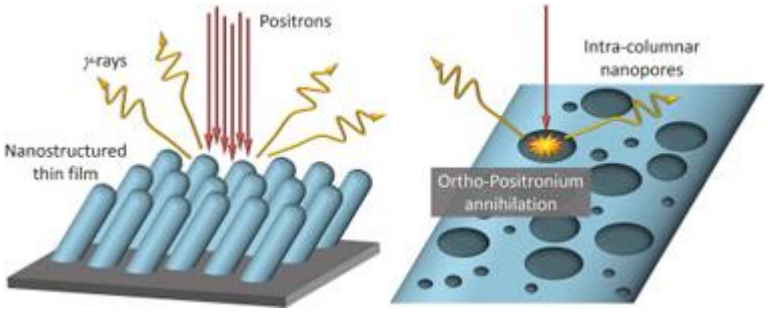
on structured supports with controlled microstructure and composition, avoiding the multi-step procedures and the generation of hazardous by-products associated with chemical routes.

In this short review, we will describe the available literature on the application of physical vapour deposition techniques for the preparation of supported metal catalysts for the hydrolysis of sodium borohydride. The effects of the deposition parameters on the properties of the catalytic materials will be discussed, and strategies for further improvement will be proposed. Here, we also present our new results on the study of nanoporous Pt catalysts that are prepared through the chemical dealloying of magnetron sputtered Pt-Cu thin films for the hydrolysis of sodium borohydride. We discuss the capabilities of the technique to tune the microstructure from columnar to closed porous microstructures, which, coupled with dealloying, produces more active supported catalysts with lower noble metal loading. At the end, we briefly mention the application of PVD for the preparation of supported catalysts for the hydrolysis of ammonia borane, another hydrogen generating reaction of high interest nowadays.

Positron annihilation analysis of nanopores and growth mechanism of oblique angle evaporated TiO_2 and SiO_2 thin films and multilayers

García-Valenzuela, A; Butterling, M; Liedke, MO; Hirschmann, E; Trinh, TT; Attallah, AG; Wagner, A; Álvarez, R; Gil-Rostra, J; Rico, V; Palmero, A; González-Elipe, AR

Microporous and Mesoporous Materials, **295** (2020) 109968
Marzo, 2020 | **DOI:** 10.1016/j.micromeso.2019.109968

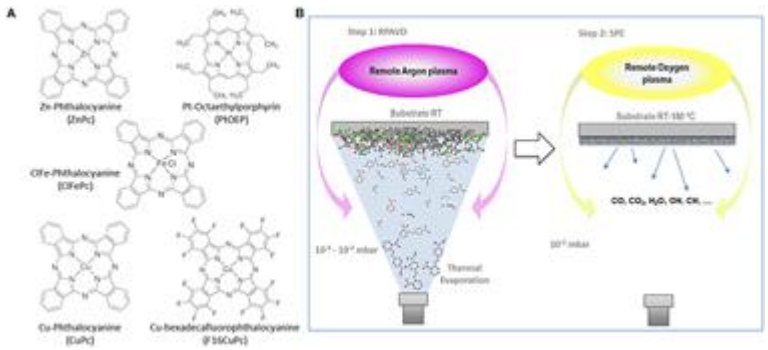


The nano-porosity embedded into the tilted and separated nanocolumns characteristic of the microstructure of evaporated thin films at oblique angles has been critically assessed by various variants of the positron annihilation spectroscopy. This technique represents a powerful tool for the analysis of porosity, defects and internal interfaces of materials, and has been applied to different as-deposited SiO_2 and TiO_2 thin films as well as SiO_2/TiO_2 multilayers prepared by electron beam evaporation at 70 and 85 zenithal angles. It is shown that, under same deposition conditions, the concentration of internal nano-pores in SiO_2 is higher than in TiO_2 nanocolumns, while the situation is closer to this latter in TiO_2/SiO_2 multilayers. These features have been compared with the predictions of a Monte Carlo simulation of the film growth and explained by

considering the influence of the chemical composition on the growth mechanism and, ultimately, on the structure of the films.

Supported Porous Nanostructures Developed by Plasma Processing of Metal Phthalocyanines and Porphyrins

Obrero, JM; Filippin, AN; Alcaire, M; Sánchez-Valencia, JR; Jacob, M; Matei, C; Aparicio, FJ; Macias-Montero, M; Rojas, TC; Espinós, JP; Saghi, Z; Barranco, A; Borrás, A
Frontiers in Chemistry, **8** (2020) 520
Junio, 2020 | **DOI:** 10.3389/fchem.2020.00520



The large area scalable fabrication of supported porous metal and metal oxide nanomaterials is acknowledged as one of the greatest challenges for their eventual implementation in on-device applications. In this work, we will present a comprehensive revision and the latest results regarding the pioneering use of commercially available metal phthalocyanines and porphyrins as solid precursors for the plasma-assisted deposition of porous metal and metal oxide films and three-dimensional nanostructures (hierarchical nanowires and nanotubes). The most advanced features of this method relay on its ample general character from the point of view of the porous material composition and microstructure, mild deposition and processing temperature and energy constrictions and, finally, its straightforward compatibility with the direct deposition of the porous nanomaterials on processable substrates and device-architectures. Thus, taking advantage of the variety in the composition of commercially available metal porphyrins and phthalocyanines, we present the development of metal and metal oxides layers including Pt, CuO, Fe_2O_3, TiO_2, and ZnO with morphologies ranging from nanoparticles to nanocolumnar films. In addition, we combine this method with the fabrication by low-pressure vapor transport of single-crystalline organic nanowires for the formation of hierarchical hybrid organic@metal/metal-oxide and @metal/metal-oxide nanotubes. We carry out a thorough characterization of the films and nanowires using SEM, TEM, FIB 3D, and electron tomography. The latest two techniques are revealed as critical for the elucidation of the inner porosity of the layers.

Thermo-optic response of MEH-PPV films incorporated to monolithic Fabry-Perot microresonators

Rostra, JG; Soler-Carracedo, K; Martín, LL; Lahoz, F; Yubero, F
Dyes and Pigments, **182** (2020) 108625
Noviembre, 2020 | **DOI:** 10.1016/j.dyepig.2020.108625

Poly[2-methoxy-5-(2'-ethylhexyloxy)-1,4-phenylene vinylene] (MEH-PPV) is a semiconducting optically active polymer widely used in optoelectronics research. MEH-PPV can be commercially acquired in a large range of molecular weights. However, the influence of this property on the optical performance of the polymer is often disregarded. In this paper, the thermal dependence of the refractive index of MEH-PPV thin films prepared from high and medium molecular weight polymers is investigated. Thus, monolithic Fabry-Perot (FP) microcavities are fabricated, in which the active polymer film is part of their defect layer. It is found that when these devices are used as optical temperature sensors, the position of the emission band of the microcavities excited with a blue diode laser shifts to lower wavelengths when temperature increases with sensitivities in the 0.2-0.3 nm/^{o}C range. This effect is ascribed to the variation in the refractive index of the polymer active layer within the resonator with temperature. According to theoretical simulations of optical transmittance by classical transfer matrix method and the evaluation of the optical eigenmodes by finite element methods of the manufactured FP resonator cavities, it is found that the MEH-PPV films present negative thermo-optic coefficients of about-0.018 K^{-1} and-0.0022 K^{-1} for high and medium molecular weight polymers, respectively, in the temperature range between 20 and 60 ^{o}C. These values are about the highest reported so far, to the best of our knowledge, and points to high performance thermal sensor applications.

Study of the influence of the precursors on the sensing properties of ZnO:Cu system

Ramos, A; Urbieta, A; Escalante, G; Hidalgo, P; Espinós, JP; Fernández, P
Ceramics International, **46** (2020) 8358-8367
Abril, 2020 | **DOI:** 10.1016/j.ceramint.2019.12.068

The properties of ZnO based materials for ethanol detection have been studied. Cu doped samples obtained from different precursors have been investigated. ZnO and ZnS have been used as host and Cu and CuO as dopant sources.
The sensing measurements have been mostly performed at room temperature. To monitor the effect of the presence of gas, resistivity and photoluminescence experiments with and without sensing gas have been carried out. The sensing behaviour is affected by the nature of the precursors used. For ZnO:Cu and ZnO:CuO series, a higher sensitivity is obtained at the lower gas concentrations, the better response is obtained for the sample ZnO:Cu with wt.1% of metallic copper. Strong segregation effects observed in these samples could be deleterious for the sensing properties. In the series ZnS:CuO, no segregation is observed, however the sensing behaviour is erratic and attributed to the reduction of Cu ions to the metallic state.

Tailoring CrN_x stoichiometry and functionality by means of reactive HiPIMS

Sánchez-López, JC; Caro, A; Alcala, G; Rojas, TC
Surface & Coatings Technology, **401** (2020) 126235
Noviembre, 2020 | **DOI:** 10.1016/j.surfcoat.2020.126235

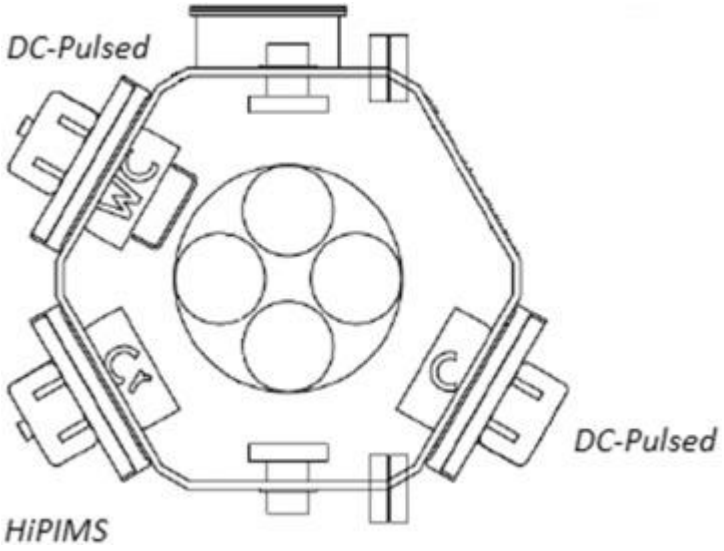
This work presents a complete study of the influence of HiPIMS pulse characteristics on the microstructure, chemical composition, mechanical and oxidation resistance properties of CrN thin films. The investigated parameters were frequency and pulse length at two different

nitrogen fluxes, maintaining constant the duty cycle conditions (2%). The effect of a negative bias of 100 V was investigated in a particular case. By changing the synthesis conditions, it was possible to tailor the N/Cr ratio and thus to control the CrN_x stoichiometry from x = 0.63 to 1.10. The selection of longer pulses (shorter frequencies) generates more disordered structures with lower N/Cr ratios. This is reflected in higher hardness and elastic modulus values on despite of a lower oxidation resistance due to existence of larger concentration of N vacancies. The best oxidation resistance is obtained at the highest peak current combined with additional ion bombardment provided by substrate biasing. The present results open the possibilities of modifying chemical composition and engineering surfaces by changing exclusively the pulse conditions in HiPIMS deposition processes.

Tribomechanical properties of hard Cr-doped DLC coatings deposited by low-frequency HiPIMS

Santiago, JA; Fernández-Martínez, I; Sánchez-López, JC; Rojas, TC; Wennberg, A; Bellido-González, V; Molina-Aldareguia, JM; Monclus, MA; González-Arrabal, R

Surface & Coatings Technology, **382** (2020) 124899
Enero, 2020 | **DOI:** 10.1016/j.surfcoat.2019.124899



Cr-doped diamond-like carbon (Cr-DLC) films with Cr contents ranging from 3 up to 20 at. % were synthesised in a codeposition process with HiPIMS (Cr deposition) and DC-pulsed technology (C deposition). The application of HiPIMS at low frequencies was observed to significantly enhance the energy density during the Cr plasma discharge due to the interaction of Cr-C species. The higher energy bombardment at low HiPIMS frequencies allowed doping with Cr the DLC structure avoiding the graphitization of the carbon structure. EELS spectroscopy was used to evaluate sp^3 content and Raman was used for sp^2 structural characterization of the films. Enhanced mechanical properties (hardness up to 30 GPa) were observed with nanoindentation for Cr-doped DLC at low frequencies. High temperature nanoindentation tests were also performed from room temperature to 425 ^oC in order to evaluate the evolution of hardness and Young Modulus with temperature. The results showed that the mechanical properties at high temperature mainly depend on the initial sp^3- sp^2 structure. Tribological tests were carried out

in air from room temperature to 250 °C. Cr-doped DLC coatings deposited by low-frequency HiPIMS showed lower friction and wear compared to undoped DLC.

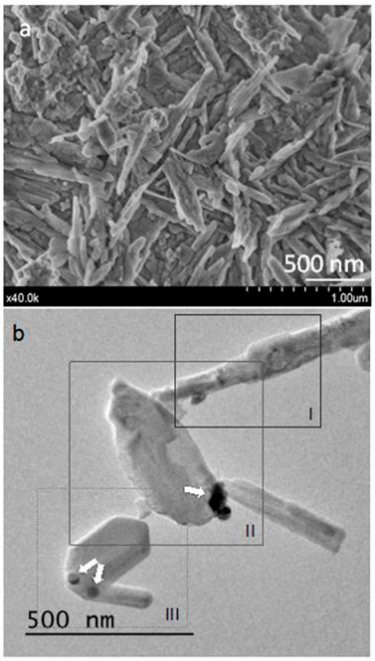
The wrinkling concept applied to plasma-deposited polymer-like thin films: A promising method for the fabrication of flexible electrodes

Thiry, Damien; Vinx, Nathan; Damman, Pascal; Aparicio, Francisco J.; Tessier, Pierre-Yves; Moerman, David; Leclere, Philippe; Godfroid, Thomas; Deprez, Sylvain; Snyders, Rony
Plasma Processes and Polymers, **17** (2020) e2000119
Septiembre, 2020 | **DOI:** 10.1002/ppap.202000119

In this communication, we report on an innovative solvent-free method that allows for the design of nano-/micropatterns with tuneable dimensions. Our approach is based on the spontaneous wrinkling phenomenon taking place in a bilayer system formed by a mechanically responsive bottom plasma polymer layer and a top aluminum thin film. The dimensions of the wrinkles can be adjusted in a wide range (i.e., from nanometer to micrometer range) by modulating the cross-linking density as well as the thickness of the plasma polymer layer. Finally, it is demonstrated that these wrinkled surfaces could efficiently be used as flexible electrodes. The whole set of our data unambiguously reveals the attractiveness of our method for the fabrication of the micro-/nanopattern with dimensions on demand.

A Microstructure Insight of MTA Repair HP of Rapid Setting Capacity and Bioactive Response

Jiménez-Sánchez, MC; Segura-Egea, JJ; Díaz-Cuenca, A
Materials, **13** (2020) 1641
Abril, 2020 | **DOI:** 10.3390/ma13071641



Mineral trioxide aggregate (MTA) is considered a bioactive endodontic material, which promotes natural mineralization at the material-tooth tissue interface. MTA Repair HP stands out because of the short setting time and the quick and effective bioactive response in vitro. The bioactivity, depens on material composition and microstructure. This work is devoted to analyze MTA Repair HP microstructural features, of both the powder precursor and set material, to get insights into the material physicochemical parameters-functionality performance relationships. Transmission electron microscopy (TEM), and field emission gun scanning electron microscopy (FEG-SEM) coupled with energy-dispersive X-ray (EDX) analyses were performed. X-ray diffraction (XRD) measurements were carried out at different times to investigate setting process. Bioactivity evaluation in vitro was carried out by soaking the processed cement disk in simulated body fluid (SBF). The presented results point out those MTA Repair HP precursor material characteristics of tricalcium silicate particles of nanometric size and high aspect ratio,

which provide an elevated surface area and maximized components dispersion of calcium silicate and very reactive calcium aluminate. The MTA Repair HP precursor powder nanostructure and formulation, allows a hydration process comprising silicate hydrate structures, which are very effective to achieve both fast setting and efficient bioactive response.

Thin film electroluminescent device based on magnetron sputtered Tb doped $ZnGa_2O_4$ layers

Gil-Rostra, J; Valencia, FY; González-Elipe, AR
Journal of Luminescence, **228** (2020) 117617
Diciembre, 2020 | **DOI:** 10.1016/j.jlumin.2020.117617

Photoluminescent (PL) layers and electroluminescent (EL) systems prepared by different methods have been systematically studied for the fabrication of flat panel displays, monitoring screens, and lighting systems. In this work we report about a new procedure of preparing Tb doped $ZnGa_2O_4$ green luminescent thin films at low temperature that consists of the simultaneous reactive magnetron sputtering (R-MS) deposition of a Zn-Ga mixed oxide acting as a matrix and the plasma decomposition (PD) of evaporated terbium acetylacetonate. The resulting films were transparent and presented a high PL efficiency making them good candidates for EL applications. Layers of this phosphor film with thickness in the order of hundreds nanometers were sandwiched between two dielectric layers of Y_2O_3 and $AlSiN_xO_y$ that were also prepared by R-MS. The response of the resulting EL device was characterized as a function of the applied voltage and the type of AC excitation signal. The high luminance and long-term stability of these thin film electroluminescent devices (TFELDs) proves the reliability and efficiency of this kind of transparent R-MS multilayer system (with a total thickness in order of 650 nm) for display and lighting applications.

Tribological performance of Nb-C thin films prepared by DC and HiPIMS

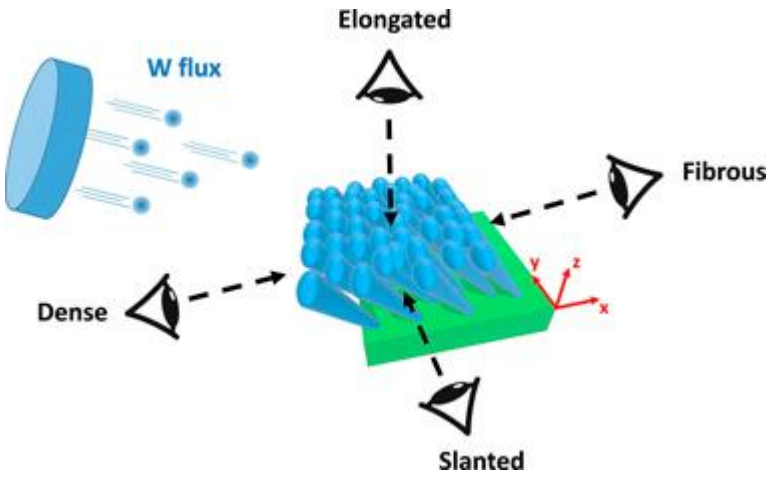
Sala, N; Abad, MD; Sánchez-López, JC; Cruz, M; Caro, J; Colominas, C
Materials Letters, **277** (2020) 12834
Octubre, 2020 | **DOI:** 10.1016/j.matlet.2020.128334

Nanostructured NbC thin films with variable contents of Nb and C were prepared by direct current (DC) magnetron sputtering, and for the first time, via high power impulse magnetron sputtering (HiPIMS) searching for an improvement in the tribological properties. X-ray diffraction shows that increasing the carbon incorporation, the crystalline composition evolves from Nb_2C to NbC phase. Further carbon enrichment leads to a nanocomposite structure formed by small NbC crystals (8-14 nm) dispersed in a-C matrix. The friction coefficient varied from high friction (0.8) to low friction (0.25) and the hardness values between 20 and 11 GPa depending on the film composition. A densification of the coatings by changing the methodology from DC to HiPIMS was not observed.

A 4-view imaging to reveal microstructural differences in obliquely sputter-deposited tungsten films

El Beainou, R; García-Valenzuela, A; Raschetti, M; Cote, JM; Álvarez, R; Palmero, A; Potin, V; Martín, N

Materials Letters, **264** (2020) 127381
Abril, 2020 | **DOI:** 10.1016/j.matlet.2020.127381



We report on the morphological disparity of the columnar growth in W thin films sputter-deposited by oblique angle deposition. Oriented tungsten thin films (400 +/- 50 nm thick) are prepared using a tilt angle α of 80 °C and a sputtering pressure of 0.25 Pa. Inclined columns (beta = 38 +/- 2 degrees) are produced and the microstructure is observed by scanning electron microscopy. A 4-view imaging is performed in order to show inhomogeneous growing evolutions in the columns. Morphological features vs. viewing direction are also investigated from a growth simulation of these tilted W columns. Experimental and theoretical approaches are successfully compared and allow understanding how the direction of the W particle flux leads to dense or fibrous morphologies, as the column apexes are in front of the flux or in the shadowing zone.

Wetting and spreading of liquid lithium onto nanocolumnar tungsten coatings tailored through the topography of stainless steel substrates

Munoz-Pina, S; García-Valenzuela, A; Oyarzabal, E; Gil-Rostra, J; Rico, V; Alcala, G; Álvarez, R; Tabares, FL; Palmero, A; González-Elipe, AR

Nuclear Fusion, **60** (2020) 126033
Diciembre, 2020 | **DOI:** 10.1088/1741-4326/abb53e

The use of liquid metal as an alternative to cover the plasma-exposed areas of fusion reactors has called for the development of substrates where refilling and metal spreading occur readily and at reasonably low temperatures. In the search for common materials for this purpose, we show that nanostructured tungsten coatings deposited on stainless steel (SS) by magnetron sputtering at oblique angles (MS-OAD) is a good option, provided that the surface microstructure of substrate is properly engineered. Tungsten thin films with nominal thicknesses of 500 and 2500 nm were deposited onto SS plates subjected to conventional surface finishing treatments (sand blasting, sand paper abrasion and electrochemical polishing) to modify the surface topography and induce the appearance of different groove patterns. In the first part of this work we show how the topographical features of the SS substrates affect the typical nanocolumnar microstructure of OAD thin films of tungsten. Subsequently, we characterize the

spreading behavior of liquid lithium onto these tungsten nanocolumnar surfaces and critically discuss whether nanocolumnar tungsten thin films are a suitable option for the wetting and spreading of molten lithium. As a result, we reveal that the features of the tungsten nanocolumnar coating, characterized by a given height and void spaces between nanocolumns in the order of 1–2 μm, is critical for the spreading of molten lithium, while the existence of wider channels affects it very weakly. Moreover, it is shown that tungsten films deposited by MS-OAD on SS substrates subjected to conventional finishing procedures represent a good alternative to other more complex surface engineering procedures utilized for this purpose.

