ELECMI

MEMORIA DE ACTIVIDADES 2016

Infraestructura Integrada de Microscopía Electrónica de Materiales

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1 Introducción y estructura del consorcio ELECMI

La Infraestructura Integrada de Microscopia Electrónica de Materiales, ELECMI, es una Infraestructura Científico Técnica Singular (ICTS) en el actual mapa de ICTS del Estado Español, compuesto por un total de 29 ICTS y un total de 59 nodos. ELECMI está formada por dos nodos, el CNME (Centro Nacional de Microscopia Electrónica) perteneciente a la Universidad Complutense de Madrid y el LMA (Laboratorio de Microscopias Avanzadas) de la Universidad de Zaragoza.

ELECMI pone a disposición de la comunidad científica e industrial los equipamientos más avanzados en el ámbito de la observación, caracterización, nanoestructuración y manipulación de materiales a escala atómica y escala molecular.

2 ELECMI en cifras 2016

3 Actividad científica

3.1 Publicaciones Científicas

A lo largo de 2016 se han publicado un total 193 artículos científicos por parte de los 2 nodos integrantes de la ICTS ELECMI.

- Número de artículos con índice de impacto ≥ 10: 17.
- Número de artículos con índice de impacto ≥ 3: 168.
- Número de artículos con índice de impacto < 3: 25.

Revistas en las que se han realizado las publicaciones:

ACS APPL MATER INTER	CLIN INFECT DIS	NANOTECHNOLOGY
ACS NANO	CO2 UTIL	NAT CHEM
ACS PHOTONICS	COMPOS SCI TECHNOL	NAT MATERIALS
ACTA BIOMATER	DALTON T	NAT PHYS
ACTA MATER	DESALINATION	NAT. COMMUN
ADVANCED FUCTIONAL MATERIALS	ELECTROCHIM ACTA	NATURE NANOTECHNOLOGY
ADVANCED MATERIALS	FOOD CHEM	NEW PHYTOL
ADVANCED SCIENCE	INORG CHEM	NON CRYST SOLIDS
ANALYST	INORG CHEM FRONT	ONCOTARGET
ANGEW CHEM INT EDIT	AM HEART ASSOC	ORE GEOL REV
ANTIMICROB AGENTS CH	BIOL CHEM	ORG CHEM
ANTIMICROB CHEMOTH	COLLOID INTERF SCI	PHYS CHEM CHEM PHYS
APL MATER	MATER CHEM C	PHYSICA STATUS SOLIDI
APL MATERIALS	PHYS CHEM C	PHYSICAL REVIEW
APPLIED PHYSICS LETTERS	JOURNAL OF ALLOYS & COMPOUNDS	PLASMA SCIENCE AND TECHNOLOGY
BEILSTEIN JOURNAL OF NANOTECHNOLOGY	JOURNAL OF PHYSICS	PLOS PATHOG
BRAIN BEHAV IMMUN	JOURNAL OF MATERIALS CHEMISTRY	POLYMER CHEMISTRY
CARBON	AND CHEMISTRY OF SOLIDS	RSC ADVANCES
CEREBR BLOOD F MET	LANGMUIR	SCI REP-UK
CHEM COMMUN	MATER CHEM	SENSOR ACTUAT B-CHEM
CHEM EDUCATOR	MATERIALS AND DESIGN	SMALL
CHEM ENG J	MATERIALS RESEARCH EXPRESS	STROKE
CHEM MATER	MICROSCOPY & MICROANALYSIS	SUPERCONDUCTOR SCIENCE AND TECHNOLOGY
CHEM SCI	NANO LETT	TALANTA
CHEM SOC REV	NANOELECTRONICS AND OPTOELECTRONICS	J OF PHYS & CHEMISTRY LETTERS
CHEM-EUR J	NANOSCALE	VIRULENCE
CHEMISTRY OF MATERIALS	NANOSCALE IN PRESS	
CHEMISTRY-A EUROPEAN JOURNAL		

Artículos destacados:

ARTICLES
PUBLISHED ONLINE: 26 OCTOBER 2015 | DOI: 10.1038/NCHEM.2374

nature chemistry

Synthesis of 'unfeasible' zeolites

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Zeolites are porous aluminosilicate materials that have found applications in many different technologies. However, although simulations suggest that there are millions of possible zeolite topologies, only a little over 200 zeolite frameworks of all compositions are currently known, of which about 50 are pure silica materials. This is known as the zeolite conundrum—why have so few of all the possible structures been made? Several criteria have been formulated to explain why most zeolites are unfeasible synthesis targets. Here we demonstrate the synthesis of two such 'unfeasible' zeolites, IPC-9 and IPC-10, through the assembly–disassembly–organization–reassembly mechanism. These now high-silica zeolites have rare characteristics, such as windows that comprise odd-membered rings. Their synthesis opens up the possibility of preparing other zeolites that have not been accessible by traditional solvothermal synthetic methods. We envisage that these findings may lead to a step change in the number and types of zeolites available for future applications.

Computer enumeration suggests that there are millions of possible zeolite topologies[1–3], yet only about 200 have so far been prepared, primarily using solvothermal procedures[4–6]. This is the zeolite conundrum[7] and to explain it several researchers formulated criteria that suggest zeolites that do not obey certain rules are unfeasible synthesis targets, and so many of the hypothetical zeolites simply cannot be made[8–9]. These criteria include a measure of how far a hypothetical zeolite lies away from the energy–density correlation of known zeolites, measured by a so-called feasibility factor, θ (ref. 6), and a realization that the known zeolites are generally flexible over a significant range of densities—the flexibility window[9]. The most recent criteria postulated are those based on local interatomic distances (LIDs) in which feasible zeolite targets are only those that obey strict criteria that describe limits on the values of the interatomic distances and angles[8]. All previously known zeolites obey all five of the LID criteria (for details of the criteria, see Supplementary Section 5).

It has long been recognized that the known zeolites show a strong correlation between framework energy and density, a finding that was predicted computationally[9,10] and confirmed experimentally[11]. Nevertheless, the largest sets of hypothetical zeolites, postulated simply by connecting SiO_4 tetrahedra in as many ways as possible, do not show such a correlation and, indeed, the potential structures cover a large region in energy–density space[12]. However, it has also been noted that the known zeolites can all be found at the low-density edge of the energy–density distribution of hypothetical zeolites[12,13]. This suggests that known zeolites all obey the correlation not because of the properties of the zeolites formed, but because of kinetic limitations of the synthesis procedure (or, at the very least, a combination of the two)[12]. Thus, solvothermal synthesis is the limiting factor that determines which zeolites are feasible or not. Hence, the development of new synthetic pathways could overcome the limitations of solvothermal synthesis and prepare zeolites that are currently thought to be unfeasible.

In addition, the topological features of zeolites are not evenly distributed in energy–density space, and in particular odd-membered rings (loops in the structure that contain odd numbers of tetrahedral units) are rare at the low density edge of the distribution but are more common away from this edge, which explains why odd-membered rings that comprise seven and nine tetrahedra are so sparse in the known zeolite structures[14,24].

Here we present the structures of two new silica zeolites, IPC-9 and IPC-10. Neither material obeys all the LID criteria and so would be considered unfeasible. Both new zeolites also contain unique two-dimensional (2D) channel systems that contain odd-membered rings. The work opens up the possibility that the majority of hypothetical zeolites, which were once thought not to be feasible synthetic targets, are potentially accessible. We believe that this outcome could revolutionize the types of zeolitic solids available in the future.

