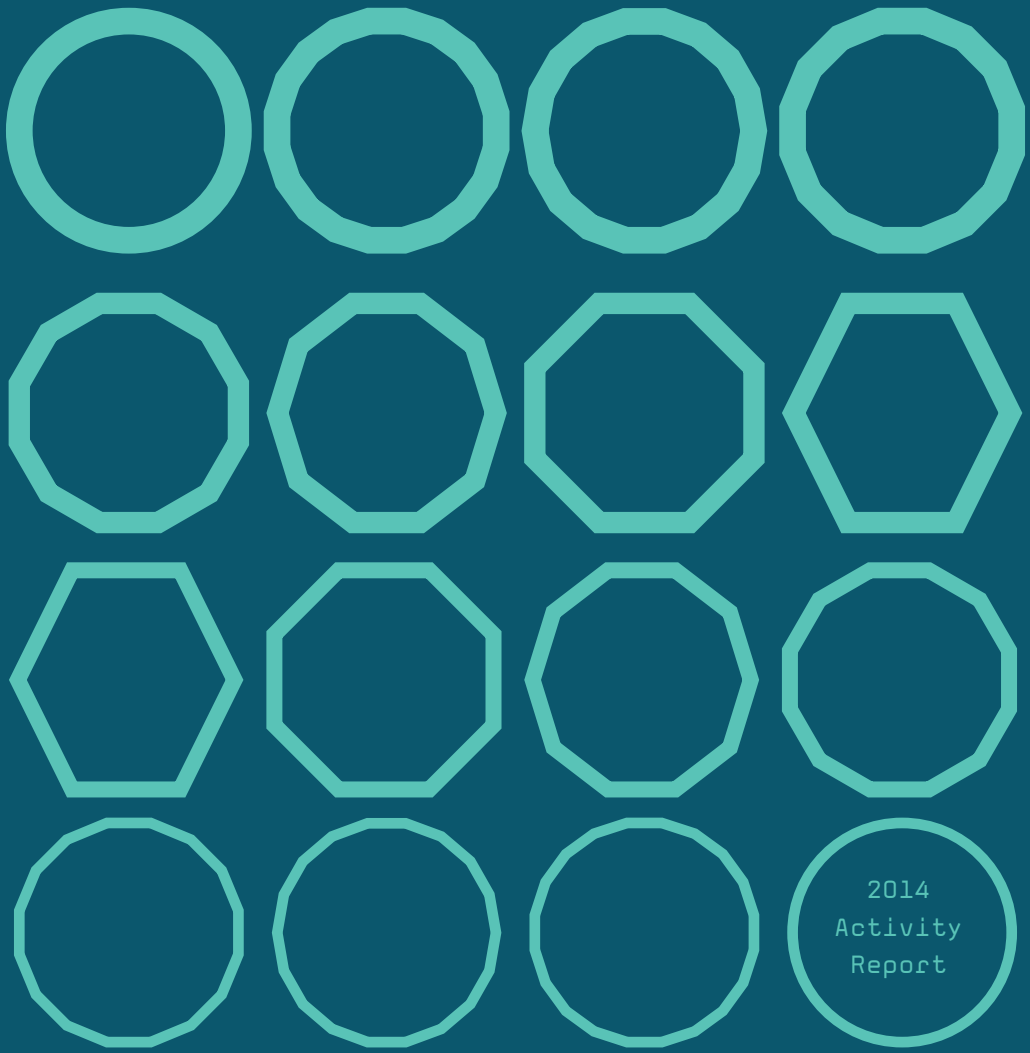
BCMaterials

BASQUE CENTER FOR MATERIAL APPLICATIONS & NANOSTRUCTURES



2014
Activity
Report

BCMATERIALS

BASQUE CENTER FOR MATERIALS, APPLICATIONS & NANOSTRUCTURES

bcmaterials.net

It's for me a pleasure to present BCMaterials 2014 annual report. 2013 was the first year of actual scientific activity, and 2014 settled the operation, incorporated new people and started opening the Center to the world, with new projects and collaborations.

Thanks to the intense and efficient work of the people directly hired by the center, and the extraordinary support of the University's researchers, the quantity and quality of the results are approaching the expected figures for steady operation of BCMaterials.

2014 has followed the showcasing BCMaterials, with an intense activity of visits to other centers, attendance to conferences and meetings, and other activities that helped making BCMaterials better known all around the world. While much has been done, there is still much to be done in the upcoming years.