In Vitro and In Vivo Study of Titanium Grade IV and Titanium Grade V Implants with Different Surface Treatments

Díaz-Sánchez, RM; de-Paz-Carrion, A; Serrera-Figallo, MA; Torres-Lagares, D; Barranco, A; Leon-Ramos, JR; Gutiérrez-Pérez, JL
Metals, **10** (2020) 449
Abril, 2020 | **DOI:** 10.3390/met10040449

The aim of our study is to evaluate different implant surface treatments using TiIV and TiV in in vitro and in vivo studies. An in vitro study was established comprising four study groups with treated and untreated TiIV titanium discs (TiIVT and TiIVNT) and treated and untreated TiV titanium discs (TiVT and TiVNT). The surface treatment consisted in a grit blasting treatment with alumina and double acid passivation to modify surface roughness. The surface chemical composition and the surface microstructure of the samples were analyzed. The titanium discs were subjected to cell cultures to determine cell adhesion and proliferation of osteoblasts on them. The in vivo study was carried out on the tibia of three New Zealand rabbits in which 18 implants divided into three experimental groups were placed (TiIVT, TiIVNT, and TiVT). Micro-computed tomography (micro-CT) was performed to determine bone density around the implants. The results showed that cell culture had minor adhesion and cell proliferation in TiIVT and TiVT within the first 6 and 24 h. However, no differences were found after 48 h. No statistically significant differences were found in the in vivo micro-CT and histological study; however, there was a positive trend in bone formation in the groups with a treated surface. Conclusions: All groups showed a similar response to in vitro cell proliferation cultures after 48 h. No statistically significant differences were found in the in vivo micro-CT and histological study.

Optical properties of molybdenum in the ultraviolet and extreme ultraviolet by reflection electron energy loss spectroscopy

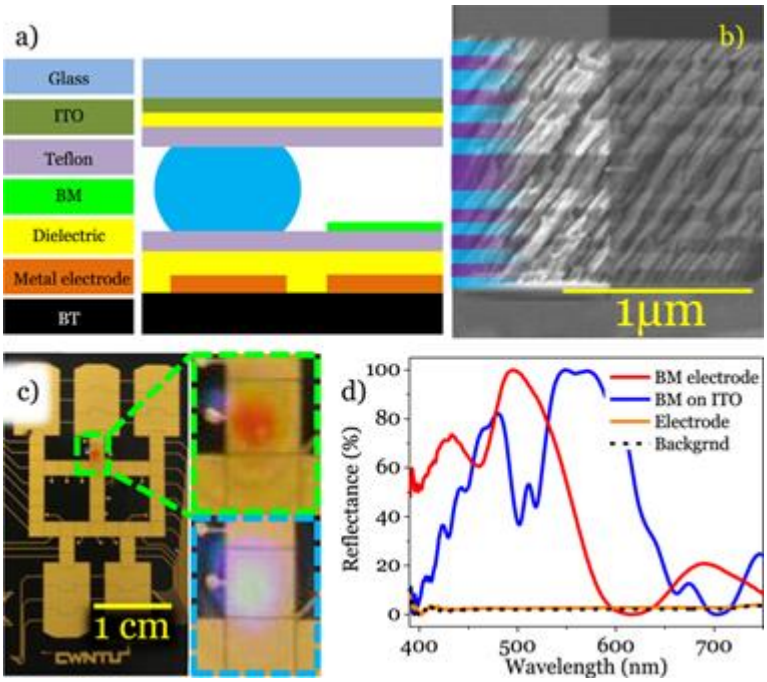
Pauly, N; Yubero, F; Tougaard, S
Applied Optics, **59** (2020) 4527-4532
Mayo, 2020 | **DOI:** 10.1364/AO.391014

Optical properties of polycrystalline molybdenum are determined from ultraviolet up to extreme ultraviolet by reflection electron energy loss spectroscopy (REELS). Calculations are performed within the dielectric response theory by means of the quantitative analysis of electron energy losses at surfaces QUEELS-$\epsilon(k,\omega)$-REELS software [Surf. Interface Anal. 36, 824 (2004)] that allows the simulation of inelastic scattering cross sections, using a parametric

energy loss function describing the optical response of the material. From this energy loss function, the real and imaginary parts of the dielectric function, the refractive index, and the extinction coefficient are deduced and compared with previously published results.

Optofluidic liquid sensing on electromicrofluidic devices

Oliva-Ramírez, M; Wang, SL; Rico-Gavira, V; López-Santos, C; Fan, SK; González-Elipe, AR
Materials Research Express, **7** (2020) 036407
Marzo, 2020 | **DOI:** 10.1088/2053-1591/ab7fdf



Electromicrofluidic (EMF) devices are used to handle and move tiny amounts of liquids by electrical actuation, including electrowetting-on-dielectric (EWOD) and dielectrophoresis (DEP). Monitoring the liquid characteristics in one of these devices requires suitable sensing transducers incorporated within the microfluidic structure. In the present work, we describe the incorporation of an optofluidic photonic transducer in an EMF device to monitor the refractive index of a liquid during its manipulation. The incorporated transducer consists of a responsive porous Bragg Microcavity (BM) deposited via physical vapor oblique angle deposition. Besides reporting the manufacturing procedure of the sensing-EMF device combining liquid handling and monitoring, the performance of the BM is verified by infiltrating several liquids dripped on its surface and comparing the responses with those of liquid droplets electrically moved from the delivery part of the chip to the BM location. This study proved that modified EMF devices can incorporate photonic structures to analyze very low liquid volumes (similar to 0.2 µL) during its handling.

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

Hydrogen Technologies

Chica, A; Fernández, A; Fernández, JR; Grasa, G; Laguna-Bercero, MA; Lázaro, MJ; Martínez, I; Pena, MA; Pinilla, JL; Sebastián, D; Serra, JM; Serra, M; Suelves, I; Valino, L
Boletín del Grupo Español del Carbón, **58** (2020) 30-37
Diciembre, 2020

LIBROS Y OTRAS PUBLICACIONES / BOOKS AND OTHER PUBLICATIONS

Advanced Strategies in Thin Films Engineering by Magnetron Sputtering
Palmero, A; Martín, N
Coatings, **10** (2020) 419
Abril, 2020 | **DOI:** 10.3390/coatings10040419
Editorial Material

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

International Conference on Nanomaterials Applied to Life Sciences 2020 (NALS 2020)
29 - 31 enero [Madrid, España]

Nanocolumnar coatings on implants exhibiting antibacterial properties
M.U. González; D. Medina-Cruz; I. Izquierdo-Barba; R. Álvarez; M. Fernández-Castro; W. Tien-Street; A. Vernet-Crua; S. Muñoz-Piña; A. García-Valenzuela; V. Rico; D. Arcos; I. Fernández-Martínez; A. Palmero; A. R. González-Elipe; M. Vallet-Regi; T. J. Webster; J. M. García-Martín
Comunicación Oral

Workshop on Plasma-Based Synthesis of Nanomaterials
10 -11 febrero [Praga, República Checa]

Geometry, Shadow and Sheaths for the Nanostructuration of Thin Films
A. García-Valenzuela; M. Oliva-Ramírez; A. Palmero; R. Álvarez; A. Borrás; M.C. López; A.R. González-Elipe
Conferencia Invitada

International Conference on Perovskite Solar Cells, Photonics and Optoelectronics (NIPHO20)
23 – 25 febrero [Sevilla, España]

Ultrathin Plasma Polymers: a new family of encapsulants to achieve waterproof perovskite solar cells
J. Idigoras; F.J. Aparicio; L. Contreras-Bernal; S. Ramons-Terron; M. Alcaire; J.R. Sánchez-Valencia; A. Borrás; J.A. Anta; A. Barranco
Comunicación Oral

Vacuum sublimation of Dopant-Free Crystalline Spiro-OMeTAD films to enhance the Stability of Perovskite Solar Cells
A. Barranco; C. López-Santos; J. Idigoras; F.J. Aparicio; J. Obrero; J. Castillo-Seoane; V. López-Flores; L. Contreras-Bernal; V. Rico; J. Ferrer; J.P. Espinós; A. Borrás; J.A. Anta; J.R. Sánchez-Valencia
Comunicación Oral

Photonic Online Meetup (POM)
25 junio [España]

Liquid switchable radial polarization converters made of sculptured thin films
M. Oliva; V. Rico; J. Gil; O. Arteaga; E. Bertrán; R. Serna; A.R. González-Elipe; F. Yubero
Comunicación Póster

Enhanced circular dichroism with superchiral surface waves
E. Mogni; G. Pellegrini; M.C. Ghezzi; E. Brenna; C. Zanchi; M. Tommasini; J. Gil-Rostra; F. Yubero; M. Celebrano; L. Duo; M. Finazzi; P. Biagioni
Comunicación Póster

Belgian Photonics Online Meetup (bePOM)
11 septiembre [Bélgica]

EUV characterization of surfaces by REELS
P. Tougaard; F. Yubero
Comunicación Póster

Plasma Surface Engineering (PSE2020)
7 - 10 septiembre [Erfurt, Alemania]

Tribomechanical properties of doped DLC coatings deposited by HiPIMS
J.A. Santiago; I. Fernández-Martínez; A. Wennberg; J.M. Molina-Aldareguia; M.A. Monclús; V. Bellido-González; R. González-Arrabal; T.C. Rojas; J.C. Sánchez-López
Comunicación Oral

Materiasl Science and Engineering MSE 2020
22-25 septiembre [Darmstadt, Alemania]

"In operando" Near Ambient Pressure Photoemission Analysis of Graphene Formation by Methanol Reforming through the Electrochemical Promotion of a Ni Catalyst
A.R. González-Elipe; J.P. Espinós; V. Rico; A. de Lucas-Consuegra; J. González-Cobos; J.R. Sánchez- Valencia
Comunicación Oral

7th Plasma Science & Entrepreneurship Workshop
2 - 3 noviembre [Bochum, Alemania]

Encapsulation of perovskite solar cells with ultrathin plasma polymers for moisture
Francisco J. Aparicio; Jesús Idígoras; Lidia Contreras-Bernal; Susana Ramos-Terron; Juan R. Sánchez-Valencia; Ana Borrás; Juan A. Anta; Ángel Barranco
Conferencia Invitada

FORMACION / TRAINING

FORMACIÓN DE GRADUADOS / MASTER DEGREE THESIS

Título:	**Estudio superficial de películas ultraporosas de dióxido de titanio para aplicaciones biomédicas**
Autor:	Triana Czermak Álvarez
Directores:	Ángel Barranco Quero
Grado:	Trabajo Fin de Grado
Fecha Defensa:	1 julio 2020

Título:	**One Dimensional nanotubes as highly sensitive Surface Enhanced Raman Scattering (SERS)-based sensors**
Autor:	Raquel Irene Cano Cordero
Directores:	Juan Ramón Sánchez Valencia y Ana Isabel Borrás Martos
Grado:	Trabajo Fin de Master
Centro:	Universidad de Sevilla
Fecha Defensa:	9 julio 2020

Título:	**1D and 3D nanomaterial fabrication and surface modification for developing Piezoelectric and Triboelectric Nanogenerators**
Autor:	Xabier García Casas
Directores:	Ángel Barranco Quero, Ana Isabel Borrás Martos y Leidy Marcela Martínez Tejada
Grado:	Trabajo Fin Master
Centro:	Universidad de Sevilla
Fecha Defensa:	9 julio 2020

Título:	**Recubrimientos duros de alto espesor basados en AlCrN**
Autor:	Oihane Hernández Rodríguez
Directores:	Juan Carlos Sánchez López
Grado:	Trabajo Fin de Master
Centro:	Universidad de Córdoba

Fecha Defensa: 21 julio 2020

Título: **Desarrollo de recubrimientos de absorción selectiva para energía solar de concentración**
Autor: Juan Martínez Romero
Directores: Juan Carlos Sánchez López y Teresa Cristina Rojas Ruiz
Grado: Trabajo Fin de Master
Centro: Universidad de Sevilla
Fecha Defensa: 10 diciembre 2020

DOCENCIA / TEACHING

Máster Oficial y Doctorado en Biotecnología Avanzada
Nanotecnología
María Aránzazu Díaz Cuenca
Lugar: Universidad Internacional de Andalucía. Universidad de Málaga

Investigadores de esta unidad participan en el Máster Interuniversitario "Láser, Plasma y Tecnología de Superficies" (página 236)

EQUIPAMIENTO CIENTÍFICO AVAILABLE EQUIPMENT

- Sistema de magnetron putttering 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)
- 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 extinción 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 ^{o}C (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).
- 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”.
- Material básico de laboratorio químico: PHmetro, agitadores, calefactores, estufa de secado a vacío, centrífuga.
- Reactores y material de vidrio convencional para síntesis de nanopartículas y catalizadores por via química.
- Rotavapor, sistemas de filtrado.
- Cámara seca MBRAUN.
- Dos campanas extractoras.
- Reactores catalíticos de lecho fijo para catálisis heterogenea solido-gas.
- Reactores catalíticos para catálisis heterogenea sólido-líquido.
- Sistemas controladores de flujo másico (gases y líquidos evaporados), bombas peristálticas para líquidos.
- Tres cromatógrafos de gases, columnas y detectors FID y TCD.
- 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.
- Buretas automáticas para medición de gases.
- 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.
- Tribómetro CSM (movimiento lineal y rotativo) para evaluación de coeficientes de fricción y desgaste.
- Tribómetro de alta temperatura (hasta 800 ^{o}C) –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.
- Cámara de deposición PVD-Magnetron sputtering (2 magnetron).
- Fuentes DC-Pulsada (ENI) ; RF (Trumpf); HiPIMS (Solvix)
- Horno tubular 1500 ^{o}C (Carbolite)
- Pulidora
- Ultrasonidos
- Microscopio óptico (Leica)
- Microscopio óptico Interferométrico 3D (Sensofar)

MATERIALES ÓPTICOS
OPTICAL MATERIALS

GRUPOS DE INVESTIGACIÓN

Materiales Coloidales
Colloidal Materials [642011]
http://colmat.icmse.csic.es

Materiales Ópticos Multifuncionales
Multifunctional Optical Materials [642013]
http://mom.icmse.csic.es

PERSONAL / PERSONNEL

Profesores de Investigación

Dr. Hernán R. Míguez García
Dr. Manuel Ocaña Jurado

Científicos Titulares

Dra. Ana Isabel Becerro Nieto
Dr. Mauricio E. Calvo Roggiani
Dr. Gabriel Lozano Barbero
Dra. Nuria Ofelia Núñez Álvarez

Investigadores Distinguidos

Dr. Juan F. Galisteo López

Contratados Juan de la Cierva

Dra. Laura Caliò

Doctores Contratados

Dra. Roxana Marisol Calderón Olvera
Dr. Víctor Castaing
Dr. Andrea Rubino
Dra. Ngo Thi Tuyen

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PROYECTOS DE INVESTIGACIÓN / RESEARCH PROJECTS

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

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

RESUMEN / ABSTRACT

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

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

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

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

brightness, colour quality and directionality of LED emission that conventional optical elements relying on geometrical optics provide.

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

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

Nanopartículas multifuncionales para la obtención de bioimágenes mediante luminiscencia, resonancia magnética y tomografía computerizada de Rayos X

Multifunctional nanoparticles for luminescent, magnetic resonance and X-ray computed tomography bioimaging

Código/Code:	RTI2018-094426-B-I00
Periodo/Period:	01-01-2019 / 31-12-20201
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	193.600 €
Investigador responsable/Research head:	Manuel Ocaña Jurado y Ana Isabel Becerro Nieto
Componentes/Research group:	Nuria O. Nuñez Álvarez

RESUMEN / ABSTRACT

El proyecto persigue la preparación de nanopartículas (NPs) multifuncionales con propiedades mejoradas y características (tamaño, estabilidad coloidal y toxicidad) adecuadas que puedan emplearse en más de una modalidad de obtención de imágenes de órganos, tejidos y células, cuyo principal interés radica en que mediante un único tipo de sonda se podría obtener información complementaria esencial para un diagnóstico clinico más riguroso. En concreto, se estudiarán sondas bifuncionales para la obtención de imágenes mediante luminiscencia y resonancia magnética (MRI) o tomografía computarizada de rayos X (CT), y sondas trifuncionales con utilidad para las tres modalidades mencionadas. Se abordarán dos tipos de biosondas luminiscentes. Por una parte, se diseñarán NPs luminiscentes constituidas por matrices singulares dopadas con cationes lantánidos (Nd^{3+} o Er^{3+}:Yb^{3+} o Tm^{3+}:Yb^{3+}), cuya excitación y emisión tiene lugar en la región del infrarrojo cercano (NIR) conocida como ventana biológica

(650-1800 nm), en la que las radiaciones no son dañinas para los tejidos y tienen alto poder de penetración. Por otra parte, se persigue la obtención de NPs cuya luminiscencia persiste después de eliminar la excitación, evitándose así los posibles efectos no deseados de ésta (autofluorescencia de los tejidos, radiaciones dañinas). En el primer caso, se pretende conseguir una mayor estabilidad química y térmica de las sondas mediante la selección de matrices tipo oxifluoruro más estables que las de tipo fluoruro propuestas hasta ahora, En el segundo caso, se abordarán sistemas con luminiscencia persistente en el NIR con composición $ZnGa_2O_4:Cr^{3+}$ y $Y_3Al_2Ga_3O_{12}:Ce^{3+},Cr^{3+},Nd^{3+}$, para los que es prioritario desarrollar nuevos métodos de síntesis que permitan la obtención de NPs uniformes, necesarias para este tipo de aplicaciones. Respecto a la modalidad MRI y en respuesta a la necesidad de desarrollar agentes de contraste para las más modernas técnicas que operan a campos magnéticos altos para aumentar la resolución de las imágenes y así obtener un diagnóstico más preciso, se planea desarrollar NPs constituidas por compuestos (oxifluoruros, vanadatos, fosfatos) de Dy y Ho. Por último, debido al alto número atómico de los elementos constituyentes de las sondas anteriores, es de esperar que éstas también tengan una alta capacidad de atenuación de rayos X, siendo por tanto también potencialmente útiles como agentes de contraste para CT. En este caso, el empleo de las NPs objeto de estudio aportará importantes ventajas respecto a los agentes comerciales utilizados en la actualidad que se traducen en un mayor control del tiempo de residencia en el organismo y de su biodistribución y, por tanto, en la posibilidad de disminuir las dosis utilizadas reportando así un beneficio para el paciente. El proyecto contempla tanto la fabricación de las sondas optimizadas como la exploración de su aplicabilidad al campo del diagnóstico clínico mediante la obtención de imágenes in vivo en ratones. El equipo investigador posee gran experiencia en la síntesis de NPs inorgánicas basadas en tierras raras y dispone de la mayoría de los medios necesarios para su caracterización. Además, dicho equipo cuenta con el apoyo de investigadores de otras instituciones, expertos en diversos aspectos del proyecto, que colaborarán en el desarrollo de algunas tareas del mismo como vienen haciendo desde hace varios años, lo que garantiza el correcto desarrollo de la propuesta.

The project pursues the preparation of multifunctional nanoparticles (NPs) with improved properties and suitable characteristics (size, colloidal stability and toxicity) that can be used to get images of cells, tissues and organs by means of more than one bioimaging technique, thus providing complementary information essential for a more reliable medical diagnosis. Specifically, we shall study bifunctional probes for both, luminescence and magnetic resonance (MRI) or luminescence and X-ray computed tomography (CT), and trifunctional probes that are useful for the three imaging techniques.Two types of luminescent probes will be addressed. On the one hand, luminescent NPs will be designed consisting of single matrices doped with lanthanide cations (Nd^{3+} o $Er^{3+}:Yb^{3+}$ o $Tm^{3+}:Yb^{3+}$), whose excitation and emission takes place in the near-infrared (NIR) region known as the biological window (650-1800 nm), in which radiation is not harmful to tissues and has a high penetration power. On the other hand, nanoprobes whose luminescence persists after ceasing the excitation will be also developed, thus avoiding the possible undesirable effects of the excitation radiation on the tissues. In the first case, our aim is to achieve greater chemical and thermal stability of the pro bes by selecting oxifluoride-type matrices, more stable than the fluoride-type matrices proposed so far. In the second case, the aim of the project resides in the exploration of new synthetic routes to obtain nanoparticulated $ZnGa_2O_4:Cr^{3+}$ and $Y_3Al_2Ga_3O_{12}:Ce^{3+},Cr^{3+},Nd^{3+}$, with uniform size and shape, which are essential for bioapplications. Regarding MRI technique, this project aims at developing NPs made up of Dy- and Ho-based oxifluorides, vanadates and phosphates in response to the

need of new contrast agents that work at high magnetic fields, which are increasingly being used in clinics to improve image resolution. Finally, due to the high atomic number of the constituent elements of the selected probes, it is expected that they show a high X-ray attenuation capacity, being therefore also useful as CT contrast agents. The advantage of the NPs proposed in this research with respect to the CT CAs currently used in clinics is the longer circulation time of the former, which will allow decreasing considerably the dosage to be given to the patient. The project contemplates both the manufacture of optimised probes and the exploration of their applicability to the field of medical diagnosis by obtaining "in vivo" images in mice. The research team has long experience in the synthesis of rare earths-based inorganic NPs and has most of the necessary equipment for their characterisation. The participation in the work plan of researchers from other institutions, with long expertise on various aspects of the project, who have successfully collaborated with the research team, gives further support to the viability of the proposal.