Results and discussion

The traditional method of synthesis should not be used to obtain 'unfeasible' zeolites. In particular, the reversibility present in solvothermal synthesis, which will always allow the final structure to avoid any high-energy configurations, should be avoided. Figure 1 shows one such new synthetic pathway for zeolites that is not limited in the same way as solvothermal synthesis, the assembly–disassembly–organization–reassembly (ADOR) mechanism[15–18]. This recent development in zeolite synthesis allows one to disassemble selectively a parent germanosilicate zeolite, in this case one with the UTL structure[19,20], to produce a material called IPC-1P (ref. 21) that comprises layers of known structure, which can then be reconnected to produce new pure silica zeolites.

If the orientational arrangement of the layers with respect to each other is the same as it was in the parent UTL zeolite then reconnecting the layers produces zeolites that obey all the LID criteria and would be described as feasible zeolite targets, irrespective of which units link the layers (such as IPC-2 (ref. 15), IPC-4 (ref. 15) and IPC-6 (ref. 16)). This is not surprising because the layers come from a fully connected zeolite, which means they are geometrically perfectly matched to each other and can be reconnected through the different units without introducing any strain into the new materials. To produce 'unfeasible' zeolites we predicted that the relative arrangement of the layers would need to be changed so as to introduce a small geometric mismatch between them. The slight

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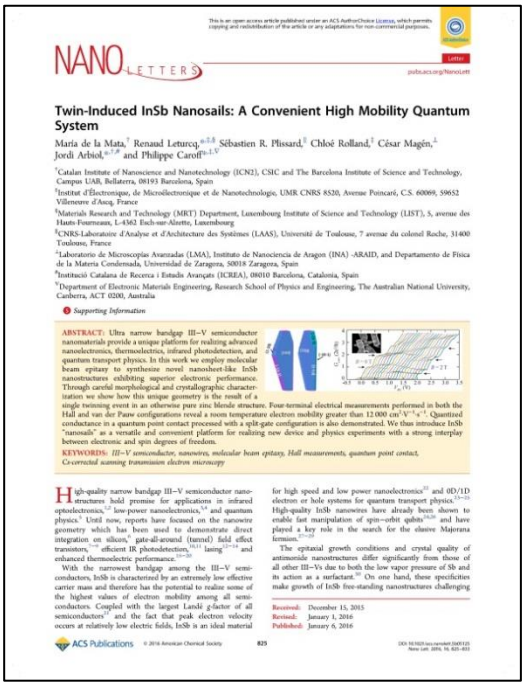

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NANO LETTERS

Letter

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Twin-Induced InSb Nanosails: A Convenient High Mobility Quantum System

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[○]*Supporting Information*

ABSTRACT: Ultra narrow bandgap III−V semiconductor nanomaterials provide a unique platform for realizing advanced nanoelectronics, thermoelectrics, infrared photodetection, and quantum transport physics. In this work we employ molecular beam epitaxy to synthesize novel nanosheet-like InSb nanostructures exhibiting superior electronic performance. Through careful morphological and crystallographic characterization we show how this unique geometry is the result of a single twinning event in an otherwise pure zinc blende structure. Four-terminal electrical measurements performed in both the Hall and van der Pauw configurations reveal a room temperature electron mobility greater than 12 000 $cm^2 V^{-1} s^{-1}$. Quantized conductance in a quantum point contact processed with a split-gate configuration is also demonstrated. We thus introduce InSb "nanosails" as a versatile and convenient platform for realizing new device and physics experiments with a strong interplay between electronic and spin degrees of freedom.

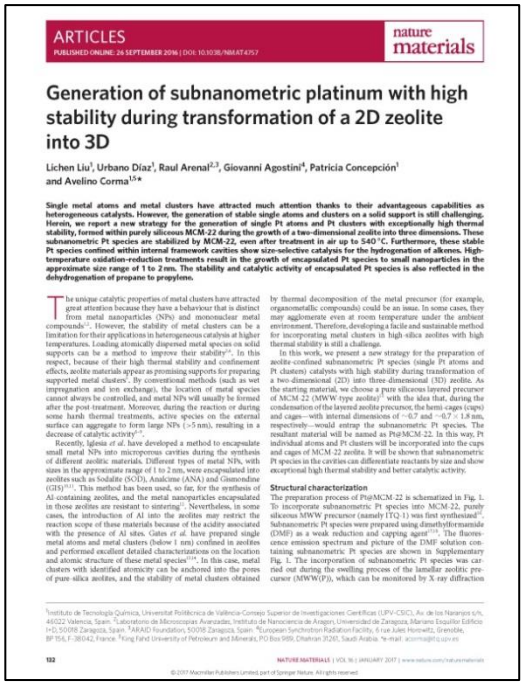
KEYWORDS: *III−V semiconductor, nanowires, molecular beam epitaxy, Hall measurements, quantum point contact, Cs-corrected scanning transmission electron microscopy*

High-quality narrow bandgap III−V semiconductor nanostructures hold promise for applications in infrared optoelectronics,[1,2] low-power nanoelectronics,[3,4] and quantum physics.[5] Until now, reports have focused on the nanowire geometry which has been used to demonstrate direct integration on silicon,[6] gate-all-around (tunnel) field effect transistors,[7,8] efficient IR photodetection,[9,11] lasing[12−14] and enhanced thermoelectric performance.[15−20]

With the narrowest bandgap among the III−V semiconductors, InSb is characterized by an extremely low effective carrier mass and therefore has the potential to realize some of the highest values of electron mobility among all semiconductors. Coupled with the largest Landé g-factor of all semiconductors[21] and the fact that peak electron velocity occurs at relatively low electric fields, InSb is an ideal material for high speed and low power nanoelectronics[22] and 0D/1D electron or hole systems for quantum transport physics.[23−25] High-quality InSb nanowires have already been shown to enable fast manipulation of spin−orbit qubits[26,27] and have played a key role in the search for the elusive Majorana fermion.[27−29]

The epitaxial growth conditions and crystal quality of antimonide nanostructures differ significantly from those of all other III−Vs due to both the low vapor pressure of Sb and its action as a surfactant.[30] On one hand, these specificities make growth of InSb free-standing nanostructures challenging.

Received: December 15, 2015
Revised: January 1, 2016
Published: January 6, 2016



ARTICLES
PUBLISHED ONLINE: 26 SEPTEMBER 2016 | DOI: 10.1038/NMAT4757

nature materials

Generation of subnanometric platinum with high stability during transformation of a 2D zeolite into 3D

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Single metal atoms and metal clusters have attracted much attention thanks to their advantageous capabilities as heterogeneous catalysts. However, the generation of stable single atoms and clusters on a solid support is still challenging. Herein, we report a new strategy for the generation of single Pt atoms and Pt clusters with exceptionally high thermal stability, formed within purely siliceous MCM-22 during the growth of a two-dimensional zeolite into three dimensions. These subnanometric Pt species are stabilized by MCM-22, even after treatment in air up to 540 °C. Furthermore, these stable Pt species confined within internal framework cavities show size-selective catalysis for the hydrogenation of alkenes. High-temperature oxidation-reduction treatments result in the growth of encapsulated Pt species to small nanoparticles in the approximate size range of 1 to 2 nm. The stability and catalytic activity of encapsulated Pt species is also reflected in the dehydrogenation of propane to propylene.