At the same time, intense work has been undertaken in house and, during the year, all the research lines are running softly, producing results in the form of publications, contributions to conferences, Master and PhD theses, etc. In the upcoming years the activity will indeed increase, yielding a rich harvest of scientific achievements and technological transfers that will be highly beneficial for the Basque society.

With this in mind, I offer you this 2014 annual report, a sample of what we think the future will bring.

With my best regards

JOSÉ M. BARANDIARÁN
Scientific Director

Derio, February 27th, 2015

ORGANIZATION AND MANAGEMENT

INTRODUCTION

Fundación BCMaterials - Basque Center for Materials, Applications and Nanostructures - is a strategic initiative of the Basque science system, whose key drivers are Ikerbasque, the Basque Foundation for Science, and the University of the Basque Country, UPV/EHU.

BCMaterials was created with the objective of establishing a center for cutting-edge research under international quality standards in the area of materials science, which is one of the key priorities of European, Spanish and Basque strategic research programs.

BCMaterials made sense based on existing groups of excellence in materials science at the University of the Basque Country. The Center provides a number of advantages that improve, the visibility, quality and productivity of these groups and boosters the scientific research in materials science in Biscay, providing a instrument for knowledge transfer of materials research to the Basque society.

The key elements of the strategy of BCMaterials are defined in its mission, vision and values, below:

MISSION, VISION, VALUES

Mission

BCMaterials' mission – Basque Center for Materials, Applications and Nanostructures – is to develop high-quality interdisciplinary research in functional and active Materials with advanced Mechanical, Thermal, Electric, Magnetic and Optical properties, from basic aspects to applications. Materials in thin film form and characterization techniques involving Large International

Facilities, such as neutron and synchrotron radiation sources, will be essential.

In order to achieve the mission outlined above, BCMaterials works towards the following challenges:

a.) To work in close collaboration with research groups in Materials Science at the University of the Basque Country (UPV/EHU) in Biscay, promoting worldwide their activity in this field.
b.) To carry out high-level post-graduate training in new materials.
c.) To promote interdisciplinary collaboration with leading centers as the driving force behind major scientific and technological advances.

Vision

BCMaterials is committed to be an international reference center for research in the area of functional & active materials and nanomaterials. The quality of publications in conjunction with the image and prestige, based on research excellence, staff satisfaction and social responsibility, will endorse international recognition.

Values

Reference values assumed by BCMaterials are:

a.) Commitment to the Principles of Excellence.
b.) Transparency, effectiveness and efficiency in resource management.
c.) Satisfaction and development of researchers and other staff through the "European Charter for Researchers".
d.) Quest for maximum return to the society and contribution to sustainability.

Policies to develop a code of conduct in line with the guidelines of the document "European Charter for Researchers" are planned.

Management system

BCMaterials is consolidating the Advanced Management Model (Euskalit) and EFQM Management Model as a reference, and continually improving and systematizing the use of scorecard indicators.

To improve the efficiency and optimize the performance management system we develop a continuous process of identifying and updating technology tools. This includes ensuring the provision of services under the headings of "Information Systems and Technology Platform" on one side, and "Systems Management Applications" on the other.

According to Excellence requirements, BCMaterials management system has developed during 2014 "Managing by Processes" using comparative data and information to drive process improvement. Indicators of process sheets are currently collected, taking into account the Balanced Score Card and aligned to BCMaterials strategy.

Aiming to "Succeed Through People", BCMaterials follows the The Euraxess´ "HR Strategy for Researchers". In October 2014 BCMATERIALS declared the commitment of the Foundation to the principles and recommendations of the Charter and the Code of Conduct and this commitment has been listed at the European Charter & Code of Researchers Endorsements page:

http://ec.europa.eu/euraxess/index.cfm/rights/charterAndCode#S

BCMaterials will implement the Human Resources Strategy for Researchers (HRS4R) during 2015. That includes developing the researchers' adhesion conditions, the Intellectual Property Rights regulations, requirements of any sponsor or funders, and representative gender balance achieving an equal opportunity policy at recruitment without jeopardising quality and competence criteria.