Materiales ópticos avanzados para dispositivos optoelectrónicos más eficientes | MODO Advanced optical materials for more efficient optoelectronic devices | MODO

Código/Code:	MAT2017-88584-R
Periodo/Period:	01-01-2018 / 30-09-2021
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	181.500 €
Investigador responsable/Research head:	Hernán Míguez García, Mauricio E. Calvo Roggiani
Componentes/Research group:	Juan F. Galisteo López, Gabriel S. Lozano Barbero

RESUMEN / ABSTRACT

El proyecto MODO busca optimizar el diseño óptico de dispositivos optoelectrónicos, ya sean fotovoltáicos o emisores de luz, con el objetivo de aumentar su eficiencia o dotarles de nueva funcionalidad. La hipótesis en la que se basa este proyecto es que este objetivo principal puede lograrse mediante la integración de materiales ópticos que permitan controlar la interacción radiación-materia en las láminas absorbentes u ópticamente activas del dispositivo. La estrategia propuesta consiste en realizar, secuencialmente, el diseño, preparación, caracterización e integración en prototipos de estructuras fotónicas de diverso tipo (cristales fotónicos, partículas metálicas, medios ópticos desordenados, superficies corrugadas), empleando principalmente técnicas de procesado en solución escalables y compatibles con las usualmente empleadas para la fabricación de los dispositivos objeto de estudio.

La tecnología optoelectrónica basada en perovskitas ha despertado en los últimos años un interés extraordinario debido a las altas eficiencias de conversión de energía lumínica en eléctrica, superiores al 20%, que se han alcanzado en un tiempo relativamente corto comparado

con la evolución de otras tecnologías fotovoltáicas. Por otro lado, presentan elevados rendimientos cuánticos de fotoemisión en el verde y el rojo, lo que también las convierte en candidatas a recubrimientos emisores de luz. Sin embargo, estas expectativas se ven parcialmente ensombrecidas debido a los problemas de estabilidad y potencial impacto ambiental que estas perovskitas presentan. Es uno de los objetivos prioritarios de este proyecto proponer soluciones a estos problemas concretos a través de diseños ópticos que permitan reducir tanto la cantidad de material empleado como la exposición de la lámina activa a ambientes que induzcan su degradación. Se presente ahondar en la compresión de los fenómenos que intervienen en la degradación fotoinducida de estos materiales en entornos de distinto tipo, lo que nos permitirá proponer soluciones concretas para desarrollar láminas de perovskita más estables y eficientes. Por otra parte, el proyecto MODO explorará la mejora de una nueva tecnología fotovoltáica, propuesta muy recientemente, basada en nanocristales semiconductores de $AgBiS_2$, de muy bajo impacto ambiental pero con una absorción óptica aún muy inferior al resto de celdas emergentes.

Simultáneamente, se aplicarán a dispositivos emisores de luz, basados en nanocristales semiconductores y compuestos orgánicos foto- y electroluminiscentes, conceptos orientados a la amplificación direccional de la luminiscencia en rangos espectrales seleccionados a través del control de la densidad local de estados fotónicos. Se explorará la posibilidad de controlar la dinámica de decaimiento de estados excitados a escala macroscópica y la de observar de emisión láser. En todos los casos, el rendimiento energético de los dispositivos objeto de estudio no ha sido optimizado anteriormente desde el punto de vista del diseño óptico.

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 is focused on the optimization of the optical design of optoelectronic devices, be they photovoltaic or light emitting ones, with the aim of increasing their efficiency or endow them with new functionalities. The hypothesis on which it is based is that this goal can be reached by means of the integration of optical materials that allow controlling the radiation-matter interaction in the absorbing or optically active layers of the device. The strategy herein proposed is based on the sequential realization of design, preparation, characterization and integration of devices of diverse photonic structures (photonic crystals, metallic particles, disordered optical media, corrugated surfaces) employing mainly solution processing techniques fully compatible with those used to fabricate the targeted devices. Optoelectronic technology based on perovskites has attracted a great deal of interest in the last years as a result of the high solar to electric power conversion efficiency, above 20%, that have been reached in a relatively short time compared to other photovoltaic technologies. At the same time, they present high photoemission quantum yields in the green and the red, which make them also good candidates as color converter layers for LEDs. However, these expectations are partially threatened by both the stability problems and potentially toxic environmental effects they present. It is one of the main goals of this project to propose solutions to specific drawbacks present in the optoelectronic technology based on hybrid perovskites through the implementation of optical designs that gives rise to a reduction of both the amount of material employed as well as the exposure to environments that typically degrade them. We seek to deepen our understanding of phenomena that give rise to the photoinduced degradation of

these materials when exposed to diverse environments, which will allow us to propose specific solutions to develop more stable and efficient perovskite layers. Simultaneously, concepts based on the strict control over the local density of photon states and oriented to the directional amplification of luminescence at selected spectral ranges will be applied to light emitting devices based on semiconductor nanocrystals as well as to photo- and electro-luminescent organic compounds. Full control over the excited state decay dynamics over large areas and observation of laser emission will also be sought after. In all cases, the energy efficiency of the targeted devices has not been optimized before from the point of view of the optical design.

The proposal is included in the framework of the Societal Challenge called "Secure, clean and efficient energy" and aims to develop photonic technology using nanotechnology tools and in the advanced materials field, all identified as Key Enabling Technologies KETs in the Spanish Strategy on Science and Technology, aligned with the European Program H2020.

Verificación de la existencia de fuerzas de Casimir repulsivas en la macroescala en láminas delgadas suspendidas y autosoportadas | VERSUS Verification of the existence of macroscale repulsive Casimir forces in suspended self-standing films | VERSUS

Código/Code:	FIS2017-91018-EXP
Periodo/Period:	01-11-2018 / 30-04-2021
Organismo Financiador/Financial source:	Ministerio de Economía y Competitividad
Importe total/Total amount:	36.300 €
Investigador responsable/Research head:	Hernán Míguez García
Componentes/Research group:	Mauricio E. Calvo Roggiani, Juan F. Galisteo López

RESUMEN / ABSTRACT

El proyecto VERSUS tiene como objetivo principal realizar la primera observación de fuerzas de Casimir-Lifshitz repulsivas en sistemas macroscópicos plano-paralelos. Para esto se centrará en el diseño, fabricación y caracterización de materiales ópticos que permitan controlar la intensidad y naturaleza de la fuerza de Casimir-Lifshitz, de forma que puedan observarse y caracterizarse fenómenos de levitación debido al balance de ésta y la fuerza gravitatoria. Esta propuesta, radicalmente novedosa, hace uso de técnicas de espectroscopía óptica (basadas en interferencia óptica entre los haces parcialmente reflejados y transmitido en las intercaras del sistema plano-paralelo) para caracterizar la distancia de equilibrio a la que el sistema levita sobre un sustrato. Para ello, se parte de diseños de materiales cuyas propiedades ópticas y densidades son tales que sumergidas en distintos fluidos leviten sobre ciertos sustratos como resultado del balance de esas fuerzas, como recientemente ha sido demostrado de forma teórica por el equipo solicitante. Nuestro grupo ha demostrado teóricamente que existen combinaciones de materiales que fabricados en forma de láminas delgadas (< 1 micra) pueden levitar a distancias del orden de las pocas decenas o centenas de nanómetros sobre un sustrato adecuado. En

particular, láminas de teflón, poliestireno y sílice inmersas en glicerol levitan sobre una oblea de silicio, siendo las distancias de equilibrio controlables y sintonizables a través del grosor de las láminas delgadas y la temperatura del sistema. Las láminas delgadas autosoportadas deben ser compactas, mecánicamente estables, de superficies planas, grosor controlado y químicamente afines al fluido en el que están sumergidas. La observación macroscópicas de fuerzas repulsivas de Casimir-Lifshitz, nunca reportada anteriormente, mediante medidas de espectroscopía óptica constituiría un logro sin precedentes en el campo del estudio de las interacciones fundamentales de la materia.

The ultimate goal of the VERSUS project is the first observation of repulsive Casimir-Lifshitz forces in macroscopic plane-parallel systems. To this end, it will focus on the design, fabrication, and characterization of optical materials that allow controlling the intensity and nature of the Casimir-Lifshitz force, so that levation phenomena can be observed and characterized due to the balance between the latter and gravity force. This radically new approcah makes use of optical spectroscopic techniques (based on optical interferometry between the partially reflected and transmitted light at the interfaces of the plane-parallel system) for characterizing the equilibrium distance at which the system levitates over a substrate. According to very recent results attained by the applicant group, it is possible to find materials whose optical constants and densities are such that when they are immersed in a fluid they can levitate over a substrate as a result of the aforementioned force balance. Our group has recently demonstrated theoretically that there is a number of materials that prepared in this films (<1 micrometer) can levitate several tens or hundreds of nanometers over a carefully selected substrate. Specifically, thin layers made of teflon, polystyrene or silicon dioxide immersed in glycerol are expected to levitate over a silicon wafer, being possible to tune the equilibrium distances at which such layers will be suspended through their thicknesses and temperature of the system. The devised selft-standing thin films (in single layers or multilayer arrangements) must be compact, mechanically stable, of smooth surfaces, of controlled thickness, and chemically compatible with the fluid in which they are immersed. The macroscopic observation of repulsive Casimir-Lifshitz forces, never reported before, through optical spectroscopic measurements would constitute an unprecedented milestone in the field of fundamental matter interactions.

Junta de Andalucía

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Andalucía se mueve con Europa

Desarrollo de Dispositivos Emisores de Luz basados en Perovskita Nanoestructurada | Nano-ABX LED
Development of light emitting devices based on nanostructured perovskite | Nano-ABX LED

Código/Code:	P18-RT-2291
Periodo/Period:	01-02-2020 / 31-01-2022
Organismo Financiador/Financial source:	Junta de Andalucía
Importe total/Total amount:	122.968 €
Investigador responsable/Research head:	Hernán R. Míguez García
Componentes/Research group:	Juan Francisco Galisteo López, Mauricio E. Calvo Roggiani, Gabriel S. Lozano Barbero

RESUMEN / ABSTRACT

El proyecto Nano-ABX LED se centra en encontrar vías de solución a los principales retos que enfrenta el campo de la emisión de luz basada en perovskitas. Estos son la inestabilidad química y térmica de las perovskitas, así como la dificultad de mantener una eficiencia cuántica elevada independientemente del color de emisión, lo que dificulta la obtención tanto de una gama de colores variada como de distintos tonos de blanco (i.e., distintas temperaturas de color).

El proyecto Nano-ABX LED surge con la motivación de encontrar soluciones a estos problemas. Partiendo de resultados preliminares recientes del grupo de Materiales Ópticos Multifuncionales, se intentará demostrar que la integración de nanocristales de perovskita híbrida en el interior de matrices con porosidad controlada mejora extraordinariamente la estabilidad ambiental de estos materiales, un aspecto que el grupo solicitante de esta propuesta ha estudiado en profundidad, así como permite aumentar el rendimiento cuántico luminiscente a longitudes de onda de emisión controladas. En otra vertiente del proyecto, se explorará el aumento de eficiencia y prestaciones (direccionalidad, control espectral) de los dispositivos a través de la integración de distintas estructuras fotónicas, tomando como punto de partida.

The Nano-ABX LED project focuses on finding ways to solve the main challenges facing the field of perovskite-based light emission. These are the chemical and thermal instability of perovskites, as well as the difficulty of maintaining a high quantum efficiency regardless of the emission color, which makes it difficult to obtain both a varied color range and different shades of white (ie, different temperatures color).

The Nano-ABX LED project arises with the motivation to find solutions to these problems. Based on recent preliminary results of the Multifunctional Optical Materials Group, an attempt will be made to demonstrate that the integration of hybrid perovskite nanocrystals inside matrices with controlled porosity dramatically improves the environmental stability of these materials, an aspect that the group requesting this proposal has studied in depth, as well as it allows to increase the luminescent quantum efficiency at controlled emission wavelengths. In another aspect of the project, the increase in efficiency and performance (directionality, spectral control) of the devices will be explored through the integration of different photonic structures, taking as a starting point.

OTROS PROYECTOS / OTHER PROJECTS

Desarrollo de Dispositivos Emisores de Luz basados en Perovskita Nanoestructurada

Código/Code:	201960E090
Periodo/Period:	01-09-2019 / 31-08-2022
Organismo Financiador/Financial source:	CSIC (Intramural)
Importe total/Total amount:	120.345 €
Investigador responsable/Research head:	Hernán Míguez García

Energía Solar y nanociencia. Kit demostrativo

Código/Code:	201560E056
Periodo/Period:	01-01-2020 / 31-12-2020
Organismo Financiador/Financial source:	CSIC
Importe total/Total amount:	2.300 €
Investigador responsable/Research head:	Mauricio Calvo Roggiani

AYUDAS PARA LA ADQUISICIÓN DE EQUIPOS

Sistema de caracterización óptica avanzada en atmósfera inerte (EQC2019-005556-P)
Financia: Ministerio de Ciencia e Innovación y Consejo Superior de Investigaciones Científicas
Importe Concedido: 121.917,11 €
Periodo: 1-1-2019 / 31-12-2021
Cofinanciado por el Grupo de Investigación "Materiales Ópticos Multifuncionales"

CONVENIOS Y CONTRATOS / CONTRACTS AND AGREEMENTS

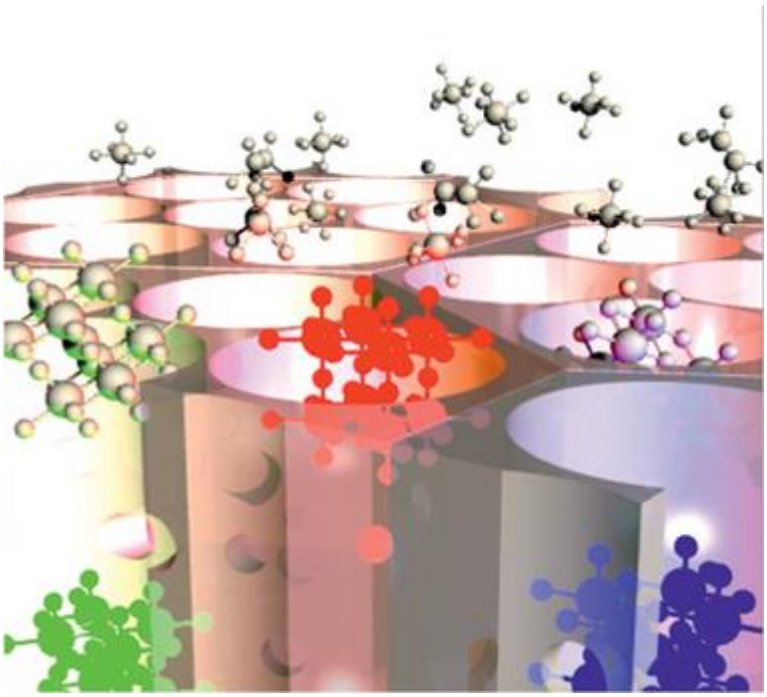
SOLAR BLUE

Periodo/Period:	01-01-2019 / 30-06-2020
Organismo Financiador/Financial source:	CAPSUN TECHNOLOGIES, S.L.
Importe total/Total amount:	50.000 €
Investigador responsable/Research head:	Hernán Míguez García

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

Mesoporous Matrices as Hosts for Metal Halide Perovskite Nanocrystals

Rubino, A; Caliò, L; García-Bennett, A; Calvo, ME; Míguez, H
Advanced Optical Materials, **8** (2020) 1901868
Febrero, 2020 | **DOI:** 10.1002/adom.201901868



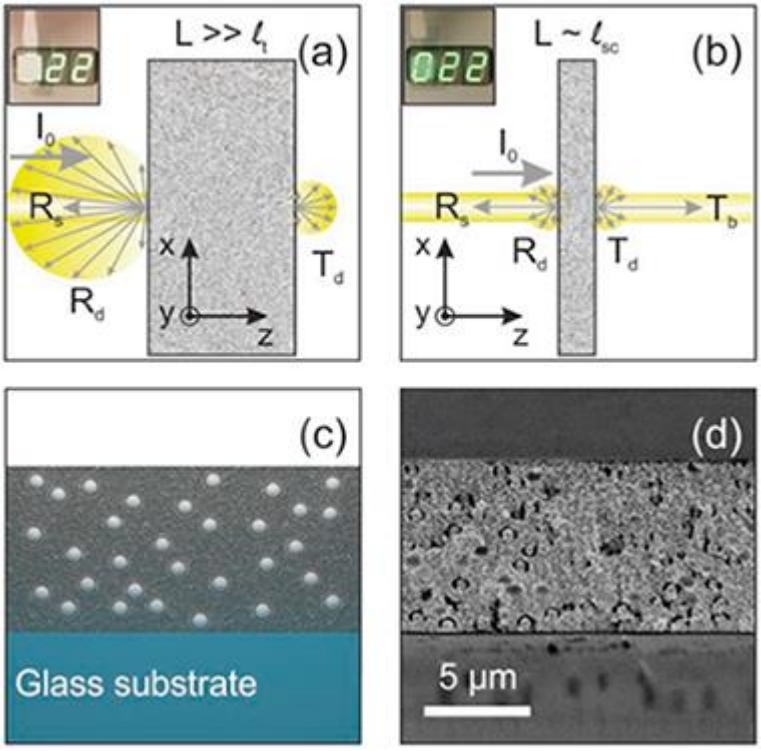
Several works have recently demonstrated that perovskite nanocrystals can be controllably formed within a variety of porous matrices employing diverse synthetic strategies. By means of the fine tuning of the pore size distribution, the thickness and composition of the walls, the geometry of the void network and its topology, strict control over the structural and morphological parameters of the hosted semiconductor can be achieved, determining its optical absorption and emission properties. Furthermore, porous hosts provide the guest semiconductor with enhanced stability and versatility in terms of processing, which favors its integration in devices. This article provides a comprehensive review of the different approaches proposed, as well as a discussion on the relevance they may have for the development of nanostructured perovskite-based optoelectronics. A critical assessment of the optical quality of the hybrid perovskite nanomaterials so obtained is presented, as well as an analysis of the fundamental and applied aspects of the nanocrystal-matrix interaction and a projected prospect of their impact in the fields of artificial lighting and renewable energy.

Finite Size Effects on Light Propagation throughout Random Media: Relation between Optical Properties and Scattering Event Statistics

Miranda-Munoz, JM; Esteso, V; Jiménez-Solano, A; Lozano, G; Míguez, H
Advanced Optical Materials, **8** (2020) 1901196
Enero, 2020 | **DOI:** 10.1002/adom.201901196

This work introduces a thorough analysis of light transport in thin optically disordered media. The diffusive properties of a turbid material are generally dictated by the transport mean free

path, lt. For depths larger than this characteristic length, light propagation can be considered fully randomized. There is however a range of thicknesses for which light becomes only partly



randomized, as it only undergoes a single or very few scattering events. The effects of such finitude are experimentally and theoretically studied on the optical properties of the material, such as the angular distribution of scattered light. Simulations provide insight into the phenomena that occur within the optically disordered slab, like the number of scattering events that photons undergo during propagation throughout the material, as a function of the built-in wavelength dependent scattering mean free path, l_{sc}. This approach provides fundamental information about photon transport in finite optically random media, which can be put into practice to design diffusers with specific requirements in terms of the spectral and angular properties of the scattered light.

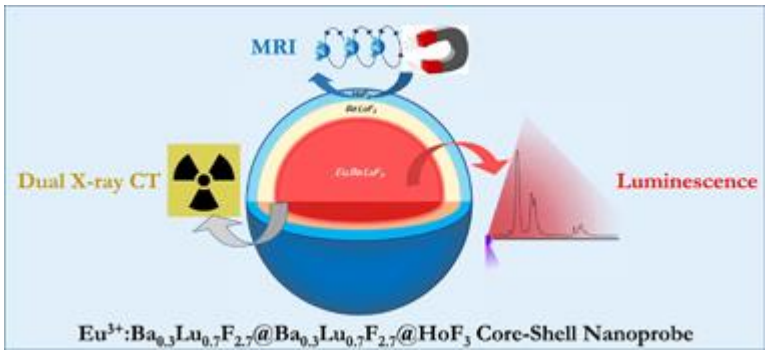
Design of a nanoprobe for high field magnetic resonance imaging, dual energy X-ray computed tomography and luminescent imaging

González-Mancebo, D; Becerro, AI; Corral, A; García-Embid, S; Balcerzyk, M; García-Martín, ML; de la Fuente, JM; Ocaña, M

Journal of Colloid and Interface Science, **573** (2020) 278-286

Agosto, 2020 | **DOI:** 10.1016/j.jcis.2020.03.101

The combination of different bioimaging techniques, mainly in the field of oncology, allows circumventing the defects associated with the individual imaging modalities, thus providing a more reliable diagnosis. The development of multimodal endogenous probes that are simultaneously suitable for various imaging modalities, such as magnetic resonance imaging (MRI), X-ray computed tomography (CT) and luminescent imaging (LI) is, therefore, highly

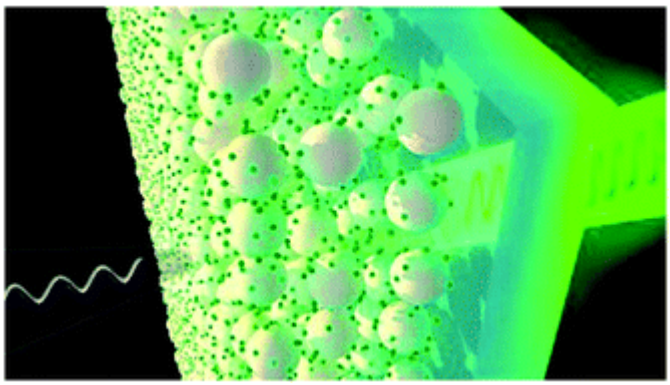


recommended. Such probes should operate in the conditions imposed by the newest imaging equipment, such as MRI operating at high magnetic fields and dual-energy CT. They should show, as well, high photoluminescence emission intensity for their use in optical imaging and present good biocompatibility. In this context, we have designed a single nanoprobe, based on a core-shell architecture, composed of a luminescent Eu^{3+}:$Ba_{0.3}Lu_{0.7}F_{2.7}$ core surrounded by an external HoF_3 shell that confers the probe with very high magnetic transverse relaxivity at high field. An intermediate, optically inert $Ba_{0.3}Lu_{0.7}F_{2.7}$ layer was interposed between the core and the shell to hinder Eu^{3+}-Ho^{3+} cross-relaxation and avoid luminescence quenching. The presence of Ba and Lu, with different K-edges, allows for good X-ray attenuation at high and low voltages. The core-shell nanoparticles synthesized are good potential candidates as trimodal bioprobes for MRI at high field, dual-energy CT and luminescent imaging.