The unique catalytic properties of metal clusters have attracted great attention because they have a behaviour that is distinct from metal nanoparticles (NPs) and mononuclear metal compounds[1–3]. However, the stability of metal clusters can be a limitation for their applications in heterogeneous catalysis at higher temperatures. Loading atomically dispersed metal species on solid supports can be a method to improve their stability[4,5]. In this respect, because of their high thermal stability and confinement effects, zeolite materials appear as promising supports for preparing supported metal clusters[6]. By conventional methods (such as wet impregnation and ion exchange), the location of metal species cannot always be controlled, and metal NPs will usually be formed after the post-treatment. Moreover, during the reaction or during some harsh thermal treatments, active species on the external surface can aggregate to form large NPs (>5 nm), resulting in a decrease of catalytic activity[7–9].

Recently, Iglesia *et al.* have developed a method to encapsulate small metal NPs into microporous cavities during the synthesis of different zeolitic materials. Different types of metal NPs, with sizes in the approximate range of 1 to 2 nm, were encapsulated into zeolites such as Sodalite (SOD), Analcime (ANA) and Gismondine (GIS)[10,11]. This method has been used, so far, for the synthesis of Al-containing zeolites, and the metal nanoparticles encapsulated in those zeolites are resistant to sintering[12]. Nevertheless, in some cases, the introduction of Al into the zeolites may restrict the reaction scope of these materials because of the acidity associated with the presence of Al sites. Gates *et al.* have prepared single metal atoms and metal clusters (below 1 nm) confined in zeolites and performed excellent detailed characterizations on the location and atomic structure of these metal species[13,14]. In this case, metal clusters with identified atomicity can be anchored into the pores of pure-silica zeolites, and the stability of metal clusters obtained by thermal decomposition of the metal precursor (for example, organometallic compounds) could be an issue. In some cases, they may agglomerate even at room temperature under the ambient environment. Therefore, developing a facile and sustainable method for incorporating metal clusters in high-silica zeolites with high thermal stability is still a challenge.

In this work, we present a new strategy for the preparation of zeolite-confined subnanometric Pt species (single Pt atoms and Pt clusters) catalysts with high stability during transformation of a two-dimensional (2D) into three-dimensional (3D) zeolite. As the starting material, we choose a pure siliceous layered precursor of MCM-22 (MWW-type zeolite)[15] with the idea that, during the condensation of the layered zeolite precursor, the hemi-cages (cups) and cages—with internal dimensions of ~0.7 and ~0.7 × 1.8 nm, respectively—would entrap the subnanometric Pt species. The resultant material will be named as Pt@MCM-22. In this way, Pt individual atoms and Pt clusters will be incorporated into the cups and cages of MCM-22 zeolite. It will be shown that subnanometric Pt species in the cavities can differentiate reactants by size and show exceptional high thermal stability and better catalytic activity.

Structural characterization

The preparation process of Pt@MCM-22 is schematized in Fig. 1. To incorporate subnanometric Pt species into MCM-22, purely siliceous MWW precursor (namely ITQ-1) was first synthesized[15]. Subnanometric Pt species were prepared using dimethylformamide (DMF) as a weak reduction and capping agent[16]. The fluorescence emission spectrum and picture of the DMF solution containing subnanometric Pt species are shown in Supplementary Fig. 1. The incorporation of subnanometric Pt species was carried out during the swelling process of the lamellar zeolitic precursor (MWW(P)), which can be monitored by X-ray diffraction

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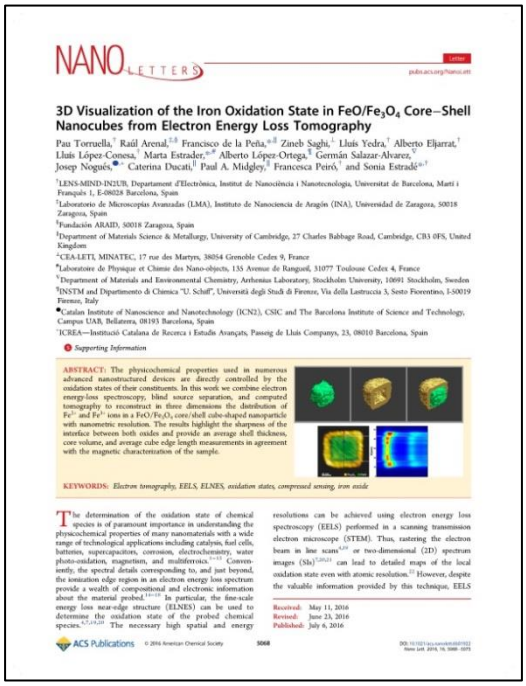

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3D Visualization of the Iron Oxidation State in FeO/Fe_3O_4 Core−Shell Nanocubes from Electron Energy Loss Tomography

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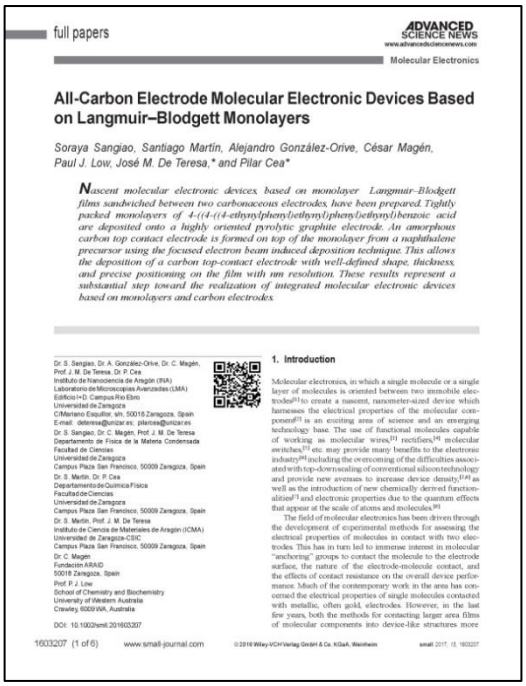
Supporting Information

ABSTRACT: The physicochemical properties used in numerous advanced nanostructured devices are directly controlled by the oxidation states of their constituents. In this work we combine electron energy-loss spectroscopy, blind source separation, and computed tomography to reconstruct in three dimensions the distribution of Fe^{2+} and Fe^{3+} ions in a FeO/Fe_3O_4 core/shell cube-shaped nanoparticle with nanometric resolution. The results highlight the sharpness of the interface between both oxides and provide an average shell thickness, core volume, and average cube edge length measurements in agreement with the magnetic characterization of the sample.