RESEARCH

NEW EQUIPMENT

Several equipment for the development of the research lines has been acquired. some are already installed. We mention:

a.) Cryogen free SQUID: Quantum Design MPMS SQUID system with EverCool; temperature: 1.8K to 400K, magnetic field: 7 Tesla (superconducting coil), sensitivity: 10-8 emu (Support SGiker UPV/EHU for FEDER- MINECO funds).

b.) Vibrating Sample Magnetometer: Microsense EV7; temperature: 77K and 100K to 1000K, magnetic field: 2 Tesla (electromagnet), sensitivity: 0.5 10-6 emu (own funds).

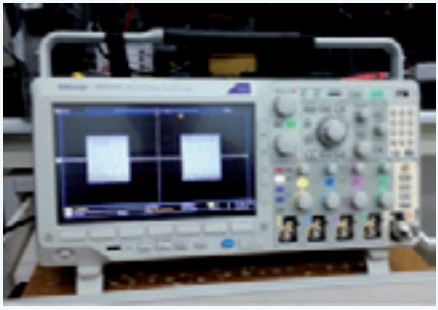
c.) DTMA apparatus for polymers /FSMA. DMA 1 METTLER TOLEDO allows us to measure the mechanical and viscoelastic properties of materials such as thermoplastics, thermosets, elastomers, ceramics and metals. This equipment is able to do measurements in all deformation modes, even in liquids or at different relative humidity levels, in a wide Wide temperature range (from -190 to 600 °C). Also TMA measurements are possible for measuring expansion coefficients, effects due to creep, and relaxation times.

d.) Optical Microscope for Biomaterials NIKON ECLIPSE Ni-E: maximum magnification x1,000, fluorescence image. (UPV/EHU Projects and own funds).

e.) Keysight E4980A Precision LCR Meter, from 20 Hz to 2 MHz (UPV/EHU Projects).

f.) Temperature dependent electrical measurements platform Linkam LTS420, from -196 to 420 oC. (UPV/EHU Projects).

g.) Oscilloscope Tektronix (MDO3014), Mixed Domain Oscilloscope: 100MHz, analog and RF channels. 50MHZ function generator and 9kHz to 3Ghz spectrum analyser (Own funds).



RESEARCH ACTIVITY

Active (Smart) Materiales

These are materials that present crossed properties so that they re-act in the determined way to a stimulus by changing a different property. They are good candidates for integrating devices and structures that can self-accommodate to changing

external conditions and behave as smart devices or systems. Those include: Thermal (thermo-chromic, thermo-electric), Mechanical (Shape Memory Alloys and Polymers, thermo-elastic), Magnetic (magneto-elastic, magneto-resistive, magneto-optic, ferrofluids, Ferromagnetic Shape Memory Alloys, etc.), Electric (photoelectric, advanced piezoelectric materials), Multiferroics, etc.

Magnetic Shape Memory Alloys (MSMA)

Bulk (polycrystalline and single crystals) and low-dimensional (thin ribbons, thin films/substrate composites) multifunctional materials for different performance like high temperature shape memory and superelasticity, magnetoplasticity, power generation and energy harvesting, magneto- and elastocaloric effect, MEMS, etc.

WORK HAS BEEN CARRIED OUT IN THE FOLLOWING SUBJECTS

Bulk MSMAs. A new phenomenon of a sudden disappearance of hysteresis on the stress-strain superelastic curves was found experimentally in single crystalline Ni-Fe(Co)-Ga MSMA. This was explained in the framework of Landau theory of ferroelastic phase transitions by the existence of the critical point on the phase diagram. A very large reversible strain behavior in the postcritical region is very important for new high performance actuator, damping and elastocaloric applications.

The experimental thermomagnetization results and their ab-initio theoretical modeling have shown that the d-electron orbital dependent magnetic ordering is the driving mechanism of magnetostructural instability in metamagnetic SMAs. Particularly this instability, accompanied by a drop of magnetization, governs the size of the inverse magnetocaloric effect.

The low electron concentration region ($e/a < 7.75$) of the magnetic phase diagram of the off-stoichiometric metamagnetic Ni–Mn–Sn SMAs was investigated in detail by DSC and magnetization measurements of the $Ni_{40+x}Mn_{39-x}Sn_{21}$ (x = 0, 2, 4, 6 and 8 at.%) alloys.

Thin Films. The structural, transformation, and magnetic behavior of Ni-Mn-Ga thin films sputter-deposited onto heated Si/SiNx substrates are investigated. These films demonstrated a Curie temperature merged with the martensitic transformation. The latter one accompanies by the inverse volume effect.