Efficient third harmonic generation from FAPbBr(3) perovskite nanocrystals

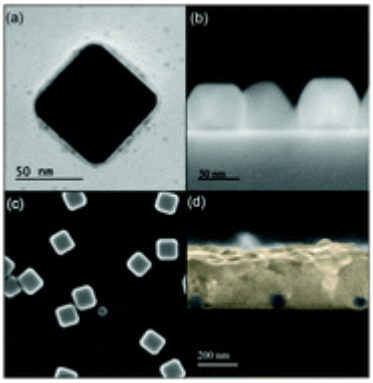
Rubino, A; Huq, T; Dranczewski, J; Lozano, G; Calvo, ME; Vezzoli, S; Míguez, H; Sapienza, R
Journal of Materials Chemistry C, **8** (2020) 15990-15995
Diciembre, 2020 | **DOI:** 10.1039/d0tc04790b

The development of versatile nanostructured materials with enhanced nonlinear optical properties is relevant for integrated and energy efficient photonics. In this work, we report third harmonic generation from organic lead halide perovskite nanocrystals, and more specifically from formamidinium lead bromide nanocrystals, ncFAPbBr$_3$, dispersed in an optically transparent silica film. Efficient third order conversion is attained for excitation in a wide spectral range in the near infrared (1425 nm to 1650 nm). The maximum absolute value of the modulus of the third order nonlinear susceptibility of ncFAPbBr$_3$, $\chi^{(3)NC}$, is derived from modelling both the linear and nonlinear behaviour of the film and is found to be $\chi^{(3)NC} = 1.46 \times 10^{-19}$ m^2V^{-2} (or 1.04×10^{-11} esu) at 1560 nm excitation wavelength, which is of the same order as the highest previously reported for purely inorganic lead halide perovskite nanocrystals (3.78×10^{-11} esu for ncCsPbBr$_3$). Comparison with the experimentally determined optical constants demonstrates that maximum nonlinear conversion is attained at the excitonic resonance of the perovskite nanocrystals where the electron density of states is largest. The ease of synthesis, the robustness and the stability provided by the matrix make this material platform attractive for integrated nonlinear devices.



Localized surface plasmon effects on the photophysics of perovskite thin films embedding metal nanoparticles

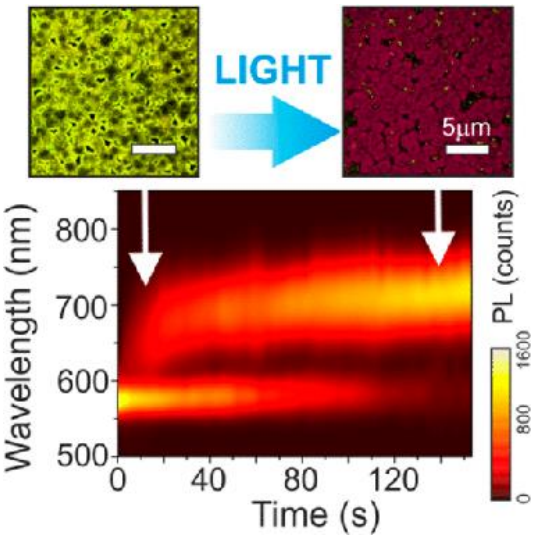
Bayles, A; Carretero-Palacios, S; Calió, L; Lozano, G; Calvo, ME; Míguez, H
Journal of Materials Chemistry C, **8** (2020) 916-921
Marzo, 2020 | **DOI:** 10.1039/c9tc05785d



Herein we provide direct experimental evidence that proves that the photophysical properties of thin methylammonium lead iodide perovskite films are significantly enhanced by localized surface plasmon resonances (SPRs). Observations are well supported by rigorous calculations that prove that improved light harvesting can be unequivocally attributed to plasmonic scattering and near field reinforcement effects around silver nanoparticles embedded within the semiconductor layer. Adequate design of the localized SPR allows raising the absorptance of a 300 nm thick film at well-defined spectral regions while minimizing the parasitic absorption from the metallic inclusions. Measured enhancements can be as large as 80% at specific wavelengths and 20% when integrated over the whole range at which SPR occurs, in agreement with theoretical estimations. Simultaneously, the characteristic quenching effect that the vicinity of metals has on the photoluminescence of semiconductors is largely compensated for by the combined effect of the enhanced photoexcitation and the higher local density of photon states occurring at SPR frequencies, with a two fold increase of the perovskite photoemission efficiency being measured.

Local Rearrangement of the Iodide Defect Structure Determines the Phase Segregation Effect in Mixed-Halide Perovskites

Tiede, DO; Calvo, ME; Galisteo-López, JF; Míguez, H
Journal of Physical Chemistry Letters, **11** (2020) 4911-4916
Junio, 2020 | **DOI:** 10.1021/acs.jpclett.0c01127



Mixed-halide perovskites represent a particularly relevant case within the family of lead-halide perovskites. Beyond their technological relevance for a variety of optoelectronic devices, photoinduced structural changes characteristic of this type of material lead to extreme photophysical changes that are currently the subject of intense debate. Herein we show that the conspicuous photoinduced phase segregation characteristic of these materials is primarily the result of the local and metastable rearrangement of the iodide sublattice. A local photophysical study comprising spectrally resolved laser scanning confocal microscopy is employed to find a correlation between the defect density and the

dynamics of photoinduced changes, which extend far from the illuminated region. We observe that iodide-rich regions evolve much faster from highly defective regions. Also, by altering the material composition, we find evidence for the interplay between the iodide-related defect distribution and the intra- and interdomain migration dynamics giving rise to the complexity of this process.

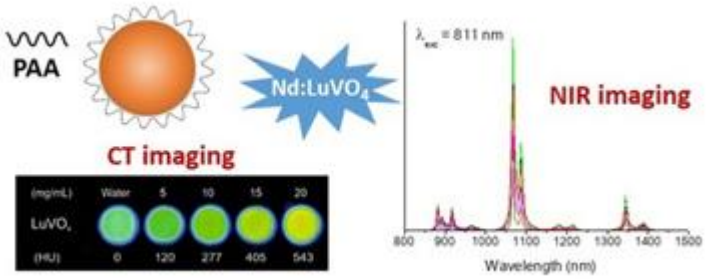
Bimodal Nd-Doped $LuVO_4$ Nanoprobes Functionalized with Polyacrilic Acid for X-Ray Computed Tomography and NIR Luminescent Imaging

Nuñez, NO; Cusso, F; Cantelar, E; Martín-Gracia, B; de la Fuente, JM; Corral, A; Balcerzyk, M; Ocaña, M

Nanomaterials, **10** (2020) 149

Enero, 2020 | **DOI:** 10.3390/nano10010149

Uniform Nd^{3+}-doped $LuVO_4$ nanophosphors have been synthesized for the first time in literature by using a poliol-based method at 120 ^oC from Nd^{3+} and vanadate precursors. After optimizing the Nd doping level, these phosphors present intense luminescence in the near-infrared biological windows. The X-ray attenuation capacity of the optimum nanophosphor has been found to be higher than that of a commercial X-ray computed tomography contrast agent. After surface coating with polyacrylic acid, such nanoparticles present high colloidal stability in physiological pH medium and high cell viability. Because of these properties, the developed Nd^{3+}-doped $LuVO_4$ nanoparticles have potential applications as a bimodal probe for NIR luminescent bioimaging and X-ray computed tomography.



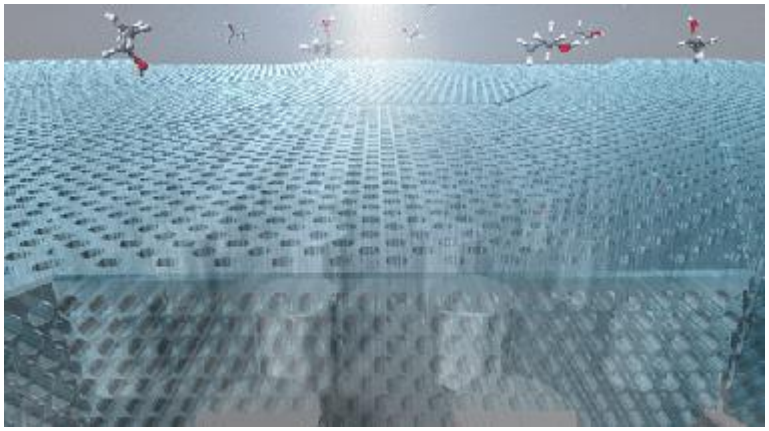
Optical Responses of Localized and Extended Modes in a Mesoporous Layer on Plasmonic Array to Isopropanol Vapor

Murai, S; Cabello-Olmo, E; Kamakura, R; Calvo, ME; Lozano, G; Atsumi, T; Míguez, H; Tanaka, K

Journal of Physical Chemistry C, **124** (2020) 5772-5779

Marzo, 2020 | **DOI:** 10.1021/acs.jpcc.9b10999

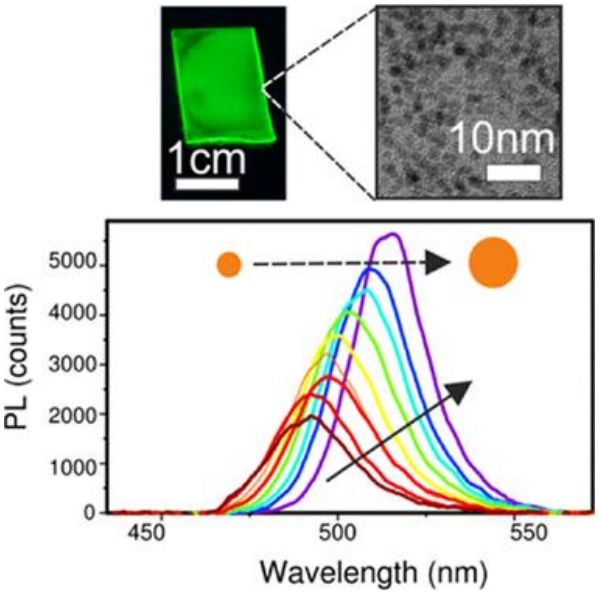
Mesoporous silica features open and accessible pores that can intake substances from the outside. The combination of mesoporous silica with plasmonic nanostructures represents an interesting platform for an optical sensor based on the dependence of plasmonic modes on the refractive index of the medium in which metallic nanoparticles are embedded. However, so far only a limited number of plasmonic nanostructures are combined with mesoporous silica,



including random dispersion of metallic nanoparticles and flat metallic thin films. In this study, we make a mesoporous silica layer on an aluminum nanocylinder array. Such plasmonic arrangements support both localized surface plasmon resonances (LSPRs) and extended modes which are the result of the hybridization of LSPRs and photonic modes extending into the mesoporous layer. We investigate in situ optical reflectance of this system under controlled pressure of isopropanol vapor. Upon exposure, the capillary condensation in the mesopores results in a gradual spectral shift of the reflectance. Our analysis demonstrates that such shifts depend largely on the nature of the modes; that is, the extended modes show larger shifts compared to localized ones. Our materials represent a useful platform for the field of environmental sensing.

Monitoring, Modeling, and Optimization of Lead Halide Perovskite Nanocrystal Growth within Porous Matrices

Tiede, DO; Rubino, A; Calvo, ME; Galisteo-López, JF; Míguez, H
Journal of Physical Chemistry C, **124** (2020) 8041-8046
Abril, 2020 | **DOI:** 10.1021/acs.jpcc.0c01750

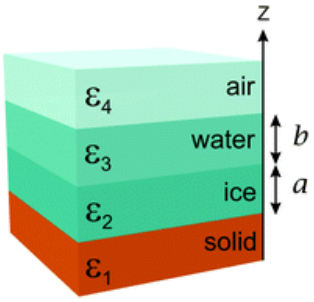


The growth of lead halide perovskites within metal-oxide nanoporous films has been recently considered as a means to obtain chemically and photostable ligand-free high-quality nanocrystals (NCs). The growth process, governed by the reactions taking place in nanoreactors dictated by the matrix pore size, has not been explored so far. In this work, we use photoluminescence as a tool to monitor the growth of perovskite NCs within the void network of an optically transparent matrix. We consider the effect of different external factors, such as temperature, light illumination, or precursor concentration, on the growth dynamics, and discuss a possible formation mechanism of the confined perovskite NCs. Based on this analysis, guidelines that could serve to improve the fabrication and optoelectronic quality of this type of NCs are also proposed.

Premelting of ice adsorbed on a rock surface

Esteso, V; Palacios, SC; MacDowell, LG; Fiedler, J; Parsons, DF; Spallek, F; Míguez, H; Persson, C; Buhmann, SY; Brevik, I; Bostrom, M

Physical Chemistry Chemical Physics, **22** (2020) 11362-11373
Mayo, 2020 | **DOI:** 10.1039/c9cp06836h

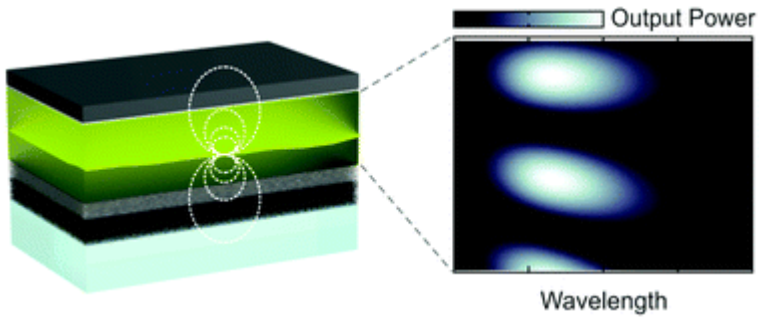


Considering ice-premelting on a quartz rock surface (i.e. silica) we calculate the Lifshitz excess pressures in a four layer system with rock-ice-water-air. Our calculations give excess pressures across (1) ice layer, (2) water layer, and (3) ice-water interface for different ice and water layer thicknesses. We analyse equilibrium conditions where the different excess pressures take zero value, stabilized in part by repulsive Lifshitz interactions. In contrast to previous investigations which considered varying thickness of only one layer (ice or water), here we present theory allowing for simultaneous variation of both layer thicknesses. For a given total thickness of ice and water, this allows multiple alternative equilibrium solutions. Consequently the final state of a system will depend on initial conditions and may explain variation in experimental measurements of the thicknesses of water and ice layers.

Dipole reorientation and local density of optical states influence the emission of light-emitting electrochemical cells

Jiménez-Solano, Alberto; Martínez-Sarti, Laura; Pertegas, Antonio; Lozano, Gabriel; Bolink, Henk J; Míguez, Hernan

Physical Chemistry Chemical Physics, **22** (2020) 92-96
Enero, 2020 | **DOI:** 10.1039/c9cp05505c



Herein, we analyze the temporal evolution of the electroluminescence of light-emitting electrochemical cells (LECs), a thin-film light-emitting device, in order to maximize the luminous power radiated by these devices. A careful analysis of the spectral and angular distribution of the emission of LECs fabricated under the same experimental conditions allows describing the dynamics of the spatial region from which LECs emit, i.e. the generation zone, as bias is applied. This effect is mediated by dipole reorientation within such an emissive region and its optical environment, since its spatial drift yields a different interplay between the intrinsic emission of the emitters and the local density of optical states of the system. Our results demonstrate that engineering the optical environment in thin-film light-emitting devices is key to maximize their brightness.

Optical interference effects on the Casimir-Lifshitz force in multilayer structures

Esteso, V; Carretero-Palacios, S; Míguez, H
Physical Review A, **101** (2020) 033815
Marzo, 2020 | **DOI:** 10.1103/PhysRevA.101.033815

The Casimir-Lifshitz force F(C-L) between planar objects when one of them is stratified at the nanoscale is herein investigated. Layering results in optical interference effects that give rise to a modification of the optical losses, which, as stated by the fluctuation-dissipation theorem, should affect the Casimir-Lifshitz interaction. On these grounds, we demonstrate that, by nanostructuring the same volume of dielectric materials in diverse multilayer configurations, it is possible to access F(C-L) of attractive or repulsive nature, even getting canceled, at specific separation distances.

Internal quantum efficiency and time signals from intensity-modulated photocurrent spectra of perovskite solar cells

Riquelme, A; Galvez, FE; Contreras-Bernal, L; Míguez, H; Anta, JA
Journal of Applied Physics, **128** (2020) 133103
Octubre, 2020 | **DOI:** 10.1103/PhysRevA.101.033815

Intensity Modulated Photocurrent Spectroscopy (IMPS) is a small-perturbation optoelectronic technique that measures the quantum efficiency of a photoelectrochemical device as a function of optical excitation frequency. Metal Halide Perovskites (MHPs) are mixed electronic-ionic semiconductors with an extraordinary complex optoelectronic behavior and a record efficiency surpassing 25%. In this paper, we propose a simplified procedure to analyze IMPS data in MHPs based on the analysis of the internal quantum efficiency and the time signals featuring in the frequency spectra. In this procedure, we look at the change of each signal when optical excitation wavelength, photon flux, and temperature are varied for an archetypical methyl ammonium lead iodide solar cell. We use drift-diffusion modeling and comparison with relatively simpler dye-sensitized solar cells (DSC) with viscous and non-viscous electrolytes to help us to understand the origin of the three signals appearing in MHP cells and the measurement of the internal quantum efficiency.

CONGRESOS Y REUNIONES INTERNACIONALES / INTERNATIONAL CONGRESS AND MEETINGS

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

International Conference on Perovskite Solar Cells, Photonics and Optoelectronics (NIPHO20)

23 – 25 febrero [Sevilla, España]

Hernán Míguez García (Conference Organizer)

CMD2020GEFES mini-colloquium. Casimir Effect and Heat Transfer Advances
31 agosto – 31 septiembre [online]

Victoria Esteso (Comité Organizador)

COMUNICACIONES / COMMUNICATIONS

International Conference on Nanomaterials Applied to Life Sciences 2020 (NALS 2020)
29 - 31 enero [Madrid, España]

Uniform Europium-Doped Sodium Lanthanum Tungstate and Molybdate (NaLa(XO4)2, X = Mo,W) Probes for Luminescent and X-ray Computed Tomography Bioimaging
Nuria O. Núñez; Ana I. Becerro; Gabriel Lozano; Manuel Ocaña
Comunicación Póster

International Conference on Perovskite Solar Cells, Photonics and Optoelectronics (NIPHO20)
23 – 25 febrero [Sevilla, España]

Determination of the optical constants of metal-halide perovskite nanocrystals
Gabriel Lozano
Conferencia Invitada

Porous metal oxide matrices as templating host of ABX_3 perovskite nanocrystals
Mauricio Calvo; Laura Caliò; Andrea Rubino; Hernán Míguez
Comunicación Oral

Lattice Dynamics of $MAPbI_3$ Nanocrystals
Andrea Rubino; Mauricio Calvo; Hernan Míguez; Alejandro Goñi
Comunicación Póster

Experimental evidence of plasmonic-enhanced absorption and emission of perovskite thin film embedding silver nanocubes
Laura Caliò; Aaron Bayles; Sol Carretero-Palacios; Gabriel Lozano; Mauricio Calvo; Hernán Míguez
Comunicación Póster

Nanolight
26 - 29 marzo [Huesca, España]

Solution processed nanomaterials for photonics and optoelectronics
Hernán Míguez
Conferencia Invitada

Experimental evidence of plasmonic-enhanced absorption and emission of perovskite thin film embedding silver nanocubes
Laura Caliò; Aaron Bayles; Sol Carretero-Palacio; Gabriel Lozano; Mauricio Calvo; Hernán Míguez
Conferencia Invitada

Nanophosphor-based photonics
Elena Cabello Olmo; Donling Geng; Gabriel Lozano; Hernán Míguez
Comunicación Póster

Optical disorder for enhanced colour conversion and efficient bifacial dye-sensitized solar cells. The Mie glass: fabrication and characterization
H. Míguez; G. Lozano; J.M. Miranda; J.M. Viaña
Comunicación Póster

Optical resonators based on Casimir forces and related aspects
V. Esteso
Contribución Oral

12th International Conference on Hybrid and Organic Photovoltaics - HOPV20 Online Conference
26 - 29 mayo [online]

Theoretical design and experimental evidence of enhanced absorption and emission by LSPR effects in perovskite thin film embedding silver nanocubes
Laura Calió; Aaron Bayles; Sol Carretero-Palacios; Gabriel Lozano; Mauricio E. Calvo; Hernán Míguez
Comunicación Póster

10th International Colloids Conference 2020
14 – 17 junio [Palma, España]

Bimodal luminescent and computed tomography nanoprobe based on Eu^{3+}:$(H_3O)Lu_3F_{10}$
A. I. Becerro; D. González-Mancebo; A. Corral; M. Balcerzyk; M. Ocaña
Conferencia Invitada

CMD2020GEFES
31 agosto – 4 septiembre [online]

Persistent Photocarrier Accumulation and Depletion in $LaAlO_3/SrTiO_3$ Quantum Wells
Laura Caliò
Conferencia

EOS Annual Meeting (EOSAM) 2020
7-11 September 2020. [Joensuu, Finland]

TOM 11- Thermal radiation and energy management
Optical Resonators based on Casimir Forces
V. Esteso, S. Carretero, H. Míguez
Contribución Oral invitada

Materials Science and Engineering MSE2020
22 – 25 septiembre [Darmstadt, Alemania]

Efficient Transparent nanophosphor films for visible light generation
Elena Cabello Olmo;Dongling Geng;Gabriel Lozano;Hernán Míguez
Comunicación Oral

Solution processed photonic structures for nanophosphor emission control
Elena Cabello Olmo; Dongling Geng; Gabriel Lozano; Hernán Míguez
Comunicación Oral

CONGRESOS Y REUNIONES NACIONALES / NATIONAL CONGRESS AND MEETINGS

COMUNICACIONES / COMMUNICATIONS

PEROVSKITA19 | Research Network. Perovskites for solar energy conversion and optoelectronics
6 mayo [Castellón, España]