KEYWORDS: *Electron tomography, EELS, ELNES, oxidation states, compressed sensing, iron oxide*

The determination of the oxidation state of chemical species is of paramount importance in understanding the physicochemical properties of many nanomaterials with a wide range of technological applications including catalysis, fuel cells, batteries, supercapacitors, corrosion, electrochemistry, water photo-oxidation, magnetism, and multiferroics.[1−15] Conveniently, the spectral details corresponding to, and just beyond, the ionization edge region in an electron energy loss spectrum provide a wealth of compositional and electronic information about the material probed.[14−19] In particular, the fine-scale energy loss near-edge structure (ELNES) can be used to determine the oxidation state of the probed chemical species.[5,7,19,20] The necessary high spatial and energy resolutions can be achieved using electron energy loss spectroscopy (EELS) performed in a scanning transmission electron microscope (STEM). Thus, rastering the electron beam in line scans[4,21] or two-dimensional (2D) spectrum images (SIs)[7,20,21] can lead to detailed maps of the local oxidation state even with atomic resolution.[22] However, despite the valuable information provided by this technique, EELS

Received: May 11, 2016
Revised: June 23, 2016
Published: July 6, 2016



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Molecular Electronics

All-Carbon Electrode Molecular Electronic Devices Based on Langmuir–Blodgett Monolayers

Soraya Sangiao, Santiago Martín, Alejandro González-Orive, César Magén, Paul J. Low, José M. De Teresa,* and Pilar Cea*

Nascent molecular electronic devices based on monolayer Langmuir–Blodgett films sandwiched between two carbonaceous electrodes, have been prepared. Tightly packed monolayers of 4-((4-((4-ethynylphenyl)ethynyl)phenyl)ethynyl)benzoic acid are deposited onto a highly oriented pyrolytic graphite electrode. An amorphous carbon top contact electrode is formed on top of the monolayer from a naphthalene precursor using the focused electron beam induced deposition technique. This allows the deposition of a carbon top-contact electrode with well-defined shape, thickness, and precise positioning on the film with nm resolution. These results represent a substantial step toward the realization of integrated molecular electronic devices based on monolayers and carbon electrodes.

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DOI: 10.1002/smll.201603207

1. Introduction

Molecular electronics, in which a single molecule or a single layer of molecules is oriented between two immobile electrodes[1] to create a nascent, nanometer-sized device which harnesses the electrical properties of the molecular component[2] is an exciting area of science and an emerging technology base. The use of functional molecules capable of working as molecular wires,[3] rectifiers,[4] molecular switches,[5] etc. may provide many benefits to the electronic industry[6] including the overcoming of the difficulties associated with top-down scaling of conventional silicon technology and provide new avenues to increase device density,[7,8] as well as the introduction of new chemically derived functionalities[9] and electronic properties due to the quantum effects that appear at the scale of atoms and molecules.[10]

The field of molecular electronics has been driven through the development of experimental methods for assessing the electrical properties of molecules in contact with two electrodes. This has in turn led to immense interest in molecular "anchoring" groups to contact the molecule to the electrode surface, the nature of the electrode-molecule contact, and the effects of contact resistance on the overall device performance. Much of the contemporary work in the area has concerned the electrical properties of single molecules contacted with metallic, often gold, electrodes. However, in the last few years, both the methods for contacting larger area films of molecular components into device-like structures more has opened a path for the exploration of new phases with improved properties compared to their bulk parents,[11–14] e.g., the emergence of a ferroelectric-ferromagnetic state in $EuTiO_3$ (ref 14), the occurrence of incipient ferroelectricity in highly strained $CaMnO_3$ (ref 12), or the formation of a two-dimensional ferromagnetic phase at the domain walls of $TbMnO_3$ (ref 11).

Recently, the coupling between strain gradients and polarization, known as flexoelectricity,[15] has been analyzed intensely due to its remarkable contribution to the polarization and permittivity of inhomogeneously strained ferroelectrics.[16–22] The most dramatic manifestations of flexoelectricity have been reported in thin films, as it is an inherently size-dependent phenomenon where strain gradients are inversely proportional to size.[16,20,22] Interestingly, in contrast to piezoelectricity, flexoelectricity is a universal effect due to the relaxed restriction on crystal symmetry and might emerge even

Received: November 2, 2015
Revised: March 21, 2016
Published: March 21, 2016

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Polar-Graded Multiferroic $SrMnO_3$ Thin Films

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Supporting Information

ABSTRACT: Engineering defects and strains in oxides provides a promising route for the quest of thin film materials with coexisting ferroic orders, multiferroics, with efficient magnetoelectric coupling at room temperature. Precise control of the strain gradient would enable custom tailoring of the multiferroic properties but presently remains challenging. Here we explore the existence of a polar-graded state in epitaxially strained antiferromagnetic $SrMnO_3$ thin films, whose polar nature was predicted theoretically and recently demonstrated experimentally. By means of aberration-corrected scanning transmission electron microscopy we map the polar rotation of the ferroelectric polarization with atomic resolution, both far from and near the domain walls, and find flexoelectricity resulting from vertical strain gradients. The origin of this particular strain state is a gradual distribution of oxygen vacancies across the film thickness, according to electron energy loss spectroscopy. Herein we present a chemistry-mediated route to induce polar rotations in oxygen-deficient multiferroic films, resulting in flexoelectric polar rotations and with potentially enhanced piezoelectricity.

KEYWORDS: *Multiferroics, ferroelectricity, flexoelectricity, aberration-corrected STEM, domain walls*

Multiferroic materials have attracted great interest recently because of their intriguing fundamental physics and the wide range of potential applications such as transducers and information storage.[1−3] Of particular technological impact is the search for materials showing efficient coupling between ferromagnetic and ferroelectric orders that persist at room temperature. Manganese-based perovskite oxides $AMnO_3$, A being an alkaline-earth cation, are particularly promising for this purpose; theoretical calculations reveal the onset of a ferroelectric ground state with strong magnetoelectric coupling since the spontaneous polarization is expected to be driven by the off-centering of the magnetic Mn^{4+} ion.[4,5] The ferroelectric instability is predicted to increase by expansion of the lattice, and in turn, strain-induced ferroelectricity was experimentally demonstrated in Ba-substituted $SrMnO_3$ single crystals in which chemical expansion of the crystal lattice is caused by partially replacing Sr with larger Ba ions.[6]

Alternatively, strains can be induced into films through the use of epitaxial growth and can be utilized to modify the ferroelectric film properties by an adequate choice of the substrate.[7−10] In particular, strain-engineering of multiferroism

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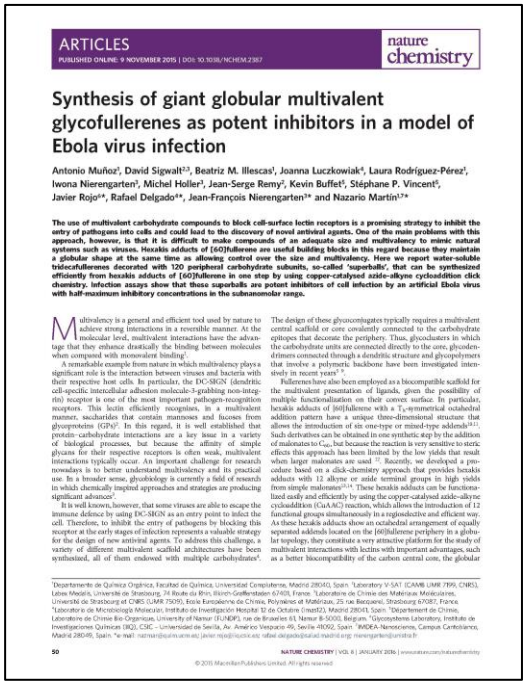

ARTICLES

PUBLISHED ONLINE: 9 NOVEMBER 2015 | DOI: 10.1038/NCHEM.2367

nature chemistry

Synthesis of giant globular multivalent glycofullerenes as potent inhibitors in a model of Ebola virus infection

Antonio Muñoz[†], David Sigwalt[†,‡], Beatriz M. Illescas[†], Joanna Luczkowiak[§], Laura Rodríguez-Pérez[†], Iwona Nierengarten[‡], Michel Holler[‡], Jean-Serge Remy[‡], Kevin Buffet[‡], Stéphane P. Vincent[‖], Javier Rojo*[§], Rafael Delgado*[§], Jean-François Nierengarten*[‡] and Nazario Martín*[†,⊥]

The use of multivalent carbohydrate compounds to block cell-surface lectin receptors is a promising strategy to inhibit the entry of pathogens into cells and could lead to the discovery of novel antiviral agents. One of the main problems with this approach, however, is that it is difficult to make compounds of an adequate size and multivalency to mimic natural systems such as viruses. Hexakis adducts of [60]fullerene are useful building blocks in this regard because they maintain a globular shape at the same time as allowing control over the size and multivalency. Here we report water-soluble tridecafullerenes decorated with 120 peripheral carbohydrate subunits, so-called 'superballs', that can be synthesized efficiently from hexakis adducts of [60]fullerene in one step by using copper-catalysed azide–alkyne cycloaddition click chemistry. Infection assays show that these superballs are potent inhibitors of cell infection by an artificial Ebola virus with half-maximum inhibitory concentrations in the subnanomolar range.