SELECTED RESULTS

1. Kosogor, A ; L'vov, VA ; Chernenko, VA ; Villa, E ; Barandiaran, JM ; Fukuda, T ; Terai, T ; Kakeshita, T, Acta Materialia 66 (2014) 79-85.
2. Aseguinolaza, IR ; Orue, I ; Svalov, AV ; Wilson, K ; Mullner, P ; Barandiaran, JM ; Chernenko, VA, Thin Solid Films 558 (2014) 449-454.
3. Lazpita, P ; Barandiaran, JM ; Chernenko, VA ; Garcia, BV ; Tajada, ED ; Lograsso, T ; Schlagel, DL, JALCOM, 594 (2014) 171-174.

Includes 2 main researchers, 2 postdocs and 1 PhD student involved. Has published 8 articles in international journals (1 PRB, 1 ActaMat, 1 JAP, 1 JALCOM, 1 Mat. Trans, 1 Thin Solid Films and 1 Archiv), got 7 invited talks and defended 1PhD thesis during 2014.

Smart Polymers

This line includes, mainly, thermally induced shape memory polymers (SMPs) based on polyolefins, and smart polymer surfaces. The aim of this study is to prepare shape memory Polycyclooctene (PCO) and blends and to study its shape memory behavior in monolithic and film presentations. Smart polymer surfaces include biomolecules responsive and nanostructurated surfaces.

Polyimides

During the last year the piezoelectric polyimides line was centered in two objectives:

- Obtaining different polyimide nanofibers by the electrospinning technique. The experimental conditions were optimized. Initial testing for use as separation membranes for lithium batteries.
- Preparation of nanocomposite films via an in situ polymerization method, using spherical cobalt ferrite ($CoFe_2O_4$) magnetic nanoparticles as fillers and polyimides as matrix.

SELECTED RESULTS

1. Maceiras A, Martins P, San Sebastián M, Lasheras a, Silva M, Laza JM, et al. Synthesis and characterization of novel piezoelectric nitrile copolyimide films for high temperature sensor applications. Smart Mater Struct 2014;105015

Surfaces

The investigation on polymer surfaces has focused on the functionalization and patterning of polymers widely used in fields like medicine or food packaging, such as, polystyrene (PS), polycyclooctene (PCO), polyacrylonitrile (PAN), polyethylene therephthalate (PET) and polylactide (PLA).The aims of this work were nano/microstructuration of polymeric matrixes and functionalization of polymeric surfaces.

- Nano/microstructuration of polymeric matrixes. Breath figures, laser interference lithography and patterning by template methods have been used to structurate surfaces of PS and PCO, among others.
- Functionalization of polymeric surfaces. The interactions between different polymers capable to form polymeric multiplayers have been studied. The studies on solution have reported an interesting information about functionalization and multilayer formation. ionic polyß-cyclodextrins (pCDs) have been synthesized and characterized. Two different types of multilayer have been prepared, ionic poly ß-cyclodextrins (pCDs) and chitosan / hyaluronic acid. Nanocomposites based on biodegradable and biocompatible polylactide (PLA) matrix and cellulose nanocrystals (CNC) have been developed.

Other nanostructuration method, the femtosecond ablation, has been done by Quanta Ray PRO 290-10 in IWS Fraunhofen center in the city of Dresden.

SELECTED RESULTS

1. Ruiz-Rubio L, Vilas JL, Rodríguez M, León LM. Thermal behaviour of H-bonded interpolymer complexes based on polymers with acrylamide or lactame groups and poly(acrylic acid): Influence of N-alkyl and ∝-methyl substitutions. Polym Degrad Stab 2014;109:147–53.
2. Ruiz-Rubio L, Laza JM, Pérez L, Rioja N, Bilbao E. Polymer–polymer complexes of poly(N-isopropylacrylamide) and poly(N,N-diethylacrylamide) with poly(carboxylic acids): a comparative study. Colloid Polym Sci 2014, 209, 423-430.

Organic functionalization of polyoxometalate clusters

One of the aims of this line is focused on the design of smart polymer/polyoxometalate (POM) hybrid coatings with a broad scope of applications (sensors, anticancer agents…). As POMs must be immobilized on a surface for translating their functionality into a device with practical application, a series of suitable POM precursors have been synthesized and chemically and structurally characterised.