Temperature-dependent photoluminescence of $MAPbI_3$ nanocrystals
Andrea Rubino, Mauricio Calvo, Hernán Míguez
Comunicación Oral

Confined $APbX_3$ nanocrystals photophysics
Andrea Rubino; Mauricio Calvo; Hernan Míguez
Comunicación Oral

FORMACION / TRAINING

TESIS DOCTORALES/ DOCTOR DEGREE THESIS

Título: **Diseño de sondas multifuncionales basadas en nanopartículas de fluoruros de lantánidos para el diagnóstico médico por imagen mediante luminiscencia, resonancia magnética y tomografía computarizada**
Autor: Daniel González Mancebo
Directores: Ana Isabel Becerro Nieto y Manuel Ocaña Jurado
Calificación: Sobresaliente "Cum Laude"
Centro: Universidad de Sevilla
Fecha Defensa: 9 marzo 2020

Título: **ABX_3 perovskite nanocrystals in porous matrices**
Autor: Andrea Rubino
Directores: Hernán Míguez García y Mauricio Calvo Roggiani
Calificación: Sobresaliente "Cum Laude"
Centro: Universidad de Sevilla
Fecha Defensa: 17 diciembre 2020
M. Europea: 25 diciembre 2020

FORMACIÓN DE GRADUADOS / MASTER DEGREE THESIS

Título: **Síntesis de nanopartículas de $NaDy(MoO_4)_2$ con vistas a posibles aplicaciones biomédicas**
Autor: María Ángeles Paulete Romero
Directores: Nuria Núñez Álvarez
Grado: Trabajo Fin de Grado
Centro: Universidad de Sevilla
Fecha Defensa: 15 julio 2020

Título: **Perovskite nanocrystals embedded in insulating scaffold for new light emitting diodes technologies**
Autor: Andrea Zanetta
Directores: Hernán Míguez García, Laura Caliò
Grado: Trabajo Fin de Master
Centro: Universidad de Sevilla
Fecha Defensa: 20 abril 2020

Título: **Desarrollo de una sonda trimodal basada en nanopartículas de vanadatos de lantánidos para aplicaciones biomédicas**
Autor: Corina Rodríguez Esteban
Directores: Manuel Ocaña Jurado, Nuria Núñez Álvarez
Grado: Trabajo Fin de Master
Centro: Universidad de Sevilla
Fecha Defensa: 9 julio 2020

Título: **Strong coupling phenomena between light and excitons in optical cavities**
Autor: Clara Bujalance Aguilera
Directores: Laura Caliò, Hernán Míguez García
Grado: Trabajo Fin de Master
Centro: Universidad de Sevilla
Fecha Defensa: 9 julio 2020

Título: **Fenómenos de acoplamiento fuerte colectivo entre semiconductores de tipo perovskita y modos de cavidad óptica**
Autor: Hilario Espinós Martínez
Directores: Felipe Gutiérrez Mora, Hernán Ruy Míguez García
Grado: Trabajo Fin de Grado
Centro: Universidad de Sevilla
Fecha Defensa: 10 julio 2020

Título: **Caracterización fotofísica de nanofósforos basados en cationes de tierras raras para recubrimientos de LEDs**
Autor: Fernando Escobar García
Directores: Felipe Gutiérrez Mora, Gabriel Sebastián Lozano Barbero
Grado: Trabajo Fin de Grado
Centro: Universidad de Sevilla
Fecha Defensa: 10 julio 2020

Título: **Efecto del desorden óptico en la fuerza de Casimir**
Autor: Yassine Lamouaraa Sedlackova
Directores: Felipe Gutiérrez Mora, Victoria Esteso Carrizo
Grado: Trabajo Fin de Grado
Centro: Física de la Materia Condensada. Universidad de Sevilla
Fecha Defensa: 18 septiembre 2020

Título:	**Determinación de la función distribución de pares en un difractómetro de monocristal**
Autor:	Sol Fernández Muñoz
Directores:	Ana Isabel Becerro Nieto
Grado:	Trabajo Fin de Master
Centro:	Universidad de Sevilla
Fecha Defensa:	11 noviembre 2020

DOCENCIA / TEACHING

Investigadores de esta unidad participan en el Máster Interuniversitario "Láser, Plasma y Tecnología de Superficies" (página 236)

PREMIOS Y RECONOCIMIENTOS / PRIZES AND ACKNOWLEDGEMENTS

Premio al mejor póster otorgado por la revista Applied Sciences (MDPI)
Febrero 2020
Laura Calió

EQUIPAMIENTO CIENTÍFICO AVAILABLE EQUIPMENT

- Potenciostato y sistema electroquímico
- Analizador de potencial Z, tamaño de partícula y pesos moleculares (Malvern, ZS90)
- 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)
- 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).
- Sistema de medida de porosidades en capas delgadas.
- Vis-NIR FTIR espectrofotómetro Bruker GmbH Fuente de excitación continua normal y angular. Specular Reflectance Attached Microscope.
- Tunable Supercontinuum White Laser Source. Fianium LTD 4W total output 400nm – 2400nm range Acousto-Optic Tunable Filter

- Perfilómetro mecánico DektakXT en su versión automática (platina XY motorizada y giro de 360º motorizado) y que incluye soporte de muestras cerámico para muestras flexibles.
- Fluorímetro Edinburgh FLS1000 con accesorios (esfera integradora y criostato).

Unidad Externa de Investigación: Física de Materiales

External Unit: Physics of Materials

PERSONAL / PERSONNEL

Catedráticos

Dra. Clara F. Conde Amiano
Dr. Alberto Criado Vega
Dr. Luis Esquivias Fedriani
Dr. Victorino Franco García
Dr. Antonio Muñoz Bernabé
Dr. Javier S. Blázquez Gámez

Profesores Titulares

Dra. Josefa María Borrego Moro
Dra. M. del Carmen Gallardo Cruz
Dra. Ángela Gallardo López
Dr. Felipe Gutiérrez Mora
Dr. José María Martín Olalla
Dra. Ana Morales Rodríguez
Dr. Francisco Javier Romero Landa

Profesores Eméritos

Dr. Alejandro Conde Amiano

Profesores Contratados Doctores

Dr. Jhon J. Ipus Bados
Dr. Víctor Morales Flórez
Dra. Rocío del Carmen Moriche Tirado

Doctores Contratados

Dr. Luis M. Moreno Ramírez
Dra. Jia Yan Law

Personal Investigador en Formación

Gdo. Álvaro Díaz García
Gda. Cristina López Pernía
Gdo. Alejandro F. Manchón Gordón
Gda. Carmen Muñoz Ferreiro
Gdo. Pedro Rivero Antúnez
Gdo. Antonio Vidal Crespo

Investigadores Honorarios

Dr. Jaime del Cerro
Dr. Arturo Domínguez Rodríguez
Dr. Justo Jiménez

PROYECTOS DE INVESTIGACIÓN / RESEARCH PROJECTS

Modelado y Control de la Histéresis en Materiales Magnetocalóricos para Refrigeración y Conversión de Energía

Código/Code: MAT2016-77265-R
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Organismo Financiador/Financial source: Ministerio de Economía y Competitividad
Investigador responsable/Research head: Victorino Franco García, Javier Sebastián Blázquez Gámez
Componentes/Research group: Josefa María Borrego Moro, Alejandro Conde Amiano, Clara Francisca Conde Amiano, Jhon J. Ipus Bados

Abordando las Limitaciones de Materiales Magnetocalóricos para su Implementación en Aplicaciones Energéticamente Eficientes

Código/Code: PID2019-105720RB-I00
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Componentes/Research group: Josefa María Borrego Moro, Jhon J. Ipus Bados

Procesado y Caracterización de Composites Cerámicos con Nanomateriales Laminados Bidimensionales Processing and characterization of ceramic composites with two-dimensional laminar nanomaterials (CMC-NANOLAM)

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Investigador responsable/Research head: Ángela Gallardo López, Rosalía Poyato Galán
Componentes/Research group: Antonio Muñoz Bernabé, Ana Morales Rodríguez, Felipe Gutiérrez Mora

La incorporación de nanomateriales bidimensionales en materiales compuestos de matriz cerámica está aumentando en un esfuerzo para superar la inherente fragilidad de las cerámicas y para conferirles nuevas funcionalidades. Aún existen cuestiones abiertas en este campo en cuanto a resistencia mecánica, tenacidad a la fractura, cinética de crecimiento de fisuras, comportamiento tribológico, papel de fases interfaciales o idoneidad para el electromecanizado, entre otras. Aunque las nanoláminas de grafeno (NLG) se han mostrado como una segunda fase muy adecuada, los nanomateriales inorgánicos análogos al grafeno podrían extender el rango de aplicabilidad de los materiales con grafeno. El uso de nanoláminas de nitruro de boro (NLNB) en estos compuestos es muy prometedor y está prácticamente inexplorado.

Este proyecto propone un estudio sistemático de materiales compuestos con usos en aplicaciones estructurales y funcionales, con dos matrices de circona estabilizada con itria y dos tipos diferentes de nanomateriales 2D -nanoláminas de grafeno o de nitruro de boro- para profundizar en la comprensión de sus comportamientos mecánico y eléctrico. Con este fin, se fabricarán materiales con matrices de circona tetragonal dopada con 3 %mol de itria y circona cúbica dopada con itria, persiguiendo una microestructura óptima con una distribución homogénea de los nanomateriales 2D en ambas matrices cerámicas. Se investigarán en profundidad materiales con NLG para dar respuesta a cuestiones abiertas en el conocimiento de estos materiales. La distribución, tamaño e integridad estructural de las NLG se caracterizarán mediante difracción de rayos X, microscopía electrónica de barrido y espectroscopía Raman, y las intercaras entre las NLG y la matriz se caracterizarán mediante microscopía electrónica de transmisión. La resistencia mecánica, resistencia a la rotura, mecanismos de refuerzo y cinética de crecimiento de grano en estos materiales se examinará en profundidad, y se establecerá la mejor combinación de ruta de procesado y contenido de NLG en términos de refuerzo a la matriz. Se realizarán medidas de conductividad eléctrica en materiales con diferentes contenidos de NLG y se evaluará la respuesta al electromecanizado de los materiales eléctricamente conductores. Se realizarán también medidas de conductividad eléctrica en función de la temperatura para describir las posibles variaciones en el tipo de conducción al aumentar el contenido en NLG. Por otra parte, se investigarán materiales con NLNB con el objetivo de obtener una primera aproximación a la comprensión de este sistema. Con este fin, tras la síntesis de las NLNB usando una estrategia de mezcla de disolventes para la exfoliación en fase líquida de nanoláminas a partir de polvo de BN hexagonal, se prepararán polvos con diferentes contenidos de NLNB usando técnicas de procesado de polvo en medio húmedo. La caracterización microestructural de los materiales sinterizados mediante “Spark Plasma Sintering" se realizará mediante microscopía electrónica de barrido y de transmisión, difracción de rayos X y espectroscopía Raman. Se estudiarán propiedades como dureza, resistencia a la flexión y resistencia al desgaste, y se realizarán ensayos mecánicos a alta temperatura. Se analizará la conductividad eléctrica en función de la temperatura para esclarecer el efecto de la incorporación de una segunda fase aislante en las fronteras de grano sobre el comportamiento eléctrico de un conductor iónico.

Two-dimensional nanomaterials are being increasingly used as fillers in ceramic composites in an effort to overcome the inherent fragility of ceramics and to provide them with

new functionalities. There are open issues in the field of these composites regarding their strength and fracture toughness mechanisms, crack growth kinetics, tribological behavior, role of interfacial phases or suitability for electrical discharge machining, among others. Although graphene nanosheets (GNS) are excellent fillers, inorganic graphene analogues could extend the range of applicability of graphene ceramic composites. The use of boron nitride nanosheets (BNNS) as fillers in ceramic composites is promising and practically unexplored.

This proposal outlines a systematic study of composites intended for use in structural and functional applications, with two different ceramic matrices from the yttria-stabilized zirconia system incorporating two different 2D laminar nanomaterials -graphene or boron nitride nanosheets-, to deepen in the understanding of their mechanical and electrical behavior. To that end, composites with 3 mol% yttria tetragonal zirconia and 8 mol% yttria cubic zirconia matrices will be fabricated, pursuing an optimum microstructure with a homogeneous distribution of the 2D nanomaterials throughout both ceramic matrices. On the one hand, ceramic composites with graphene nanosheets will be investigated in depth to complete the gaps in the current knowledge of these materials. The distribution, size and structural integrity of the GNS will be characterized by X-ray diffraction, scanning electron microscopy and Raman spectroscopy while the interfaces between the GNS and the matrix will be characterized by transmission electron microscopy. The strength, failure resistance, reinforcement mechanisms and crack growth kinetics of these composites will be thoroughly examined, and the best combination of processing route and GNS content in terms of reinforcement will be established. Electrical conductivity measurements of composites with different GNS contents will be carried out at room temperature and the response to electrical discharge machining of the electrically conductive composites will be evaluated. Conductivity measurements will be carried out also as a function of temperature in order to describe the possible variations of conduction type when increasing the GNS content. On the other hand, ceramic composites with boron nitride nanosheets will be investigated in order to get a first approach to the understanding of this system. For this purpose, after the synthesis of the BN nanosheets using a mixed-solvent strategy for liquid exfoliation of BNNS from h-BN powder, composites with different contents of BNNS will be prepared using wet powder processing techniques. The microstructural characterization of the spark plasma sintered composites will be carried out by scanning and transmission electron microscopy, X-ray diffraction and Raman spectroscopy. Mechanical properties as hardness, flexural strength and wear resistance will be studied at room temperature, whereas deformation tests at high temperatures will be also performed. The electrical conductivity as a function of temperature will be analyzed in order to clarify the effect of incorporating an insulating second phase at the grain boundaries on the electrical performance of an ionic conductor.

MINISTERIO DE ECONOMÍA Y COMPETITIVIDAD
FEDER Fondo Europeo de Desarrollo Regional
UNIÓN EUROPEA "Una manera de hacer Europa"

Refuerzo Intragranular de Cerámicas con Fases de Baja Dimensionalidad

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Organismo Financiador/Financial source: Plan Estatal 2017-2020 Generación Conocimiento - Proyectos I+D+i

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Investigador responsable/Research head: Víctor Morales Flórez
Componentes/Research group: Luis Esquivias Fedriani, Francisco de Paula Jiménez Morales, Florentino Sánchez Bajo

Influencia de excitaciones múltiples sobre transiciones de fase termomagnéticas para aplicaciones energéticas

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Organismo Financiador/Financial source: Junta de Andalucía
Investigador responsable/Research head: Victorino Franco García
Componentes/Research group: Javier Sebastián Blázquez Gámez, Josefa María Borrego Moro, Alejandro Conde Amiano, Clara Francisca Conde Amiano, Jhon Jairo Ipus Bados

Transiciones de fase termo-magnéticas para un uso eficiente de la energía y de los recursos

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Organismo Financiador/Financial source: Junta de Andalucía
Investigador responsable/Research head: Victorino Franco García
Componentes/Research group: Javier Sebastián Blázquez Gámez, Josefa María Borrego Moro, Alejandro Conde Amiano, Clara Francisca Conde Amiano, Jhon Jairo Ipus Bados

ARTÍCULOS PUBLICADOS EN REVISTAS SCI / PAPERS IN SCI JOURNALS

Monitoring crack propagation in skin-stringer elements using carbon nanotube doped adhesive films: Influence of defects and manufacturing process

Sánchez-Romate, Xoan F.; Moriche, Rocio; Renato Pozo, Ángel; Jiménez-Suarez, Alberto; Sánchez, María; Guemes, Alfredo; Urena, Alejandro
Composites Science and Technology, **193** (2020) 108147
DOI: 10.1016/j.compscitech.2020.108147

Standard Mode-I and skin-stringer sub-elements were manufactured using novel adhesive films reinforced with carbon nanotubes. Peeling tests were conducted to analyse the different crack propagation mechanisms. In this context, the influence of manufacturing methods and artificial defects is deeply explored. It was observed that the electrical resistance increased with crack length due to a breakage of electrical pathways, depending on manufacturing and induced defects. Co-bonded specimens showed a more stable behaviour due to a better interface between the adhesive and substrate than joints manufactured by secondary bonding. Moreover, by analysing the influence of artificial defects, it was observed that larger discontinuities induced more unstable electromechanical behaviours as there is a more prevalent breakage of electrical pathways. In this regard, samples with Teflon inserts showed sharper increases of electrical resistance than those previously treated with a liquid agent simulating a kissing bond. Therefore, the proposed technique shows a high potential and applicability for Structural Health Monitoring (SHM) of integrated composite structures.

Regulation of phase transition and magnetocaloric effect by ferroelectric domains in FeRh/PMN-PT heterojunctions

Qiao, Kaiming; Wang, Jianlin; Hu, Fengxia; Li, Jia; Zhang, Cheng; Liu, Yao; Yu, Zibing; Gao, Yihong; Su, Jian; Shen, Feiran; Zhou, Houbo; Bai, Xuedong; Wang, Jing; Franco, Victorino; Sun, Jirong; Shen, Baogen
Acta Materialia, **191** (2020) 51-59
DOI: 10.1016/j.actamat.2020.03.028

The narrow temperature span, DTspan, of first-order magnetocaloric materials is a serious problem that limits the application as refrigerants. Here we report tunable phase transition and magnetocaloric effect controlled by ferroelectric (FE) domains in FeRh films grown on (001)- and (011)-cut PMN-PT substrates. Adjacent twostep phase transition, and hence significantly broadened Delta T-span, has been achieved in FeRh films by utilizing the multi-domain structure of PMN-PT substrates. The results of aberration corrected (ac)-STEM, EELS and EDX analysis revealed that a 3 similar to 4 nm buffer layer with AB_2O_4-type spinel structure is naturally formed at the interface, which largely reduces the lattice mismatch between FeRh and PMN-PT and plays a key role for the successful growth of epitaxial (oriented) FeRh film on either (001)- or (011)-oriented PMN-PT. The switched FE domains by electric field govern the phase transition of FeRh films. As a result, regulated entropy change and refrigeration capacity in a wide temperature span have been achieved. On this basis, a feasible magnetic refrigeration cycle facilitated by electric field is designed. The present study provides an experimental basis for expanding the refrigeration temperature span by ferroelectric domain engineering, which is

significant for promoting refrigeration application of first-order magnetocaloric materials particularly in micro-devices.

Tunable structure and connectivity of organosilica hybrid films by using different epoxy based precursors in atmospheric plasma deposition

Moriche, R.; Ding, Y.; Dong, S.; Zhao, O.; Dauskardt, R. H.
Applied Surface Science, **508** (2020) 145233
DOI: 10.1016/j.apsusc.2019.145233

Organosilica hybrid films and fluorinated organosilica hybrid films have been successfully deposited using atmospheric plasma deposition. Different precursors, which incorporates epoxy groups in their molecules, were used to obtain connected structures with tunable chemistry and connectivity. The study demonstrated the capability of tuning the chemical composition, connectivity and mechanical properties of organosilica films by selecting the precursors and operational parameters of the process. The use of (3-glycidiloxypropyl) trimethoxysilane (GPTMS) and 2-(3,4-epoxy-cyclohexylethyl) trimethoxysilane (TRIMO) as precursors made possible the deposition of hybrid films with high organic character, increasing the relative C content up to 20 at% and 40 at%, respectively, due to the lower fragmentation caused during the deposition by atmospheric plasma. The obtained structures had higher concentration of symmetric cages than those obtained from precursors without epoxy groups, which lead to a more compact structure and higher stiffness than conventional organic films. The combination with a fluorinated molecule also had a strong influence in the resultant structure caused by the preferential orientation of fluorinated molecules due to the low surface energy of FDTS that causes migration of fluorinated tails to the film-air interface during the film growth.

Electrochemically Exfoliated Graphene-Like Nanosheets for Use in Ceramic Nanocomposites

Poyato, R; Verdugo, R; Munoz-Ferreiro, C; Gallardo-López, A
Materials, **13** (2020) 11
Junio, 2020 | **DOI:** 10.3390/ma13112656

In this work, the synthesis of graphene-like nanosheets (GNS) by an electrochemical exfoliation method, their microstructural characterization and their performance as fillers in a ceramic matrix composite have been assessed. To fabricate the composites, 3 mol % yttria tetragonal zirconia (3YTZP) powders with 1 vol % GNS were processed by planetary ball milling in tert-butanol to enhance the GNS distribution throughout the matrix, and densified by spark plasma sintering (SPS). According to a thorough Raman analysis and SEM observations, the electrochemically exfoliated GNS possessed less than 10 graphene layers and a lateral size lower than 1 mu m. However, they contained amorphous carbon and vacancy-like defects. In contrast the GNS in the sintered composite exhibited enhanced quality with a lower number of defects, and they were wavy, semi-transparent and with very low thickness. The obtained nanocomposite was fully dense with a homogeneous distribution of GNS into the matrix. The Vickers hardness of the nanocomposite showed similar values to those of a monolithic 3YTZP ceramic sintered in the same conditions, and to the reported ones for a 3YTZP composite with the same content of commercial graphene nanosheets.

Milling effects on the distribution of Curie temperatures and magnetic properties of Ni-doped $La_{0.7}Ca_{0.3}MnO_3$ compounds

Manchon-Gordon, A. F.; Ipus, J. J.; Kowalczyk, M.; Wojcik, A.; Blazquez, J. S.; Conde, C. F.; Maziarz, W.; Svec, P., Sr.; Kulik, T.; Conde, A.
Journal of Alloys and Compounds, **848** (2020) 156566
DOI: 10.1016/j.jallcom.2020.156566

A systematic study of the magnetic and magnetocaloric properties around the ferromagnetic-paramagnetic phase transition temperature has been carried out on $La_{0.2}Ca_{0.3}Mn_{1-x}Ni_xO_3$ (x = 0, 0.02, 0.07, 0.10) compounds synthesized by ball milling from oxides and pure Ni. The order of the transition, analyzed by the Banerjee's criterion and the field dependence of the magnetic entropy change, was found to be of second order for all the studied compositions. The existence of small traces of β-MnO_2 phase and the distortions produced during the milling process may induce a competition character of the ferromagnetic and the antiferromagnetic interactions at low fields and motivate the existence of a distribution of Curie temperatures. Once the antiferromagnetic contributions are avoided, the parameters of the distribution, the average Curie temperature $\overline{T}_C$ and the broadening of the distribution ΔT_C, have been obtained from the analysis of the approach to saturation curves and the magnetocaloric effect.