Multivalency is a general and efficient tool used by nature to achieve strong interactions in a reversible manner. At the molecular level, multivalent interactions have the advantage that they enhance drastically the binding between molecules when compared with monovalent binding[1].

A remarkable example from nature in which multivalency plays a significant role is the interaction between viruses and bacteria with their respective host cells. In particular, the DC-SIGN (dendritic cell-specific intercellular adhesion molecule-3-grabbing non-integrin) receptor is one of the most important pathogen-recognition receptors. This lectin efficiently recognizes, in a multivalent manner, saccharides that contain mannoses and fucoses from glycoproteins (GPs)[2]. In this regard, it is well established that protein–carbohydrate interactions are a key issue in a variety of biological processes, but because the affinity of simple glycans for their respective receptors is often weak, multivalent interactions typically occur. An important challenge for research nowadays is to better understand multivalency and its practical use. In a broader sense, glycobiology is currently a field of research in which chemically inspired approaches and strategies are producing significant advances[3].

It is well known, however, that some viruses are able to escape the immune defence by using DC-SIGN as an entry point to infect the cell. Therefore, to inhibit the entry of pathogens by blocking this receptor at the early stages of infection represents a valuable strategy for the design of new antiviral agents. To address this challenge, a variety of different multivalent scaffold architectures have been synthesized, all of them endowed with multiple carbohydrates[4]. The design of these glycoconjugates typically requires a multivalent central scaffold or core covalently connected to the carbohydrate epitopes that decorate the periphery. Thus, glycoclusters in which the carbohydrate units are connected directly to the core, glycodendrimers connected through a dendritic structure and glycopolymers that involve a polymeric backbone have been investigated intensively in recent years[5–9].

Fullerenes have also been employed as a biocompatible scaffold for the multivalent presentation of ligands, given the possibility of multiple functionalization on their convex surface. In particular, hexakis adducts of [60]fullerene with a T_h-symmetrical octahedral addition pattern have a unique three-dimensional structure that allows the introduction of six one-type or mixed-type addends[10–12]. Such derivatives can be obtained in one synthetic step by the addition of malonates to C_{60}, but because the reaction is very sensitive to steric effects this approach has been limited by the low yields that result when larger malonates are used [13]. Recently, we developed a procedure based on a click-chemistry approach that provides hexakis adducts with 12 alkyne or azide terminal groups in high yields from simple malonates[10,14]. These hexakis adducts can be functionalized easily and efficiently by using the copper-catalysed azide–alkyne cycloaddition (CuAAC) reaction, which allows the introduction of 12 functional groups simultaneously in a regioselective and efficient way. As these hexakis adducts show an octahedral arrangement of equally separated addends located on the [60]fullerene periphery in a globular topology, they constitute a very attractive platform for the study of multivalent interactions with lectins with important advantages, such as a better biocompatibility of the carbon central core, the globular

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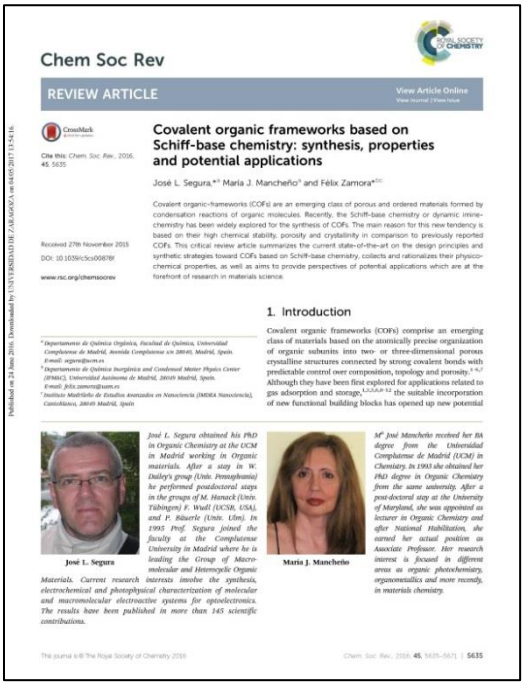

Chem Soc Rev

REVIEW ARTICLE

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Cite this: *Chem. Soc. Rev.*, 2016, 45, 5635

Covalent organic frameworks based on Schiff-base chemistry: synthesis, properties and potential applications

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Received 27th November 2015
DOI: 10.1039/c5cs00878f
www.rsc.org/chemsocrev

Covalent organic-frameworks (COFs) are an emerging class of porous and ordered materials formed by condensation reactions of organic molecules. Recently, the Schiff-base chemistry or dynamic imine-chemistry has been widely explored for the synthesis of COFs. The main reason for this new tendency is based on their high chemical stability, porosity and crystallinity in comparison to previously reported COFs. This critical review article summarizes the current state-of-the-art on the design principles and synthetic strategies toward COFs based on Schiff-base chemistry, collects and rationalizes their physico-chemical properties, as well as aims to provide perspectives of potential applications which are at the forefront of research in materials science.

1. Introduction

Covalent organic frameworks (COFs) comprise an emerging class of materials based on the atomically precise organization of organic subunits into two- or three-dimensional porous crystalline structures connected by strong covalent bonds with predictable control over composition, topology and porosity.[1–4,7] Although they have been first explored for applications related to gas adsorption and storage,[1,2,5,6,8–12] the suitable incorporation of new functional building blocks has opened up new potential

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[c]Instituto Madrileño de Estudios Avanzados en Nanociencia (IMDEA Nanociencia), Cantoblanco, 28049 Madrid, Spain

José L. Segura

José L. Segura obtained his PhD in Organic Chemistry at the UCM in Madrid working in Organic materials. After a stay in W. Dailey's group (Univ. Pennsylvania) he performed postdoctoral stays in the groups of M. Hanack (Univ. Tübingen) F. Wudl (UCSB, USA), and P. Bäuerle (Univ. Ulm). In 1995 Prof. Segura joined the faculty at the Complutense University in Madrid where he is leading the Group of Macromolecular and Heterocyclic Organic Materials. Current research interests involve the synthesis, electrochemical and photophysical characterization of molecular and macromolecular electroactive systems for optoelectronics. The results have been published in more than 345 scientific contributions.

María J. Mancheño

Mª José Mancheño received her BA degree from the Universidad Complutense de Madrid (UCM) in Chemistry. In 1993 she obtained her PhD degree in Organic Chemistry from the same university. After a post-doctoral stay at the University of Maryland, she was appointed as lecturer in Organic Chemistry and after National Habilitation, she earned her actual position as Associate Professor. Her research interest is focused in different areas as organic photochemistry, organometallics and more recently, in materials chemistry.