- Validation of the preparative approach: Immobilization of clusters by immersion of the functional polymer sufaces in POM solutions has been demonstrated by XPS, confocal Raman spectroscopy and AFM.

- Exploring the POM reactivity toward coordination of organic ligands: the behaviour of a well-know family of POM clusters with accessible metal centres toward organic ligands has been studied. The obtained molecular hybrid species illustrate their potential as structural models for POM-polymer interactions.

- Preparation of novel POMs with exposed metal centers: the synthesis of new POM clusters with accessible 3d- and/or 4f-metal centers suitable for further organic derivatization has been carried out. Some interesting properties of these clusters, such as photoluminescence or magnetism have also been analyzed. The solution behavior has also been studied by a combination of ESI-MS experiments and 183W-NMR spectroscopy. Furthermore, the gigantic and highly charged polyoxotungstates obtained in this section have shown the ability to self-assemble in solution into hollow, single-layered and vesicle-like blackberry-type structures. The formation of the blackberries has been monitored by laser light scattering techniques (DLS and SLS).

SELECTED RESULTS

1. Iturrospe, A.; San Felices, L.; Reinoso, S.; Artetxe, B.; Lezama, L.; Gutiérrez-Zorrilla, J. M. Reversible Dehydration in Polyoxometalate-Based Hybrid Compounds: A Study of Single-Crystal to Single-Crystal Transformations in Keggin-Type Germanotungstates Decorated with Copper(II) Complexes of Tetradentate N-Donor Ligands. Cryst. Growth Des. 2014, 14, 2318-2328.

2. Artetxe, B.; Reinoso, S.; San Felices, L.; Lezama, L.; Gutiérrez-Zorrilla, J. M.; García, J. A.; Galán-Mascarós, J. R.; Haider, A.; Kortz, U.; Vicent, C. Cation-Directed Dimeric vs. Tetrameric Assemblies of Lanthanide-Stabilized Dilacunary Keggin Tungstogermanates. Chem. Eur. J. 2014,20, 12144-12156.

Includes 1 main researcher and 1 PhD student involved. Has published 2 articles in international journals (1 CEJ, 1 CGD) and defended 2PhD thesis (outstanding, cum laude, International mention) during 2014.

Shape memory polymers (SMP)

Polycyclooctene (PCO) sheets have been irradiated at different dosages of gamma rays (from 25 to 200 kGy) in order to obtain shape memory polymers, so we get a set of samples with a crosslinking gradient. All the samples have been thermally and mechanically characterized. Additionally, Soxhlet extraction has been realized in order to remove the not crosslinked fraction of the polymer and thus the gel percentage of each sample can be calculated. The influence of the received radiation dose on the melting temperature as well as on the thermal stability has been studied. Thermal studies have been realized before and after Soxhlet extraction in order to know if the not crosslinked fraction affects these properties. Moreover, shape memory properties have been qualitatively evaluated by studying the behaviour of the polymers at temperatures above and below the transition temperature of the shape memory effect of the polymers. Additionally, the free volume of the samples has been measured. The obtained results have been related with the shape memory results obtained by thermal mechanical analysis. It is important to note that the first obtained results in these studies have been very successful and they allow to add a new characterisation technique which is not usually employed in this area, so a priori this can be really interesting for shape memory polymers

SELECTED RESULTS

1. García-Huete N, Laza JM, Cuevas JM, Gonzalo B, Vilas JL, León LM. Shape memory effect for recovering surface damages on polymer substrates. J Polym Res 2014;21.

2. García-Huete N, Laza JM, Cuevas JM, Vilas JL, Bilbao E, León LM. Study of the effect of gamma irradiation on a commercial polycyclooctene I. Thermal and mechanical properties. Radiat Phys Chem 2014;102:108-16.

Includes 2 main researchers, and 2 PhD student. 4 articles in: J Polymer Res., Rad. Phys. and Chem.y, Polymer Degradation and Stability, and Colloid and Polymer Science, + 2 poster presentation.

Nanostructured Materials

Nanoscale magnetic materials are the basis of a large number of devices and applications in many human activity fields, like Medicine, Electronics, Computer parts, Information storage, etc.

Magnetic Nanoparticles for biomedical applications

This research line includes magnetic nanoparticles prepared by chemical methods and synthesized from bacteria. The main goal is to prepare and characterize multifunctional nanoparticles.