Study of the kinetics and products of the devitrification process of mechanically amorphized $Fe_{70}Zr_{30}$ alloy

Manchon-Gordon, A. F.; Ipus, J. J.; Blazquez, J. S.; Conde, C. F.; Conde, A.; Svec, P., Sr.
Journal of Alloys and Compounds, **825** (2020) 154021
DOI: 10.1016/j.jallcom.2020.154021

Devitrification of mechanically alloyed amorphous $Fe_{70}Zr_{30}$ at. % compound consists on a two-step process: amorphous -> amorphous + bcc Fe + Fe_2Zr -> Fe_2Zr + $Fe_{23}Zr_6$. This sequence is inferred from the evolution of the Mössbauer spectra, the thermomagnetic experiments and the X-ray diffraction (XRD) patterns. Hyperfine parameters for both intermetallics have been obtained from Mössbauer spectroscopy in correlation with the phase identification from XRD results. The broadening of the stable compositional range of Fe_2Zr intermetallic above 1000 K is responsible for a strong dependence of the phase fractions on heating and cooling rates. Despite the overlapping of the two processes involved in the devitrification, the individual Avrami exponents of each one have been estimated.

Effect of pressure on the phase stability and magnetostructural transitions in nickel-rich NiFeGa ribbons

Manchon-Gordon, A. F.; Ipus, J. J.; Kowalczyk, M.; Wojcik, A.; Blazquez, J. S.; Conde, C. F.; Maziarz, W.; Svec, P., Sr.; Kulik, T.; Conde, A.
Journal of Alloys and Compounds, **844** (2020) 156092
DOI: 10.1016/j.jallcom.2020.156092

Ribbons of a $Ni_{55}Fe_{19}Ga_{26}$ Heusler alloy were prepared by melt spinning technique. The effect of pressure on magnetostructural transitions was studied comparing as-spun ribbons to ribbons previously sub mitted to an axial pressure and to powder obtained from manually grinded as-spun ribbons. The martensitic transformation present in the as-spun ribbon almost vanishes after pulverization. The structural change driven by mechanical treatment is a stress-induced intermartensitic transformation from a 14 M modulated to a non-modulated tetragonal (NM) structure. There is a progressive change from the structure of as-spun samples to that of the pulverized one in samples submitted to axial pressures as it increases.

Mechanical characterization of sol-gel alumina-based ceramics with intragranular reinforcement of multiwalled carbon nanotubes

Rivero-Antunez, Pedro; Cano-Crespo, Rafael; Esquivias, Luis; de la Rosa-Fox, Nicolas; Zamora-Ledezma, Camilo; Domínguez-Rodríguez, Arturo; Morales-Flórez, Víctor
Ceramics International, **46** (2020) 19723-19730
DOI: 10.1016/j.ceramint.2020.04.285

Multiwalled carbon nanotubes (MWCNTs) have been widely considered for mechanical reinforcement of ceramic matrix composites. Nevertheless, the efficiency of this reinforcement strategy is under debate due to fabrication issues, such as a good homogenization or the location of the MWCNTs inside the matrix composite. Regarding this, the intragranular location of the MWCNTs has been deemed a crucial feature for optimizing the reinforcement compared to the typical intergranular placement achieved by conventional procedures. Recently, the sol-gel method has been reconsidered, as it promotes the intragranular placement of the MWCNTs. This work presents the mechanical characterization of these composites synthesized by the sol-gel method, where crack-bridging has been revealed as toughening mechanism. Finally, the conventional use of the bibliographical Young's modulus of pure alumina for the estimation of the fracture toughness is discussed, obtaining significant improvements of the fracture toughness when indentation measurements are treated by considering elastic moduli obtained by nanoindentation.

Influence of Manufacturing Parameters and Post Processing on the Electrical Conductivity of Extrusion-Based 3D Printed Nanocomposite Parts

Paz, Ruben; Moriche, Rocio; Monzon, Mario; García, Joshua
Polymers, **12** (2020) art. 733
DOI: 10.3390/polym12040733

The influence of manufacturing parameters of filament extrusion and extrusion-based Additive Manufacturing (AM), as well as different post processing techniques, on the electrical conductivity of 3D printed parts of graphene nanoplatelets (GNP)-reinforced acrylonitrile butadiene styrene (ABS) has been analyzed. The key role of the manufacturing parameters to obtain electrically conductive filaments and 3D printed parts has been demonstrated. Results have shown that an increase in extrusion speed, as well as lower land lengths, induces higher extrudate swelling, with the consequent reduction of the electrical conductivity. Additionally, filaments with lower diameter values, which result in a higher surface-to-cross-section ratio, have considerably lower electrical conductivities. These factors tune the values of the volume

and surface electrical conductivity between 10^{-4}-10^{0} S/m and 10^{-8}-10^{-3} S/sq, respectively. The volume and surface electrical conductivity considerably diminished after 3D printing. They increased when using higher printing layer thickness and width and were ranging between 10^{-7}-10^{-4} S/m and 10^{-8}-10^{-5} S/sq, respectively. This is attributed to the higher cross section area of the individual printed lines. The effect of different post processing (acetone vapor polishing, plasma and neosanding, which is a novel finishing process) on 3D printed parts in morphology and surface electrical conductivity was also analyzed.

Influence of Milling Time on the Homogeneity and Magnetism of a $Fe_{70}Zr_{30}$ Partially Amorphous Alloy: Distribution of Curie Temperatures

Manchón-Gordón, A.F.; Ipus, J.J.; Blázquez, J.S.; Conde, C.F.; Conde, A.
Materials, **13** (2020) 490
DOI: 10.3390/ma13020490

In this work, the mechanically alloyed $Fe_{70}Zr_{30}$ (at. %) composition has been used to study the influence of milling time on its homogeneity and magnetic properties. The microstructure and Fe environment results show the formation of an almost fully amorphous alloy after 50 h of milling in a mixture of pure 70 at. % Fe and 30 at. % Zr. The soft magnetic behavior of the samples enhances with the increase of the milling time, which is ascribed to the averaging out of the magnetocrystalline anisotropy as the crystal size decreases and the amorphous fraction increases. The formation of a non-perfectly homogenous system leads to a certain compositional heterogeneity, motivating the existence of a distribution of Curie temperatures. The parameters of the distribution (the average Curie temperature, $\overline{T}_C$, and the broadening of the distribution, ΔT_C) have been obtained using a recently reported procedure, based on the analysis of the approach towards the saturation curves and the magnetocaloric effect. The decrease of ΔT_C and the increase of $\overline{T}_C$ with the milling time are in agreement with the microstructural results. As the remaining α-Fe phase decreases, the amorphous matrix is enriched in Fe atoms, enhancing its magnetic response.

Sensitive response of GNP/epoxy coatings as strain sensors: analysis of tensile-compressive and reversible cyclic behaviour

Sánchez-Romate, Xoan F.; Moriche, R.; Jiménez-Suarez, A.; Sánchez, M.; Prolongo, S. G.; Urena, A.
Smart Materials and Structures, **29** (2020) 065012
DOI: 10.1088/1361-665X/ab8316

The electromechanical performance and reversibility of sensitive GNP/epoxy strain sensors were experimentally and theoretically analyzed. Under tensile loads, the strain sensors showed lower sensitivity and more linearity than bulk sensors, behavior attributed to a slight preferential orientation of the GNPs along the in-plane direction. The Gauge Factor (GF) obtained was 9.1 ± 0.9 and 11 ± 1 for strain values up to 0.005 mm mm^{-1} and above 0.015 mm mm^{-1}, respectively. In contrast, the electromechanical response when subjected to compressive strain is more complex and three different regions are distinguished: (I) diminution, (II) stabilization and (III) increase of the normalized electrical resistance. Here, GF under compressive loads was negative at low strain values (region I), being -13 ± 2, and positive at high strain (region III), with a value

of 8 ± 1. Theoretical analysis revealed that at low strain, there is prevalence of in-plane tunneling mechanisms whereas at higher strain, the out of plane mechanisms dominate, explaining the apparently anomalous behavior at compressive loads. Additionally, strain sensors showed high reversibility with cyclic load in the electromechanical response, but under compressive forces, the loading-unloading electrical resistance curve was asymmetric due to the opening and closing of microcavities and defects in the vicinities of the GNPs.

Environmental Impact of Phosphogypsum-Derived Building Materials

Romero-Hermida, M. I.; Flores-Ales, V.; Hurtado-Bermudez, S. J.; Santos, A.; Esquivias, L.
International Journal of Environmental Research and Public Health, **17** (2020) 4248
DOI: 10.3390/ijerph17124248

The aim of the present work was to characterize the products obtained from the treatment of phosphogypsum residue by means of two recovery routes, and also to evaluate the concentrations of heavy metals and radionuclides in the materials obtained and their leachates. In this way, it is possible to determine how the most hazardous components of phosphogypsum behave during procedures until their stabilization through CO_2 fixation. This study provides an initial estimate of the possibilities of reusing the resulting products from a health and safety risk standpoint and their potential polluting capacity. The phases resulting from the transformations were controlled, and the behaviour of standard mortars manufactured from the resulting paste lime was studied. In all cases, an additional control of the leachate products was performed.

Structure, magnetic and magnetocaloric properties of Ni_2MnGa Heusler alloy nanowires

Zhang, Y. C.; Qin, F. X.; Estevez, D.; Franco, V; Peng, H. X.
Journal of Magnetism and Magnetic Materials, **513** (2020) 167100
DOI: 10.1016/j.jmmm.2020.167100

Heusler alloys are favorable candidates for fabricating functional devices and sensors due to their characteristic structural and magnetic properties, which vary at different length scales. In this paper, for the sake of expanding the fields of application and systematically studying the mechanisms at the nanoscale, Ni_2MnGa Heusler alloy nanowires were fabricated via the electrospinning method followed by optimized heat treatments. While the nanowires exhibited ferromagnetic-paramagnetic transition near room temperature, anomalies in magnetic behaviors were observed by power fitting of χ^{-1}-T curves disobeying Curie-Weiss law. The Ni_2MnGa nanowires exhibited maximum isothermal entropy change of 0.35 J/kgK accompanied by extremely wide working temperature region (150 K), with a peak temperature (305 K) suitable for room temperature applications. Arrott plots and quantitative analysis of field/temperature dependence of magnetic entropy change determined second order magnetic phase transition in the nanowires. Construction of phenomenological universal curves addressed demagnetizing effect on magnetocaloric response, which was compared to the multiphase composition obtained by structural analysis. These novel findings in Ni-Mn-Ga nanowires enrich our knowledge of structure and magnetism of Heusler alloys at the nanoscale and could be taken as reference facilitating future relevant research.

Gel combustion synthesis and magnetic properties of $CoFe_2O_4$, $ZnFe_2O_4$, and $MgFe_2O_4$ using 6-aminohexanoic acid as a new fuel

Chavarriaga, EA; Lopera, AA; Franco, V; Bergmann, CP; Alarcon, J
Journal of Magnetism and Magnetic Materials, **497** (2020) 166054
DOI: 10.1016/j.jmmm.2019.166054

For the first time, 6-aminohexanoic acid is used as an alternative fuel in the synthesis of the spinel ferrites with compositions $CoFe_2O_4$, $ZnFe_2O_4$ and $MgFe_2O_4$ using gel combustion synthesis with different oxidizer-to-fuel (O/F) ratios. The gel precursors were studied by differential thermal analysis and thermogravimetry (DTA/TG), which showed that the ignition temperature depends on the gel precursor, being around 230 °C, 130 °C and 275 °C for $CoFe_2O_4$, $ZnFe_2O_4$, and $MgFe_2O_4$, respectively. These results showed than the 6-aminohexanoic acid has an ignition temperature lower than the urea and the citric acid when were used in the synthesis of the spinel ferrites by gel combustion. Moreover, the adiabatic flame temperature (T_{ad}) of the reactions of combustion were calculated using thermodynamic analysis, which showed that T_{ad} increases when the mass of the 6-aminohexanoic acid increases. X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR) techniques confirmed the formation of the spinel structures for all the O/F ratios, but there is a minor impurity phase for some experiments. The particle morphology was evaluated using scanning electron microscopy (SEM), showing aggregated particles with a sponge-like structure due to the released gases during the combustion. A vibrating sample magnetometer (VSM) was used for measuring the magnetic properties of the as-prepared powders. The values of saturation magnetization, M_s, and coercitivity, H_c, were M_s = 66.9 emu/g and H_c = 1505 Oe for $CoFe_2O_4$, M_s = 44.6 emu/g and H_c = 75.2 Oe for $ZnFe_2O_4$ and M_s = 28.66 emu/g and H_c = 111.4 Oe for $MgFe_2O_4$. The optical band gap for $CoFe_2O_4$, $ZnFe_2O_4$, and $MgFe_2O_4$ were 1.11 eV, 1.37 eV and 1.64 eV, respectively. These results show that 6-aminohexanoic acid can be used as an alternative fuel in the synthesis of spinel ferrites using gel combustion synthesis.

Obtaining magnetocaloric MnCo(Fe)Ge intermetallics from low temperature treatment of mechanically alloyed precursors

Vidal-Crespo, A.; Ipus, J. J.; Blazquez, J. S.; Conde, A.
Journal of Magnetism and Magnetic Materials, **514** (2020) 167127
DOI: 10.1016/j.jmmm.2020.167127

Production of the intermetallic $MnCo_{0.8}Fe_{0.2}Ge$ phase, interesting as magnetocaloric material, is obtained by using an almost fully amorphous mechanically alloyed precursor. Thermal treatment to develop the intermetallic phase has been reduced in time (from several hours or even days to few minutes), and temperature (from 1123 K to 723 K). Thermomagnetic measurements allow us to analyze the magnetocaloric effect and to compare the results with those obtained from conventional annealing treatments. Close relationship between magnetocaloric properties and crystal size provides further optimization of the system preserving the advantages of the production method proposed.

Magnetocaloric response of binary Gd-Pd and ternary Gd-(Mn,Pd) alloys

Gebara, Piotr; Díaz-García, Alvaro; Yan Law, Jia; Franco, Victorino
Journal of Magnetism and Magnetic Materials, **500** (2020) 166175
DOI: 10.1016/j.jmmm.2019.166175

This work investigates the MCE of alloying Pd or (Mn,Pd) to Gd, which yields the formation of an extra Gd_7Pd_3 or $Gd_7(Mn,Pd)_3$ phase in addition to the Gd phase, forming a composite. The phase coexistence is observed from XRD and SEM/EDX results, whereby the phase fraction of secondary phase increases with the dopant content. The magnetocaloric behavior of the binary samples present two characteristic ΔS_M peaks, attributed to the Curie transitions of the coexisting biphases (separated by ΔT_C = 45 K). Two minima are observed from the exponent n of field dependence of ΔS_M, reinforcing the presence of the Curie transitions of the two phases. The largest phase proportion of Gd_7Pd_3 observed in $Gd_{80}Pd_{20}$ sample gives rise to the largest RC value, which is also 10% increase compared to single phase Gd.

Devitrification of Mechanically Alloyed Fe-Nb System: Mössbauer Study of the Intermetallic Phases

Manchon-Gordon, A. F.; Svec, P.; Ipus, J. J.; Kowalczyk, M.; Blazquez, J. S.; Conde, C. F.; Conde, A.; Svec, P.; Kulik, T.
Metallurgical and Materials Transactions A-Physical Metallurgy and Materials Science, **51** (2020) 1395-1401
DOI: 10.1007/s11661-019-05610-5

Intermetallic phases in the Fe-Nb system have been obtained as products of the devitrification of a homogeneous amorphous $Fe_{70}Nb_{30}$ alloy prepared by mechanical alloying. Besides Fe_2Nb Laves and Fe_7Nb_6 intermetallic phases, α-Fe phase has been detected by X-ray diffraction (XRD). Hyperfine parameters for both Fe_2Nb and Fe_7Nb_6 intermetallics have been obtained from Mössbauer spectroscopy in correlation with phase identification from XRD results. Thermomagnetic measurements show changes in the Curie temperatures of the amorphous and Fe_2Nb phases during the crystallization, due to compositional variations related to the developing of the α-Fe phase. Kinetics of the crystallization process has been analyzed using the classical Johnson-Mehl-Avrami-Kolmogorov kinetic theory in both isothermal and non-isothermal regimes.

Phase Deconvolution of Multiphasic Materials by the Universal Scaling of the Magnetocaloric Effect

Díaz-García, Alvaro; Law, Jia Yan; Gebara, Piotr; Franco, Victorino
JOM, **72** (2020) 2845-2852
DOI: 10.1007/s11837-020-04251-z

Multiphase magnetocaloric materials result from design, where different phases are combined in a composite, or residual phases in the production of the material. The universal scaling of the magnetocaloric effect, commonly used for the study of single-phase second-order phase transitions, shows distortions when applied to multiphase materials with phases that undergo second-order phase transitions. In this work, universal scaling has been applied to a composite

with coexistent Gd and Gd_7Pd_3phases with comparable magnetocaloric responses and Curie temperatures separated by similar to 45 K. The biphasic nature of the sample leads to distortions in universal scaling that have been exploited to deconvolute the magnetocaloric response of each phase. The phase ratio has been obtained through the deconvoluted magnetocaloric responses, and the results are comparable to those from x-ray diffraction refinement. This procedure allows the prediction of the response of a desired pure phase even in the presence of residual magnetic contributions.

Setting the Basis for the Interpretation of Temperature First Order Reversal Curve (TFORC) Distributions of Magnetocaloric Materials

Moreno-Ramírez, Luis M.; Franco, Victorino
Metals, **10** (2020) 10
DOI: 10.3390/met10081039

First Order Reversal Curve (FORC) distributions of magnetic materials are a well-known tool to extract information about hysteresis sources and magnetic interactions, or to fingerprint them. Recently, a temperature variant of this analysis technique (Temperature-FORC, TFORC) has been used for the analysis of the thermal hysteresis associated with first-order magnetocaloric materials. However, the theory supporting the interpretation of the diagrams is still lacking, limiting TFORC to a fingerprinting technique so far. This work is a first approach to correlate the modeling of first-order phase transitions, using the Bean-Rodbell model combined with a phenomenological transformation mechanism, with the features observed in experimental TFORC distributions of magnetocaloric materials. The different characteristics of the transformations, e.g., transition temperatures, symmetry, temperature range, etc., are correlated to distinct features of the distributions. We show a catalogue of characteristic TFORC distributions for magnetocaloric materials that exhibit some of the features observed experimentally.

Distribution of Transition Temperatures in Magnetic Transformations: Sources, Effects and Procedures to Extract Information from Experimental Data

Manchon-Gordon, Alejandro F.; López-Martín, Raúl; Vidal-Crespo, Antonio; Ipus, Jhon J.; Blazquez, Javier S.; Conde, Clara F.; Conde, A.
Metals, **10** (2020) 10
DOI: 10.3390/met10081039

First Order Reversal Curve (FORC) distributions of magnetic materials are a well-known tool to extract information about hysteresis sources and magnetic interactions, or to fingerprint them. Recently, a temperature variant of this analysis technique (Temperature-FORC, TFORC) has been used for the analysis of the thermal hysteresis associated with first-order magnetocaloric materials. However, the theory supporting the interpretation of the diagrams is still lacking, limiting TFORC to a fingerprinting technique so far. This work is a first approach to correlate the modeling of first-order phase transitions, using the Bean-Rodbell model combined with a phenomenological transformation mechanism, with the features observed in experimental TFORC distributions of magnetocaloric materials. The different characteristics of the transformations, e.g., transition temperatures, symmetry, temperature range, etc., are

correlated to distinct features of the distributions. We show a catalogue of characteristic TFORC distributions for magnetocaloric materials that exhibit some of the features observed experimentally.

Temperature-FORC analysis of a magnetocaloric Heusler alloy using a unified driving force approach (T*FORC)
Franco, Victorino
Journal of Applied Physics, **127** (2020) 13
DOI: 10.1063/5.0005076

Temperature-first order reversal curve (FORC) distributions of thermomagnetic phase transitions are a fingerprinting tool to identify features of the phase transformations of the material. However, they have two major limitations: qualitative character, due to the shift of the loops with increasing driving forces, and long experimental time. The use of an effective temperature that takes into account the different driving forces affecting the transformation allows for a more quantitative comparison of the features of the FORC distributions, as it eliminates the need for an ad hoc selection of the origin of the distribution axes. At the same time, experimental measurements as a function of this effective temperature are significantly faster than purely temperature loops, hinting at a future possibility of time and cost efficient FORC characterization of temperature dependent transitions.