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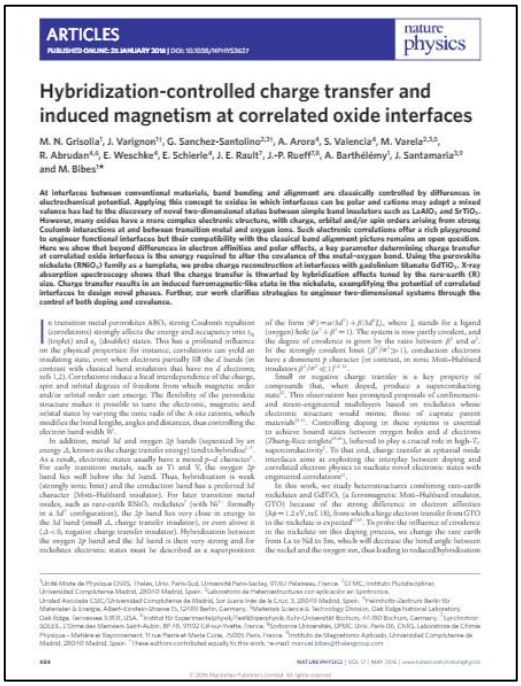
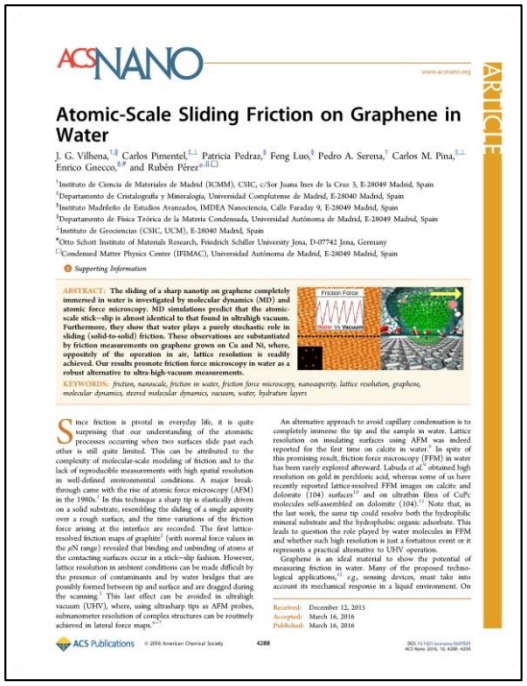


ARTICLES

nature physics

Hybridization-controlled charge transfer and induced magnetism at correlated oxide interfaces



ACS NANO

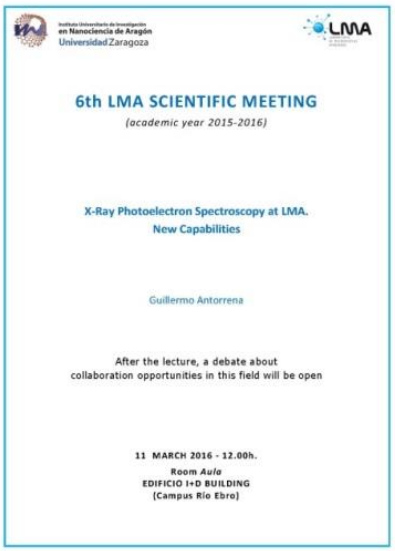
ARTICLE

Atomic-Scale Sliding Friction on Graphene in Water

3.2 Seminarios Científicos LMA-ELECMI

Por parte del nodo LMA-ELECMI se han realizado una serie de seminarios/coloquios temáticos dirigidos a informar a los investigadores locales sobre las posibilidades de nuestras infraestructuras y para fomentar el trabajo interdisciplinar, impartidos por miembros del personal vinculado al LMA-ELECMI:

- VI Seminario Científico Área DUAL BEAM (XPS).
- VII Seminario Científico Área SPM.
- VIII Seminario Científico Área DUAL BEAM (SEM).



3.3 Seminarios Científicos CNME-ELECMI

Por parte del nodo CNME-ELECMI se han realizado los siguientes seminarios científicos:

- **Workshop: "Innivations in In-situ nano characterization for electron microscopes".** Hysitron- IRIDA- UCM. 3-4 Febrero 2016.
- **Trainning: "GRAND ARM300Cf".** JEOL-IZASA. Akira Yamagashi. 22-26 febrero 2016.
- **Conferencia Invitada: "X-ray spectroscopy of transition metal systems".** Dr. Frank de Groot. Debye Institute of Nanomaterials Science Utrecht University (Netherlands). 15 abril 2016.
- **ATRESMEDIA. "Documental Científico".** Dra. Ana Vicente (ICTS-CNME). 16 junio 2016.
- **Workshop: "Microscopía Electrónica".** ALAVA INGENIEROS. 21 octubre 2016.

4 Actividades de formación

International Summer School on Nanofabrication and Transmission Electron Microscopy Characterization.

Organizado en el ámbito de los Cursos Extraordinarios de la Universidad de Zaragoza. El curso tuvo lugar en Jaca los días 18 a 22 de julio de 2016, contó con la participación de investigadores de renombrado prestigio como ponentes y la asistencia de 22 alumnos procedentes de toda la geografía española.

Los objetivos del curso consistieron en introducir a los estudiantes en los campos de:

- **Nanofabricación de materiales.** Con especial hincapié en la importancia de la estructura de dichos materiales en las propiedades finales del compuesto y sus posibles aplicaciones. Profundización en algunas de las técnicas más avanzadas en nanolitografía que hacen uso de microscopias de sonda local, nanoimpresión y haces de electrones e iones focalizados.
- **Caracterización de materiales mediante Microscopia de Transmisión Electrónica (TEM)**. Se ahondó en los fundamentos de este tipo de microscopia analizando su gran versatilidad y potencial de uso haciendo ver al estudiante como diferentes técnicas de imagen en campo claro (BF), campo oscuro (DF), alta resolución (HRTEM) o distintas técnicas de imagen de TEM en barrido (STEM) permiten obtener información precisa sobre la

OBJETIVOS

Los objetivos del curso son introducir a los estudiantes en los campos de:
Nanofabricación de materiales. Se hará hincapié en la importancia de la estructura de dichos materiales en las propiedades finales del compuesto y sus posibles aplicaciones. Se profundizará en algunas de las técnicas más avanzadas en nanolitografía que hacen uso de microscopias de sonda local, nanoimpresión y haces de electrones e iones focalizados.
Caracterización de materiales mediante Microscopia de Transmisión Electrónica (TEM). Se ahondará en los fundamentos de este tipo de microscopia analizando su gran versatilidad y potencial de uso haciendo ver al estudiante como diferentes técnicas de imagen en campo claro (BF), campo oscuro (DF), alta resolución (HRTEM) o distintas técnicas de imagen de TEM en barrido (STEM) permiten obtener información precisa sobre la morfología, la microestructura, la presencia y naturaleza de defectos y la estructura cristalina a escala atómica.
Se estudiarán, asimismo, técnicas asociadas al TEM incluyendo EELS, HAADF y ABF. Se pondrán ejemplos de aplicación para una mejor comprensión de las posibilidades de esta técnica en materiales inorgánicos, magnéticos y biomateriales.

PROGRAMA:

Lunes, 18 de Julio
09:00 h. Overview of the main nanolithography techniques (1)
10:45 h. Electron Beam Lithography and its application in Spintronics (2)
12:30 h. Fabrication of nanodevices (3)

Martes, 19 de Julio
09:00 h. Focused Electron Beam Induced Deposition technique: basics and applications (4)
10:45 h. AFM- based nanolithography techniques (5)
12:30 h. Workshop on practical examples in nanolithography (6)

Miércoles, 20 de Julio
09:00 h. Principles of Transmission Electron Microscopy (7)
10:00 h. Introduction to Diffraction and use of spatially resolved Precession Electron Diffraction PED data to analyze structural deformation in individual nanostructures (8)
12:00 h. HRTEM (9)
13:00 h. STEM (7)
16:00 h. Nanofabrication using microfluidics (10)
17:00 h. Nanowires foo fotacatlytic conversion (11)
18:00 h. Iron based organometallic nanoparticles : physical and catalytic properties (12)
19:00 h. Functional nanostructures (13)
20:00 h. Round table: Nanofabricación y nanocaracterización (12) (13) (10) (15).

Jueves, 21 de Julio
09:00 h. EELS-EDX (14)
10:00 h. Characterization of Advanced Materials using atomic resolution EELS (15)
11:00 h. STEM-EELS en óxidos (14)
12:00 h. Non-planar nanostructures under the electron beam: down to single atom detection (16)
13:00 h. Workshop (I) on practical examples in TEM (7)

Viernes, 22 de Julio
09:00 h. Tomography (17)
10:00 h. Holography (18)
11:00 h. Structural and local spectroscopic TEM studies on (Bio-)Nano-materials (17)
12:00 h. Workshop (II) on practical examples in TEM (17)
13:00 h. Exam on Nanofabrication and TEM (19) (6)
14:00 h. Closure session (19) (6)

PONENTES:
1. Soraya Sangiao Barral (Universidad de Zaragoza)
2. Félix Casanova Fernández (CIC nanoGUNE (San Sebastián))
3. Francesc Pérez Murano (Instituto de Microelectrónica de Barcelona - CSIC)
4. Harald Plank (Graz University of Technology (Graz, Austria))
5. Ricardo García García (ICMM, CSIC)
6. José María de Teresa Nogueras (ICMA (Universidad de Zaragoza-CSIC))
7. César Magén Domínguez (INA, Universidad de Zaragoza)
8. Daniel Mario Ugarte (Dept. Fís. Aplic., Inst. Física "Gleb Waraghin", Univ. Estadual Campinas (Brasil))
9. Mª Luisa Ruiz González (Universidad Complutense de Madrid)
10. Jesús Marcos Santamaría Ramiro (INA-Universidad de Zaragoza)
11. Lun Yeung King (Hong Kong University of Science and Technology)
12. Bruno Chaudret (Instituto LPCNO (Toulouse))
13. Manuel Ricardo Ibarra García (INA-Universidad de Zaragoza)
14. María Varela del Arco (Universidad Complutense de Madrid)
15. Almudena Torres Pardo (Universidad Complutense de Madrid)
16. Jordi Arbiol Cobos (Institut Català de Nanociència i Nanotecnologia (ICN2), CSIC and The Barcelona Institute of Science and Technology (BIST))
17. Raúl Arenal de la Concha (INA, Universidad de Zaragoza)
18. Etienne Snoeck (CEMES/CNRS, Toulouse (Francia))
19. Pilar Cea Mingueza (INA-Universidad de Zaragoza)

RECONOCIMIENTO DE CRÉDITOS: 3 de libre elección 0,5 ECTS.

morfología, la microestructura, la presencia y naturaleza de defectos y la estructura cristalina a escala atómica.

- Se estudiaron, asimismo, t**écnicas asociadas al TEM incluyendo EELS, HAADF y ABF**, con ejemplos de aplicación para una mejor comprensión de las posibilidades de esta técnica en materiales inorgánicos, magnéticos y biomateriales.

Curso Básico de Microscopía Electrónica de Transmisión. Edición Curso 2016-2017

El objetivo del curso fue dar al alumno la formación necesaria para que pueda manejar el equipo como usuario independiente y pueda interpretar por sí mismo los resultados obtenidos. Por ello el curso va dirigido principalmente a aquellas personas que tienen una necesidad continuada de esta tan versátil herramienta. El curso se articula en una parte teórica y una parte eminentemente práctica en laboratorio y lo imparte el personal técnico del área TEM del LMA-ELECMI. El número de alumnos inscritos fue de 24.

Participación del CNME-ELECMI en la XVI SEMANA DE LA CIENCIA

Título: “Viendo átomos con microscopios electrónicos”
Fecha: 16 de noviembre de 2016.
Responsable de la actividad: José Mª González Calbet.
Eje de actuación: Ver funcionamiento de microscopios electrónicos de alta resolución.
Área temática: Química Inorgánica y de Materiales.

Total participantes: 156

Institutos:

- Mirabal (Boadilla del Monte, Madrid): 75 alumnos de 1º y 2º bachillerato.
- Federico García Lorca (Las Rozas, Madrid): 25 alumnos de 1º y 2º bachillerato.
- Pedro Mercedes (Cuenca): 25 alumnos de 2º bachillerato.

UCM:

- Facultad de Químicas, Grupo Materiales Inorgánicos Funcionales: 8 estudiantes.

Particulares:

- 23 visitantes.

5 Actividades con el ámbito empresarial

Curso de Micro- y Nano- Caracterización de Superficies y Materiales Dirigido a Empresas. (2ª Edición)

Los pasados 28, 29 y 30 de Septiembre de 2016 tuvo lugar en las instalaciones del Laboratorio de Microscopías Avanzadas (LMA), la segunda edición del Curso sobre Micro y Nano Caracterización de Superficies y Materiales, dirigido a empresas y usuarios industriales. El curso contó con la presencia de empresas de sectores diversos como el **petroquímico, biotecnológico, alimentación animal, recubrimientos industriales, automoción, materiales de construcción, fabricación de grafeno o sensórica** entre otras. El número de asistentes al curso fue de 18 personas. Los asistentes al curso pudieron conocer las técnicas más avanzadas aplicables a la caracterización y diseño de materiales así como las infraestructuras de vanguardia del LMA, que sin duda utilizarán en un futuro inmediato las empresas asistentes.

Alumnos en las clases teóricas

Grupo de alumnos y docentes del curso

Alumnos usando XPS

Alumnos en sala blanca

Jornada práctica sobre caracterización de materiales y superficies

El pasado 8 de Noviembre de 2016 tuvo lugar en las instalaciones del Instituto de Nanociencia de Aragón (INA) y del Laboratorio de Microscopías Avanzadas (LMA - ELECMI) la jornada práctica sobre caracterización de materiales y superficies. La jornada fue patrocinada por la Cátedra SAMCA de Nanotecnología y contó con la colaboración del Instituto Tecnológico de Aragón (ITAINNOVA).

La jornada fue inaugurada por el Profesor Jesús Santamaría, Director de la Cátedra Samca de Nanotecnología, y por Margarita Canales en representación de ITAINNOVA. La jornada contó con la presencia de empresas de sectores diversos así como con personal del ámbito académico e investigación interesado en conocer las técnicas más avanzadas aplicables a la caracterización y diseño de materiales así como las infraestructuras de vanguardia del LMA.

Número total de asistentes: 30.

Los asistentes mostraron gran interés por el uso futuro de las instalaciones del LMA-ELECMI así como a asistir a próximos cursos y jornadas con el fin de profundizar en las distintas técnicas.