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

Flexure strength and fracture propagation in zirconia ceramic composites with exfoliated graphene nanoplatelets
Á. Gallardo-López, J. Castillo-Seoane, C. Muñoz-Ferreiro, C. López-Pernía, A. Morales-Rodríguez, R. Poyato
Ceramics, 3 (2020) 78-91
Marzo, 2020 **DOI:** 10.3390/ceramics3010009

FORMACION / TRAINING

FORMACIÓN DE GRADUADOS / MASTER DEGREE THESIS

Título:	**Simulación de las propiedades estructurales y magnéticas de sistemas de alta entropía**
Autor:	Javier Olivares Herrador
Directores:	Javier Blázquez Gámez
Grado:	Trabajo Fin de Grado

Centro: Física de la Materia Condensada. Universidad de Sevilla
Fecha Defensa: 3 julio 2020

Título: **Prismas de reflexión total: funcionamiento, tipos y aplicaciones**
Autor: Lucía del Castillo Cantos
Directores: Francisco Javier Romero Landa
Grado: Trabajo Fin de Grado
Centro: Física de la Materia Condensada. Universidad de Sevilla
Fecha Defensa: 6 julio 2020

Título: **Ferrofluidos para aplicaciones térmicas**
Autor: Jesús Castillo Pareja
Directores: Victorino Franco García, Luis Miguel Moreno Ramírez
Grado: Trabajo Fin de Grado
Centro: Física de la Materia Condensada. Universidad de Sevilla
Fecha Defensa: 10 julio 2020

Título: **Estudio, caracterización y fabricación de materiales conductores y flexibles para dispositivos wearables e implantables**
Autor: Irene García Macías
Directores: Gloria Huertas Sánchez, Rocío Moriche Tirado
Grado: Trabajo Fin de Grado
Centro: Electrónica y Electromagnetismo. Universidad de Sevilla
Fecha Defensa: 10 julio 2020

Título: **Estudio estructural de materiales nanoestructurados mediante SAXS**
Autor: Laura Garrido Regife
Directores: Víctor Morales Flórez
Grado: Trabajo Fin de Grado
Centro: Física de la Materia Condensada. Universidad de Sevilla
Fecha Defensa: 10 julio 2020

Título: **Fenómenos de acoplamiento fuerte colectivo entre semiconductores de tipo perovskita y modos de cavidad óptica**
Autor: Hilario Espinós Martínez
Directores: Felipe Gutiérrez Mora, Hernán Ruy Míguez García
Grado: Trabajo Fin de Grado
Centro: Física de la Materia Condensada. Universidad de Sevilla
Fecha Defensa: 10 julio 2020

Título: **Caracterización fotofísica de nanofósforos basados en cationes de tierras raras para recubrimientos de LEDs**
Autor: Fernando Escobar García
Directores: Felipe Gutiérrez Mora, Gabriel Sebastián Lozano Barbero
Grado: Trabajo Fin de Grado
Centro: Física de la Materia Condensada. Universidad de Sevilla

Fecha Defensa: 10 julio 2020

Título: **Estudio de las propiedades mecánicas de compuestos de matriz cerámica con alótropos de carbono mediante simulación por ordenador**
Autor: Miguel Ángel Balmaseda Márquez
Directores: Víctor Morales Flórez, Francisco de Paula Jiménez Morales
Grado: Trabajo Fin de Grado
Centro: Física de la Materia Condensada. Universidad de Sevilla
Fecha Defensa: 10 julio 2020

Título: **Diseño de un proceso de síntesis de andamios microporosos de alúmina para ingeniería tisular**
Autor: Noelia Verdu Molina
Directores: Víctor Morales Flórez, Rafael Cano Crespo
Grado: Trabajo Fin de Grado
Centro: Física de la Materia Condensada. Universidad de Sevilla
Fecha Defensa: 17 septiembre 2020

Título: **Simulación de células solares**
Autor: Estefanía Chacón Chacón
Directores: Felipe Gutiérrez Mora, José María Delgado Sánchez
Grado: Trabajo Fin de Grado
Centro: Física de la Materia Condensada. Universidad de Sevilla
Fecha Defensa: 17 septiembre 2020

Título: **Efecto del desorden óptico en la fuerza de Casimir**
Autor: Yassine Lamouaraa Sedlackova
Directores: Felipe Gutiérrez Mora, Victoria Esteso Carrizo
Grado: Trabajo Fin de Grado
Centro: Física de la Materia Condensada. Universidad de Sevilla
Fecha Defensa: 18 septiembre 2020

Título: **Caracterización de compuestos de matriz cerámica y óxido de grafeno reducido**
Autor: Paloma Luna Garrido
Directores: Rosalía Poyato Galán, Ana Morales Rodríguez
Grado: Trabajo Fin de Máster
Fecha Defensa: 9 julio 2020

Título: **Caracterización de compuestos de matriz cerámica y óxido de grafeno reducido**
Autor: Paloma Luna Garrido
Directores: Rosalía Poyato Galán, Ana Morales Rodríguez
Grado: Trabajo Fin de Máster
Fecha Defensa: 9 julio 2020

Título:	**Estudio de la cinética de las transformaciones magnetoestructurales en aleaciones Heusler**
Autor:	Raúl López Martín
Directores:	Javier S. Blázquez Gámez, Jhon J. Ipus Bados
Grado:	Trabajo Fin de Máster

Título:	**Diseño de andamios de alúmina para ingeniería tisular ósea**
Autor:	Manuela González
Directores:	Víctor Morales Flórez, Diego Gómez García
Grado:	Trabajo Fin de Máster

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

Investigadores de esta unidad participan en el Máster en Ciencia y Tecnología de Nuevos Materiales (página 235)

Investigadores de esta unidad, imparten docencia en siete titulaciones de Grado, cuatro titulaciones de Doble Grado y dos titulaciones de Máster de la Universidad de Sevilla. La docencia se desarrolla en diversos centros: Facultad de Física, Facultad de Biología, Facultad de Química, Facultad de Farmacia, Escuela Técnica Superior de Ingeniería Informática y Centro Internacional de Posgrado
(http://www.departamento.us.es/dmatcon/docencia.html)

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/cm^2 y campo eléctrico hasta 800V/cm
- Calorímetro de conducción, rango 80-400K, campo eléctrico 2000V/cm
- Medida de constante dieléctrica, en los mismos calorímetros,
- Medida de ciclo de histéresis en Ferroeléctricos.
- Analizador de Impedancia
- Adelgazador iónico Gatan. Model 691. Precision ion polishing system.
- Autoclave SanoClav
- Balanzas de precisión: GR.-200; AND EK-300i.
- Centrifugadora Rotofix 32A
- 4 Cortadoras Isomet Low Speed Saw (Buehler)
- Cortadora mediana Isomet 1000. Precision saw.
- Cortadora Isomet 4000. Linear Precision Saw. Buehler.
- Embutidora Pneumet II. Mounting Press. Buehler/Metaserv.
- Embutidora Simplimet 1000. Automatic Mounting Press. Buehler
- Estufa
- 2 Discoplan TS Struers
- 2 Dimple Grinder. Model 656. Gatan.
- Durómetro Wilson VH1150
- Durómetro Duramin Struers
- Hornillo/agitador magnético SBS, Heat-stir “Stuart” , CB302
- Horno de tubo de argón. Termolab, Hornos eléctricos TH1700
- Horno de aire Lenton Furnaces
- Horno mufla
- Limpiadores ultrasonidos Bandelin, Sonorex Digitec; Branson 3510
- Máquina de ensayos de deformación Zwick / Roell Zmart. Pro 1185
- Máquina de ensayos de deformación Instron 5982
- Máquina de fluencia con flujo de gases.
- Máquina de fluencia estanca de argón.
- Microscopio Óptico Confocal Leica DCM3D
- Microscopio Óptico Leica DMRD / Leica DMRE / Leica DFC420
- Molino de agitación PM100 Retsch
- Molino de bolas Molino mezclador MM200, Retsch
- Multímetro Keithley 2000 multimeter.
- Plasma etching Emitech k1050X
- Pulidoras Buehler. Beta. Grinder-Polisher yMotopol 8. Buehler Metaserv
- Prensa Manual Astur Sinter
- Prensa Fluxana, Vaneox, Pressing Technology
- 2 Refrigeradoras. (P. Castro Ibérica)
- Registro Tesatronic (Tesa TT60)
- Registro gráfico (Servogor 122 DC/kipp & Zonen)
- Reómetro Reactor Controller Controller 4836
- Rotavapor R-100. Buehi
- 4 Sondas de Ultrasonidos
- Tribómetro Microtest MT/30/SCM/T
- 2 Ultrasonic Disc Cutter Model 601 Gatan.

Servicios Generales
General Services

AYUDAS PARA LA ADQUISICIÓN DE EQUIPOS

Adquisición de un equipo de medida de Espectroscopía Ultrarrápida de estados transitorios para el Servicio de Espectroscopía del ICMS (EQC2018-004413-P)
Financia: Ministerio de Ciencia e Innovación y Consejo Superior de Investigaciones Científicas
Importe Concedido: 441.843,34 €
Periodo: 1-1-2018 / 31-03-2021
Cofinanciado por el Grupo de Investigación "Materiales Ópticos Multifuncionales"

Renovación y Mejora del Servicio de Análisis Térmico del ICMS (EQC2018-004201-P)
Financia: Ministerio de Ciencia e Innovación y Consejo Superior de Investigaciones Científicas
Importe Concedido: 113.024,17 €
Periodo: 1-1-2018 / 31-12-2019
Cofinanciado por el Instituto de Ciencia de Materiales de Sevilla

Microscopio óptico interferométrico 3D (EQC2018-004363-P)
Financia: Ministerio de Ciencia e Innovación y Consejo Superior de Investigaciones Científicas
Importe Concedido: 155.040,00 €
Periodo: 1-1-2018 / 31-12-2020
Cofinanciado por los Grupos de Investigación "Tribología y Protección de Superficies", "Nanotecnología en Superficies y Plasma" y "Reactividad de Sólidos"

Actualización Analizador del Servicio de Análisis de Superficies del ICMS (EQC2019-005712-P)
Financia: Ministerio de Ciencia e Innovación y Consejo Superior de Investigaciones Científicas
Importe Concedido: 173.653,02 €
Periodo: 1-1-2019 / 31-12-2021
Cofinanciado por el Instituto de Ciencia de Materiales de Sevilla

Adquisición de un equipo de difracción de rayos X multicomponente (monocromador Ka1, microdifracción y textura) para el servicio general de difracción de rayos X (IE17-5351)
Financia: Junta de Andalucía
Importe Concedido: 270.000,00 €
Periodo: 1-1-2019 / 31-12-2021
Cofinanciado por el Instituto de Ciencia de Materiales de Sevilla

Adquisición e Instalación de un calorímetro de altas prestaciones (IE17-5798)
Financia: Junta de Andalucía
Importe Concedido: 202.300,00 €
Periodo: 1-1-2019 / 31-12-2021
Cofinanciado por el Instituto de Ciencia de Materiales de Sevilla

Equipo de Nanoindentación para la evaluación de propiedades mecánicas en la nanoescala (IE17-5311)
Financia: Junta de Andalucía
Importe Concedido: 200.000,00 €
Periodo: 1-1-2019 / 31-12-2021
Cofinanciado por el Instituto de Ciencia de Materiales de Sevilla

Adquisición e instalación de un equipo de medida de espectrometría, eficiencia cuántica y distribución angular de electroluminiscencia, para el servicio de espectroscopias del ICMS (IE19_221)
Financia: Junta de Andalucía
Importe Concedido 120.020,00 €
Periodo: 29-12-2020 / 28-12-2022
Cofinanciado por el Instituto de Ciencia de Materiales de Sevilla

SERVICIO DE ESPECTROSCOPIAS / SPECTROSCOPY SERVICE

El Servicio de Espectroscopias incluye las Unidades de Espectroscopía Raman, Espectroscopía Infrarroja y Espectroscopía Ultravioleta-Visible. Este servicio está dedicado a la determinación de la estructura molecular de los compuestos químicos y la caracterización de materiales.

This Service consists of four different spectroscopies: Raman Spectroscopy, Infrared Spectroscopy and Ultraviolet-Visible Spectroscopy. It is devoted to the determination of molecular structure of chemical compounds and materials.

ESPECTROSCOPÍA MICRO-RAMAN / MICRO-RAMAN SPECTROSCOPY

La espectroscopía Raman se basa en un proceso fotónico en el que la radiación incidente es dispersada por la muestra, produciéndose transiciones de tipo vibracional y rotacional. En general, el espectro Raman se interpreta como un espectro vibracional que ofrece información muy similar al espectro de infrarrojo, aunque las vibraciones que se ven reflejadas en el espectro Raman no son siempre las mismas que en aquél. Para que un modo vibracional sea activo en espectroscopía Raman es necesario que se produzcan cambios en la polarizabilidad de los enlaces químicos o la molécula considerada, lo que conlleva la producción de momentos dipolares inducidos. Su campo de aplicación es muy extenso: semiconductores, compuestos del carbono (grafito, diamante, nanotubos, fibras…), catalizadores, pigmentos, etc.

Raman spectroscopy is based on a photonic process in which the incident radiation is dispersed by the sample. This latter is perturbed leading to vibrational and rotational transitions. In general, the Raman spectrum is interpreted like a vibrational one, providing information very similar to the infrared spectroscopy, although the Raman active vibrations are not always the same as those excited with infrared radiation. A Raman vibration mode is active if there is a change of polarizability of the chemical bonds or the considered molecule, which in turn results in the generation of induced dipolar momentam. Its application fields are very broad: semiconductors, carbon compounds (graphite, diamond, nanotubes, fibers…), catalysts, pigments, etc.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- LabRAM Horiba Jobin Yvon dotado de un microscopio confocal y 3 longitudes de excitación (785 cm^{-1} rojo, 532 cm^{-1} verde, y 325 cm^{-1} UV)
  LabRAM Horiba Jobin Yvon equipped with a confocal microscope and 3 excitation lasers (785 cm^{-1} red, 532 cm^{-1} green, and 325 cm^{-1} UV)

Responsables Científicos/ Scientific Responsibles: Dr. Juan Carlos Sánchez López, Dr. Hernán Míguez García y Dr. Miguel Ángel Centeno Gallego
Personal Técnico/ Technical Assistant: Dr. Miguel Ángel Avilés Escaño

ESPECTROSCOPÍA INFRARROJA / INFRARED SPECTROSCOPIES

La espectroscopía de Infrarrojos (FT-IR) se basa en la absorción de radiación infrarroja por parte de los materiales. Esta absorción supone un cambio en la energía vibracional de los enlaces, siempre que se produzca un cambio en la polarización de dicho enlace. El resultado obtenido es un espectro en el que se representa la radiación absorbida o transmitida en función del número de onda de la radiación, lo cual permite identificar el enlace correspondiente.
El equipo en el ICMS cubre un rango de número de ondas que va desde 5000 a 250 cm-1 (óptica de CsI) y se puede trabajar con purga o en vacío. Se halla equipado con accesorios para trabajar en los modos de Reflectancia Difusa (DRIFT), Reflectancia Total Atenuada (ATR) y Reflexión Especular. Dispone de un microscopio de Infrarrojos que tiene una resolución lateral de 10 µm.

Infrared spectroscopy (FT-IR) is based on the selective absorption of the infrared radiation by the materials. This absorption means a change in the vibrational energy of the chemical bonds, whenever it occurs a change in the polarization. The result is a spectrum showing the absorbed or transmitted radiation as a function of the wavenumber of the radiation, which can be assigned to the corresponding chemical bound.
The equipment at the ICMS works in a wavenumber range from 5000 to 250 cm^{-1} (CsI optic), and can operate with a gas purge or in vacuum. It is equipped with several accessories to do Diffuse Reflectance (DRIFT), Attenuated Total Reflectance (ATR) or Specular Reflectance. It has got an Infrared Microscope with a lateral resolution of 10 µm.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- JASCO FT/IR-6200 IRT-5000
  JASCO FT/IR-6200 IRT-5000

Responsables Científicos/ Scientific Responsibles: Dr. Juan Carlos Sánchez López, Dr. Hernán Míguez García y Dr. Miguel Ángel Centeno Gallego
Personal Técnico/ Technical Assistant: Dr. Miguel Ángel Avilés Escaño

ESPECTROSCOPÍA ÓPTICA EN EL RANGO VIOLETA, VISIBLE E INFRARROJO CERCANO / ULTRAVIOLET-VISIBLE SPECTROSCOPIES

La técnica de espectroscopía en el rango ultravioleta, visible e infrarrojo cercano (UV-Vis-NIR) nos permite conocer como materiales de distinta morfología (principalmente polvos, láminas y partículas o moléculas en suspensión) reflejan y transmiten la luz incidente en el rango comprendido entre 190 nm y 3000 nm. De esta forma, es posible extraer información sobre su eficiencia como filtros ópticos, ya sean especulares o difusores, y/o sobre la luz absorbida por ellos, lo que indirectamente nos permite estimar su gap electrónico (en el caso de dieléctricos), las transiciones electrónicas que tienen lugar (en el caso de moléculas o sistemas dopados con átomos de otra especie), o las resonancias plasmónicas (en el caso de metales).

The Ultraviolet-Visible Spectroscopy (UV-Vis) reports on the existing energy differences between the more external occupied electronic levels and the nearer unoccupied ones.
There are equipments in the laboratory, which work in the wavelength range of 190 nm to 900 nm. It can operate in the Transmission mode or in Diffuse Reflectance Modes.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- SHIMADZU UV-2101 PC
  SHIMADZU UV-2101 PC
- Cary 5000 + UMA (Universal Measurement Accesory)
  Cary 5000 + UMA (Universal Measurement Accesory)
- Cary 300
  Cary 300

Responsables Científicos/ Scientific Responsibles: Dr. Juan Carlos Sánchez López, Dr. Hernán Míguez García y Dr. Miguel Ángel Centeno Gallego
Personal Técnico/ Technical Assistant: Dr. Miguel Ángel Avilés Escaño

ESPECTROSCOPÍA DE EMISIÓN ATÓMICA / ATOMIC EMISSION SPECTROSCOPY

La espectroscopía de emisión atómica de plasma acoplado inductivamente (ICP-OES) es una técnica analítica que permite la cuantificación de elementos hasta nivel traza en muestras en solución. La muestra a analizar es nebulizada y conducida a un plasma de argón, en donde se produce la desolvatación, vaporización, atomización e ionización de los elementos a analizar. Los átomos e iones excitados por la elevada energía térmica suministrada por el plasma emiten durante el proceso de relajación radiación electromagnética de longitudes de onda características de cada elemento. La intensidad de las distintas líneas de emisión es proporcional a la concentración del analito y con la correspondiente curva de calibración es posible realizar su cuantificación. Esta técnica presenta elevada sensibilidad, excelente límite de detección (en el rango ppb, µg/L), buena precisión, alto rendimiento y capacidad multi-elemental, aunque en determinadas ocasiones se pueden producir interferencias espectrales debido a un alto número de líneas de emisión.
Inductively coupled plasma atomic emission spectroscopy (ICP-OES) is an analytical technique that allows the quantification of elements up to the trace level in samples in solution. The sample to be analyzed is nebulized and conducted to an argon plasma, where desolvation, vaporization, atomization and ionization of the elements take place. The atoms and ions reach an excited state by the high thermal energy supplied by the plasma and during the relaxation process electromagnetic radiation is emitted with wavelengths characteristic of each element. The intensity of the different emission lines is proportional to the concentration of the elements, which can be quantified by using appropriate calibration curves. This technique has high detection limits (in the ppb range, µg / L), good reliability, high throughput and multi-elemental capacity, although in some cases spectral interferences can occur due to a high number of emission lines.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- iCAP 7200 ICP-OES Duo (ThermoFisher Scientific)
  iCAP 7200 ICP-OES Duo (ThermoFisher Scientific)

Responsables Científicos/ Scientific Responsibles: Dr. Francisco José Gotor Martínez
Personal Técnico/ Technical Assistant: Lda. Belinda Sigüenza Carballo

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. Alfonso Caballero Martínez
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 1500 °C, 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 1500 °C, 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 Rosa Cejudo

LABORATORIO DE PREPARACIÓN DE MUESTRAS PARA MICROSCOPÍA ELECTRÓNICA / ELECTRON MICROSCOPY SAMPLES PREPARATION LABORATORY

El laboratorio de preparación de muestras para TEM y SEM dispone de metalizador de oro, evaporador de carbón, metalizador de Cr y carbón, cortadora de disco, pulidora, "disc-grinder", cortadora ultrasónica, pulidora cóncava (dimple) y adelgazador iónico (Fischione 1010).

The laboratory for TEM and SEM samples preparation has a gold coater, a carbon evaporator, a metallization system for Cr and carbon, a diamond wheel cutter, a grinder with disc-grinder device, an ultrasonic cutter, a concave polishing (dimple) and ion thinning (Fischione 1010).

Responsable Científico/ Scientific Responsible: Dra. Asunción Fernández Camacho
Personal Técnico/ Technical Assistants: Dª María Inmaculada Roja Cejudo, Dª Olga Montes Amorín (CicCartuja) y Dra. M. Carmen Jiménez de Haro

SERVICIO DE DIFRACCIÓN DE RAYOS X
X-RAY DIFFRACTION LABORATORY SERVICE

La difracción de rayos-X permite la identificación cualitativa y cuantitativa de sustancias cristalinas y su caracterización microestructural y textural.
El servicio dispone en la actualidad de cuatro difractómetros independientes, configurados específicamente para abordar el análisis de muestras policristalinas de muy distinta naturaleza, en lo referente a su composición, estabilidad química, cristalinidad, etc.
Asimismo, con alguno de ellos se pueden llevar a cabo, además de los análisis rutinarios (θ-2θ), otros varios mas avanzados, como pueden ser:

- Seguir las transformaciones de fase "in situ" provocadas por calentamientos en atmósfera inerte (vacio, Ar) o reactiva (H2, O2,..).
- Caracterizar materiales en la nanoescala (1-100 nm) mediante el estudio de la dispersión de rayos-X a ángulos bajos (SAXS).
- Determinar el grosor, densidad y rugosidad de películas delgadas, mediante Reflectometría de rayos-X.
- Obtener la estructura cristalina de materiales inestables a la atmósfera o muy transparentes a los rayos-X, mediante el empleo de capilares.