Fotografías de la jornada

6 Acciones de divulgación, promoción y diseminación

Presentación de la ICTS ELECMI en la X Jornada AIN

El pasado 7 de Noviembre de 2016 se celebró en la Cámara de Comercio, Industria y Servicios de Zaragoza la X Jornada sobre Aplicaciones Industriales de la Nanotecnología (AIN). En la jornada, empresas de diversos sectores dieron a conocer las múltiples aplicaciones industriales de la nanotecnología así como proyectos y líneas de actuación que se están llevando a cabo en las respectivas empresas. En este acto, organizado por el Instituto de Nanociencia de Aragón (INA) y el Instituto Catalán de Nanociencia y Nanotecnología (ICN2), se hizo una

presentación de la ICTS ELECMI, por parte de su Promotor Tecnológico, donde se mostraron las capacidades de la ICTS ante el numeroso público asistente, en gran parte del ámbito empresarial.

Inauguración Jornada AIN

Presentación de la ICTS ELECMI

Puesta en marcha del boletín mensual de noticias ELECMI

En el mes de Septiembre de 2016 se lanzó el boletín electrónico de noticias ELECMI con periodicidad mensual. En este boletín se hace un resumen de la información más destacada del consorcio ELECMI, informando sobre eventos, publicaciones y técnicas disponibles en las instalaciones de ELECMI con el objetivo de potenciar el uso de las mismas y dar a conocer a los posibles usuarios las capacidades existentes.

Visitas institucionales y de empresas

Visitas a las instalaciones LMA-ELECMI:

- 18 de Enero de 2016: Visita de Avalor (Asociación de Gestión del Valor y Mejora Continua de Aragón).
- 20 de Abril de 2016: Visita de la Delegación de la Nanjing Tech University, encabezada por su Rector, Profesor Huang Wei.
- 7 de Septiembre de 2016: BSH España, encabezada por su consejero delegado, Sr. José Longás.
- 23 de Septiembre de 2016: Visita de Laboratorios Saphir, encabezada por su Directora Técnica.
- 22 de Diciembre de 2016: Comisión de Innovación, Investigación y Universidad de la Cortes de Aragón.

Visitas a las instalaciones CNME-ELECMI:

- 11 de Marzo de 2016. Visita del Director General GATAN EUROPA, Daniel Momviels.
- 12 de Marzo de 2016. Visita de la Delegación de Rectores de Universidades de Francia.
- 25 de Abril de 2016. Acto de inauguración del microscopio Grand ARM300cF. Asistencia del Rector UCM, Consejero de Educación de la Comunidad de Madrid, Directora General de Innovación y Competitividad del MINECO y del Vice Presidente de JEOL.
- 23 de Mayo de 2016. Visita de la Delegación de la Universidad Regional Amazonas IKIAM encabezada por su Rector Dr. Carlos Ávila.
- 25 de Mayo de 2016. Visita de la Delegación de la Universidad Lima, Perú, encabezada por su Vicerrector Dr. Jaime León.

Entrevista en el programa Ágora de Aragón Radio

Ágora es el programa de Aragón Radio dedicado a la divulgación científica cuyo objetivo es convertirse en un lugar de encuentro para investigadores, docentes, científicos, estudiantes y empresas que compartan el espíritu innovador y la pasión por la ciencia.

El 29 de Septiembre, José Antonio Romero, responsable de promoción de ELECMI, habló acerca de los equipamientos disponibles en ELECMI con el fin de dar a conocer a los oyentes las oportunidades de uso de las instalaciones.

Artículo en Heraldo de Aragón 06/Nov/2016

Con motivo de la celebración de la celebración de la X Jornada sobre Aplicaciones Industriales de la Nanotecnología (AIN), Heraldo de Aragón publicó el artículo “Investigación, la magia de lo más pequeño”, donde se destacaban las líneas de trabajo de las empresas participantes en la jornada, y se hizo una referencia a las instalaciones de la ICTS ELECMI como las más avanzadas del país en el ámbito de la observación y caracterización de materiales a escala nanométrica.

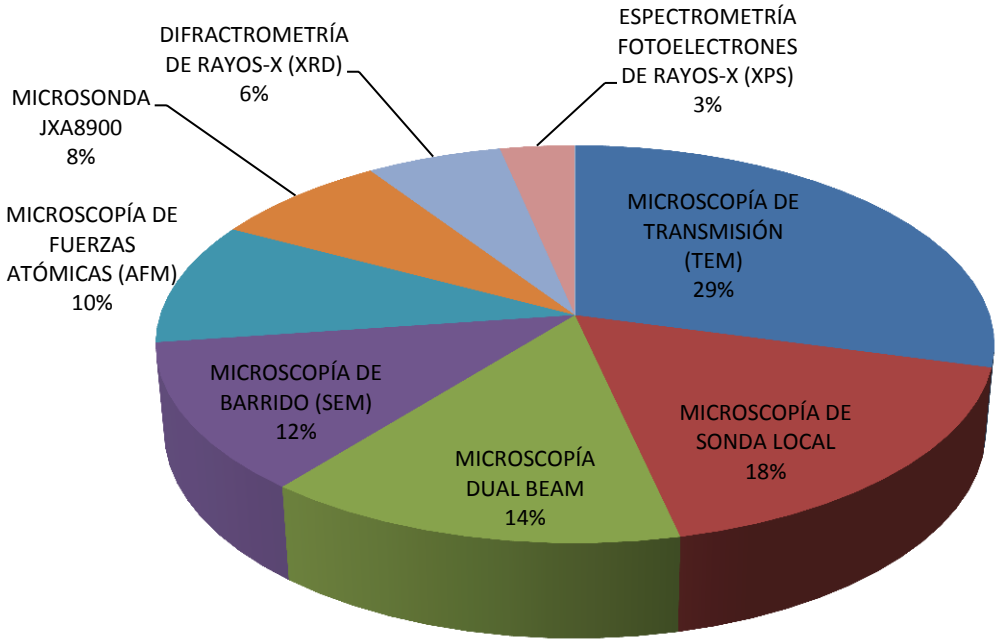
INVESTIGACIÓN
LA MAGIA DE LO MÁS PEQUEÑO

La nanociencia es la nueva revolución industrial, un campo casi infinito abierto a la investigación que impulsa avances determinantes en áreas tan diversas como la textil o la sanitaria

Texto: Picos Laguna

Argonel

7 Estadísticas de uso de la ICTS ELECMI por técnica utilizada

TÉCNICA UTILIZADA	CNME - ELECMI	LMA - ELECMI	ELECMI (conjunto)
MICROSCOPÍA DE TRANSMISIÓN (TEM)	2675	3840	6515
MICROSCOPÍA DE BARRIDO (SEM)	1786	913	2699
MICROSCOPÍA DE FUERZAS ATÓMICAS (AFM)	520	1710	2230
MICROSONDA JXA8900	1755	Técnica no disponible	1755
ESPECTROMETRÍA DE FOTOELECTRONES DE RAYOS-X (XPS)	Técnica no disponible	743	743
DIFRACTROMETRÍA DE RAYOS-X (XRD)	Técnica no disponible	1344	1344
MICROSCOPÍA DUAL BEAM	Técnica no disponible	3176	3176
MICROSCOPÍA DE SONDA LOCAL	Técnica no disponible	3871	3871
NUMERO TOTAL HORAS	**6735**	**15597**	**22331**

% USOS TOTALES ELECMI POR TÉCNICA