X-ray diffraction allows the qualitative and quantitative identification of crystalline substances and their microstructural and textural characterization.
At present, four independent diffractometers are available in this service, specifically configured to analyze the composition, chemical stability, crystallinity and many other properties in polycrystalline samples of a varied nature. Besides ordinary analyses (θ-2θ), part of the equipment can perform some advanced studies as:

- Direct monitoring of transformations undergone in materials under heating, such as phase changes, under inert o reactive atmosphere.
- To characterize materials at the nanoscale (1-100 nm) through X-ray scattering at low angles, using the SAXS technique.
- To measure some physical parameters of layers such as density, thickness and surface roughness with the reflectometry setup.
- To obtain the diffraction patterns of samples either sensitive to the atmosphere or highly transparent to X-rays (organic compounds) employing the capillary configuration.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

- Difractómetro Panalytical X'PERT PRO con cargador automático de muestras
  Diffractometer PANALYTICAL X'PERT PRO with automatic sample charger
- Difractómetro PHILIPS X'PERT PRO con cámara de alta temperatura (1200 ^{o}C) ANTON PAAR HTK 1200
  Diffractometer PHILIPS X'PERT PRO with high temperature chamber (1200 ^{o}C) 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: Dr. 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 PHOIBOS 100-DLD, compuesto de:
Photoelectron Spectrometer PHOIBOS 100-DLD, consisting on:

- Cámara de análisis, analizador hemiesférico multicanal PHOIBOS 100-DLD, 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, ISS y REELS, así como estudios angulares.
  Analysis Chamber, equipped with a hemispheric multichannel analyser PHOIBOS 100-DLD, 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. ISS 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<800 °C) 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 (< 800 °C) 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 °C, 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 800 °C (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 < 150 °C). 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 < 150 °C). It is also possible to sputter the samples under an accelerated ion beam (0.5- 5.0 kV) using inert or reactive gases. Incorporation of some other equipment (Visible light illumination, metal evaporators) is also contemplated.

Responsables Científicos/ Scientific Responsibles: Dr. Juan Pedro Espinós Manzorro y Dr. Juan Pedro Holgado Vázquez
Personal Técnico/Technical Assistant: Dra. Florencia Vattier Lagarrigue

SERVICIO DE MECANIZADO/ MECHANIZED WORKSHOP

Se trata de un servicio horizontal fundamental para el Instituto y unidades externas adscritas al mismo. Ya que permite mejorar, modificar y adecuar el material y equipamiento científico a las necesidades de cada investigador y/o investigación en curso. Incluso llegando a su fabricación partiendo de una necesidad concreta. Ofreciendo asesoramiento técnico, diseño y fabricación de todos los elementos anteriormente descritos.
Además brinda la posibilidad de realizar pequeñas reparaciones y parte del mantenimiento general del equipamiento científico y de laboratorio.

This is a service essential for the Institute and external drives attached to the same. Because it allows you to improve, modify and adapt the material and scientific equipment to the needs of each researcher and/or research in progress. Even going to the extent of their manufacture on

the basis of a specific need. Offering technical advice, design and manufacture of all elements described above.

INSTRUMENTAL DISPONIBLE / AVAILABLE EQUIPMENT

El servicio cuenta con herramientas manuales y herramientas eléctricas para la conformación de materiales muy diversos. Contando con la posibilidad unión de diversos materiales mediante los procesos de soldeo que se pueden realizar en el servicio:

The service account with hand tools and power tools for the formation of very diverse materials. With the possibility union of various materials by welding processes that can be performed in the service:

- Soldadura fuerte con diferentes aportes
  Brazing with different contributions
- Soldadura por arco eléctrico
  Electric arc welding
- Soldadura TIG sobre aceros
  TIG welding on steel

Para los procesos de mecanizado por arranque de viruta se cuenta con las siguientes máquinas-herramientas:

For the processes of machining by chip is has the following machine-tools:

- Centro de mecanizado,HAAS TM 1P
  HAAS CNC milling machine,TM 1P
- Taladro vertical, ERLO TSAR32
  Vertical drill, ERLO TSAR32
- Torno paralelo convencional PINACHO SC200
  Conventional lathe PINACHO SC200
- Torno paralelo semiautomático PINACHO SMART TURN180
  Semi-automatic lathePINACHO SMART TURN180
- Fresadora de torreta Fortex FTX-4-FC VARIO
  Fortex FTX-4-FC VARIO Milling Machine

Personal Técnico/Technical Assistant: D. Juan Carlos Sánchez Martín, D. Manuel Perea Domínguez y Adrián Gómez Castaño

Actividades Divulgativas y Formativas

Outreach and Teaching Activities

DOCENCIA / TEACHING

MÁSTER / MASTER

MÁSTER EN CIENCIA Y TECNOLOGÍA DE NUEVOS MATERIALES

Organizado por la Universidad de Sevilla
Coordinador: Dra. Regla Ayala Espinar
Fecha de Celebración: Curso Académico 2019-20
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

Profesorado ICMS

Aplicaciones Tecnológicas de Materiales Funcionales (Créditos: 5) | Dr. Victorino Franco García
Catalizadores para la Energía y el Medio Ambiente (Créditos: 5) | Dra. Francisca Romero Sarria | Dr. José Manuel Córdoba Gallego
Corrosión y Recubrimientos Protectores (Créditos: 5) | Dr. Leidy Marcela Martínez Tejada | Antonio Paul Escolano (US)
Fisica del Estado Sólido (Créditos: 5) | Dra. Ángela Gallardo López | Dr. Diego Gómez García
Materiales con Funcionalidad Superficial (Créditos: 5) | Dra. Leidy Marcela Martínez Tejada
Modelización Aplicada a la Caracterización Estructural de Medios Condensados (Créditos: 5) | Dr. Carlos López Cartes (US)| Dra. Regla Ayala Espinar
Propiedades Magnéticas de Materiales (Créditos: 5) | Dr. Victorino Franco García
Procesado de Materiales Estructurales (Créditos: 5) | Dr. Felipe Gutiérrez Mora | Dr. José Antonio Rodríguez Ortíz (US)
Propiedades Térmicas, Dieléctricas y Ópticas (Créditos: 5) | Dr. Francisco J. Romero Landa
Química del Estado Sólido (Créditos: 5) | Dr. Antonio Perejón Pazo | Dr. José Manuel Córdoba Gallego
Recuperación y Transformación de Materiales (Créditos: 5) | Dr. Antonio Perejón Pazo | Dr. Leidy Marcela Martínez Tejada | Dra. Svetlana Ivanova
Síntesis de Materiales y Nanoestructuras (Créditos: 7) | Dr. Luis Bobadilla Baladrón | Dra. Svetlana Ivanova
Técnicas de Caracterización de Materiales (Créditos: 8) | Dra. Ana Morales Rodríguez |Dra. Anna Dimitrova Penkova | Dr. Joaquín Ramírez Rico | Dr. Juan Manuel Montes Martos (US)
Trabajo Fin de Máster (Créditos: 10) | Dra. Ana Morales Rodríguez | Dr. Diego Gómez García | Dr. Felipe Gutiérrez Mora | Dr. Javier Blázquez Gámez | Dr. Jhon Jairo Ipus Bados | Dr. Joaquín Ramírez Rico | Dr. Víctor Morales Flórez

MÁSTER INTERUNIVERSITARIO "LÁSER, PLASMA Y TECNOLOGÍA DE SUPERFICIES"

El Máster Interuniversitario Plasma, Láser y Tecnologías de Superficie es un máster conjunto de la Universidad de Córdoba y la Universidad Politécnica de Madrid, en el que intervienen también investigadores del Instituto de Ciencia de Materiales de Sevilla (Centro mixto CSIC – US) y del Instituto de Ciencia de Materiales de Madrid (CSIC)

Fecha de Celebración: Curso Académico 2019-20
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
https://www.uco.es/organiza/departamentos/fisica/es/novedades/112-master-plasma-laser-y-tecnologias-de-superficie

Profesorado ICMS

Principios Básicos
Interaccion de partículas y radiación con la materia. Láseres (Créditos: 4) | Dra. Asunción Fernández Camacho
Materiales e Ingeniería de Superficies (Créditos: 4) | Dra. Ana Isabel Becerro Nieto | Dr. Alfonso Caballero Martínez | Dr. Agustín R. González-Elipe |Dr. Francisco Yubero Valencia

Tecnologias
Plasmas y Tecnología de superficies (Créditos: 4) | Dr. Alberto Palmero | Dr. José Cotrino Bautista
Tecnologías de lámina delgada (Créditos: 5) | Dra. Ana Isabel Borrás Martos | Dr. Agustín R. González-Elipe | Dra. María del Carmen López Santos
Técnicas de caracterización de superficies y láminas delgadas (Créditos: 5) | Dra. T. Cristina Rojas Ruiz | Dr. Francisco Yubero Valencia

Aplicaciones
Nanotecnología de superficies y sus aplicaciones (Créditos: 4) | Dr. Ángel Barranco Quero | Dr. Juan Ramón Sánchez Valencia (US) | Dr. Francisco Javier Aparicio Rebollo
Nuevos materiales para dispositivos (Créditos: 4) | Dra. Ana Isabel Borrás Martos
Funcionalización de superficies (Créditos: 4) | Dr. Juan Carlos Sánchez-López | Dra. María Carmen López Santos | Dr. Francisco Yubero Valencia

Lugar: Universidad de Córdoba, Universidad Politécnica de Madrid, Instituto de Ciencia de Materiales de Madrid, Instituto de Ciencia de Materiales de Sevilla

Asimismo, el personal del ICMS imparte docencia en titulaciones de Grado y doble Grado de la Universidad de Sevilla. La docencia se desarrolla en diversos centros: Facultad de Física, Facultad de Biología, Facultad de Química, Facultad de Farmacia y Escuela Técnica Superior de Ingeniería Informática.

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

27 agosto | **Ingeniería en la nanoescala: aplicaciones al diseño de recubrimientos funcionales**
Dr. Juan Carlos Sánchez López
Lugar: 1er Webinar en Ciencias Básicas Aplicadas, Universidad del Sinú (Colombia)

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

Ciclo de Conferencias / ICMS Invited Lectures

15 enero | **Cold Plasma Processes for Surface Modification of Materials**
Prof. Charafeddine Jama
Universidad de Lille, Francia

Multifunctional effects are essential for producing higher value added materials, important not only for new technical applications but also for more traditional uses. The growing environmental and energy-saving concerns will also lead to the gradual replacement of many traditional wet chemistry-based processing, using large amounts of water, energy and effluents, by various forms of low-liquor and dry-finishing processes.
The dominant role of plasma-treated surfaces in key industrial sectors, such as microelectronics is well known, and plasmas are being used to modify a huge range of material surfaces, including plastics, polymers, papers, food packaging and biomaterials. In previous works, it was evidenced that cold plasma technologies can induce several surface modifications such as change in surface polarity, grafting of chemicals or deposition of functional coatings. Such modifications are effective to confer new and durable properties to synthetic or natural polymers, without altering their bulk properties.
The presentation will give a comprehensive description and review of the science and technology related to plasmas, with particular emphasis on their potential use in the industry. Examples of surface functionalization of materials achieved by means of cold plasma grafting and/or deposition of hydrophilic or hydrophobic coatings, antibacterial, anticorrosion and fire retardant materials will be presented (Figs. 1&2).

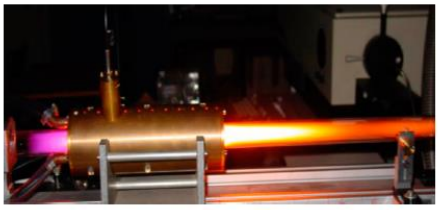


Fig.1 : Nitrogen Plasma Process

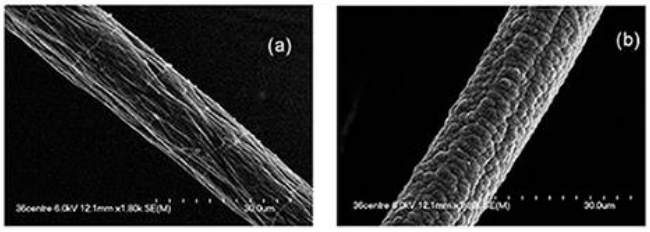


Fig. 2. Plasma deposition of organosilicon coatings: SEM images of (a) Uncoated fiber; (b) Coated fiber

ICMS - sci - talks

19 febrero | **Effect of gold particles size on Au/C catalyst selectivity in HMF oxidation reaction**
Dra. Svetlana Ivanova

10 de marzo | **Influence of Al and Y content on the oxidation resistance of CrAlYN protective coatings for high temperature applications: New insights about the Y role**
Dra. T. Cristina Rojas Ruiz

Seminarios del Grupo Materiales Ópticos Multifuncionales | MOM Seminars

22 abril | **My take on disorder, or how disorder fuels complexity**
Prof. Ceferino López Fernández
Instituto de Ciencias de Materiales de Madrid (CSIC)

29 abril | **Intracellular nanostructures in light harvesting organism as inspiration for biomimetic photonicsen**
Dr. Martín López García
International Iberian Nanotechnology Laboratory

6 mayo | **Bismuth-based colloidal nanocrystals: lead-free materials for solution-processed solar cells**
Dra. María Bernechea Navarro
Instituto de Nanociencia de Aragón. Universidad de Zaragoza

20 mayo | **Introduction to impedance spectroscopy of perovskite solar cells**
Dr. Iván Mora Seró
Institute of Advanced Materials [INAM] de la Universidad Jaume I

10 junio | **Extended open cavities for polaritonic devices**
Prof. Jaime Gómez Rivas
Department of Applied Physics, Eindhoven University of Technology (TU/e), Paises Bajos

17 junio | **Atomic-Scale Insights into Perovskite Solar Cell Materials: Ion Transport, Local Structure and Cation Substitution**
Prof. M Saiful Islam
Dept of Chemistry, University of Bath, Reino Unido

28 octubre | **Fluorescent 2D Nanocrystals for Photonic and Energy Applications**
Prof. Dr. Ir. Iwan Moreels
Department of Chemistry, Faculty of Sciences. Ghent University, Bélgica

4 noviembre | **Strong Coupling in Nanoparticle Arrays: Polaritons, Coherence, And Bose-Einstein Condensation**
Prof. Päivi Törmä
Department of Applied Physics, Aalto University, Finlandia

18 noviembre | **Nanophotonic lasers on a graph**
Prof. Riccardo Sapienza
Faculty of Natural Sciences, Department of Physics, Imperial College London, Reino Unido

2 diciembre | **Persistent phosphors: defect induced opportunities and limitations**
Dr. Philippe Smet
Faculty of Sciences, Department of Solid state sciences, Universiteit Gent, Bélgica

DIVULGACIÓN / DISSEMINATION

FERIA DE LA CIENCIA / FAIR OF SCIENCE

La 18ª Feria de la Ciencia de Sevilla del 2020, un reto virtual (del 17 al 19 de junio de 2020), constituye un punto de encuentro donde se desarrollan actividades de divulgación de la Ciencia y la Tecnología, realizando demostraciones y experimentos para facilitar la comprensión de contenidos científicos.
En esta edición virtual, en el stand del Instituto de Ciencia de Materiales de Sevilla se presentaron: un video institucional, paneles informativos con la investigación que se realiza en las distintas unidades del instituto y una serie de videos con charlas y experimentos:

Video presentación del ICMS: Teresa Cristina Rojas
Un mundo lleno de Catálisis: Dr. Gerardo Colón
Materiales para el control de la luz. Dr. Mauricio Calvo
Celdas solares con nanomateriales y frutos rojos: Dr. Mauricio Calvo, Andrea Rubino, Carlos Romero Pérez

The Fair of Science (17 to 19 June 2020, in Seville) constitutes 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. In this year, a virtual edition was done, were the ICMS presented several videos and panels.

SEMANA DE LA CIENCIA Y LA TECNOLOGÍA / SCIENCE AND TECHNOLOGY WEEK

La semana de la Ciencia (celebrada del 5 al 15 de noviembre de 2020) es un evento de carácter europeo 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.

Café con Ciencia

El café con Ciencia 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 compartió un desayuno virtual 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. Juan Ramón Sánchez Valencia. Tema Nanomateriales: mucho más que miniaturización”

PARTICIPACION EN LA NOCHE EUROPEA / EUROPEAN RESEARCHERS' NIGHT

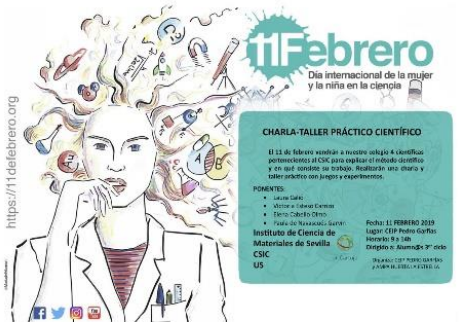
LA NOCHE EUROPEA
DE L@S INVESTIGADOR@S
MUJERES Y HOMBRES QUE HACEN CIENCIA PARA TI

La Noche Europea de los Investigadores, celebrada el Sevilla 27 noviembre de 2020, es un proyecto de divulgación científica enmarcado en Horizonte 2020, bajo las acciones Marie Skłodowska-Curie. Su principal objetivo es acercar los investigadores a los ciudadanos para que conozcan su trabajo, los beneficios que aportan a la sociedad y su repercusión en la vida cotidiana. Se celebra simultáneamente en 371 ciudades europeas desde 2005.

El ICMS ha participado con la siguiente actividad:

Charla: Colourful light emission from tiny particles. Ngo Thi Tuyen

PARTICIPACION EN EL DÍA INTERNACIONAL DE LA MUJER Y LA NIÑA EN LA CIENCIA/ INTERNATIONAL WOMEN AND GIRL'S DAY IN SCIENCE



La ciencia y la igualdad de género son vitales para alcanzar los Objetivos de Desarrollo Sostenible (ODS). Por ello y con el fin de lograr el acceso y la participación plena y equitativa en la ciencia para las mujeres y las niñas, y además para lograr la igualdad de género, la Asamblea General de las Naciones Unidas decidió proclamar en 2016 el 11 de febrero como el Día Internacional de la Mujer y la Niña en la Ciencia. Para la celebración de este dia y con el objetivo de dar a conocer el papel de la mujer en la ciencia y fomentar las vocaciones científicas en las niñas, se organizan en España, a través de la plataforma 11F, muchas actividades donde han participado científicos/as y becarios/as del ICMS.

"Viaje al mundo de los nuevos materiales". 11 febrero. CEIP Pedro Garfias. Laura Caliò, Víctoria Esteso Carrizo, Elena Cabello Olmo, Paula de Navascués Garvín y María Morán Pedroso.

"Plasmanía". Días 11 y 12 de febrero. CEIP Alfares. M. Carmen López Santos y Ana Gómez

"Científicas ilustres: de la piedra al grafeno". Días : 10 de febrero (IES Margarita Salas), 11 de febrero (IES Macarena), 13 de febrero (IES Nervión), 19 de febrero (IES Galileo Galilei). Rosalía Poyato, Ana Morales, Rocío Moriche, Cristina López, Ester López, Encarna Arroyo

"Diamante, grafito y grafeno...¿son familia?" Día 20 de febrero. IES Galileo Galilei. Rosalía Poyato, Ana Morales, Rocío Moriche, Carmen Muñoz, Cristina López.

"¿Qué, para qué y cómo investigamos?" 10 de febrero. Mª Auxiliadora Salesianas (Valverde del Camino (Huelva)). Ana Isabel Becerro

"Viaje al interior de los Nanomateriales a través de la Microscopía Electrónica". Días: 14 de febrero (Colegio Salesiano Nuestra Señora del Águila. Alcalá de Guadaira) y 17 de febrero (IES Galileo Galilei). Teresa Cristina Rojas

"Celebración del Dia Internacional de la Mujer y la Niña en la Ciencia del Colegio María Auxiliadora". Dias: del 12 al 19 de febrero (Colegio María Auxiliadora, Salesianas de San Vicente de Sevilla). José Manuel Obrero, Paula de Navascués Garvin, Ester López, Javier Castillo-Seoane; Aurelio García Valenzuela; Xabier García-Casas, Ángel Barranco, Ana Borrás, Víctor Rico, Jorge Gil, Carmen López-Santos, Juan Ramón Sánchez-Valencia, Víctor López-Flores, Isabel Montealegre Meléndez, Felipe Gutiérrez-Mora.

Actividades:

- Por qué celebramos el 11F, historias de científicas.
- ¿Qué es la Nanotecnología? El plasma, cuarto estado de la materia, en las estrellas y algo más. Nanogeneradores
- Experimentos:
- Montaje de un robot con elementos infantiles
- Microscopio Digital
- Lámpara de plasma activada por sonido y tacto
- Reacciones químicas y electroquímicas
- Láseres de diferentes colores, activación de materiales luminiscentes
- Fabricación de nanogeneradores triboeléctricos y encendido de LEDs con triboelectricidad

PARTICIPACION EN FESTIVAL DE NANOCIENCÍA Y NANOTECNOLOGÍA 10 A LA MENOS 9 / NANOSCIENCE AND NANOTECHNOLOGY 10 A LA MENOS 9

El VI Festival de Nanociencia, es un festival organizado a nivel nacional, que pretende, de una forma amena, acercar a todos los públicos la escala nanométrica, sus efectos y cómo este conocimiento va a cambiar nuestras vidas a través de innumerables aplicaciones y productos. Este año se ha celebrado en Sevilla del 17 al 20 febrero con la realización de las siguientes actividades dirigidas a alumnos de 4 de la ESO y de 1 y 2 de bachillerato del IES Galileo Galilei:

Charlas:

Nanomateriales, mucho más que miniaturización. Juan Ramón Sánchez-Valencia

Viaje al nanomundo a través de la microscopía electrónica. Teresa Cristina Rojas

Paradigma energético y cambio climático. Materiales para eficiencia energética y control de la luz. Gabriel Lozano

Talleres:

Fabricación de una Celda Solar de Colorante. Mauricio Calvo Roggiani, Andrea Rubino, Carlos Romero Pérez

Grafito, grafeno y diamante ¿son familia? Rosalía Poyato, Carmen Muñoz Ferreiro, Cristina López.

Exposición:

Viaje al interior de los Nanomateriales a través de la microscopía electrónica

CONCURSO DE NANORELATOS:

Este concurso, a nivel de Andalucía, está organizado por el Instituto de Ciencia de Materiales de Sevilla, dentro del marco del VI Festival de la Nanociencia y la Nanotecnología 10alamenos9. El objetivo de este concurso es fomentar en la comunidad educativa el interés por el mundo a escala nanométrica, el denominado nanomundo, que a pesar de su diminuto tamaño es muy diverso e interesante. Hay dos categorías, una para estudiantes de 3 y 4 de la ESO y otra para 1 y 2 bachillerato, dotadas con premios de 200€ y diploma.

NANODAY: 9 octubre

Para conmemorar el día Internacional de la Nanociencia y la nanotecnología se realizaron actividades virtuales: videos de charlas científicas, talleres y exposición de imágenes de microscopía.

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