WORK HAS BEEN CARRIED OUT IN THE FOLLOWING SUBJECTS

We have studied novel approaches to develop multifunctional magnetic nanostructures for magnetic hyperthermia. For example, by tuning the shape (spheres, cubes, rods…) and exchange coupling (Fe_3O_4/FeO) of the magnetic nanoparticles, we have managed to remarkably improve their heating efficiency (SAR).

In the case of the preparation of magnetic nanoparticles by chemical methods we have focused the attention in the preparation of magnetic nanoparticles of the type Fe_3O_4, $Fe_{1-x}Ni_xFe_2O_4$ and FePd. They have been synthesized by a thermal decomposition method based on seeded-growth technique, achieving size tunable nanoparticles with high crystallinity and high saturation magnetization. Electron magnetic resonance (EMR) spectroscopy becomes a very efficient complementary tool to determinate the fine details of size distributions of MNPs and even to estimate directly the size in a system composed of a given type of magnetic nanoparticles. The size and size dispersity affects directly to the efficiency of MNPs for hyperthermia (Specific Absorption Rate, SAR value) and EMR provides a direct evaluation of these characteristics almost exactly in the same preparation and with the same concentration as used in

hyperthermia experiments. Optimization of synthesis methods has yield to obtain SAR values greater than 700 W/gFe.

Biocompatibility of these nanoparticles has been obtained in two different ways. Fe3O4 nanoparticles have been recovered by poly(isobutylene-alt-maleic anhidride) polymer provided with COOH groups which have been linked to an intermediate with an alkyne terminal group. By click reaction these groups have been anchored to RGD peptides in order to be delivered to colocarcinome tumors induced in the liver of rats. Hyperthermia experiments have shown increasing temperatures for animals injected with nanoparticles. Fe1-xNixFe2O4 ($0.3 < x < 0.9$) nanoparticles in the 8-18 nm range obtained by the seed growth method have been encapsulated by quitosane. In this sense, biocompatible capsules with different magnetic response have been prepared.

COLLABORATIONS

Dr. J. Martinez de la Fuente (CSIC – Instituto Nanotecnología de Aragón) for in vitro experiments and for studying polymer encapsulation. The student X. LasHeras who is performing his Ph Thesis in BCMaterials has stayed in INA installations for 6 months. Dr. M.P. Morales (Instituto de Ciencia de Materiales de Madrid) for ligand exchange around magnetic nanoparticles. O.K. Arriortua, Ph student, has stayed in these laboratories for three months. Dr. J. Echevarria (Galdakao Hospital) for in vivo experiments with animals inoculated with magnetic nanoparticles. Prof. W. Parak (Marburg University – CIC Biomagune) for transfering NPs to water.

SELECTED RESULTS

1. "Fe3O4 nanoparticles prepared by the seeded-growth route for hyperthermia: electron magnetic resonance as a key tool to evaluate size distribution in magnetic nanoparticles". Idoia Castellanos-Rubio, Maite Insausti, Eneko Garaio, I. Gil de Muro, Fernando Plazaola, Teófilo Rojo and Luis Lezama. Nanoscale, 2014, 6, (7542-7552).

Magnetic nanoparticles from bacteria for hyperthernia applications

Magnetotactic bacteria are ubiquitous motile aquatic prokaryotes that mineralize intracellular magnetic nanoparticles called magnetosomes. In recent years, magnetosomes have attracted great attention because of their potential uses in biomedical and

biotechnological applications. An important characteristic of magnetosomes is their ability to generate heat on application of an alternating magnetic field (AMF). This feature promotes their usefulness in the destruction or inactivation of tumor cells through hyperthermia therapy, currently one of the most promising alternative treatments against cancer.

During last year, we have investigated the interaction of magnetosomes with eukaryotic cells. We have analyzed the biocompatibility of magnetosomes isolated from Magnetospirillum gryphiswaldense and their therapeutic potential as hyperthermia agents in vitro on murine macrophage ANA-1 cell line.

The quality of the isolated magnetosomes was evaluated by Transmission Electronic Microscopy, Dynamic Light Scattering and Infrared Spectroscopy. The magnetosomes present cuboctahedral structure with an average diameter of 45 nm and a moderate stability in aqueous solution, with hydrodynamic diameters centered at 101 and 577 nm and Z potential of -38 mV. We have also measured the specific absorption rate (SAR) of the magnetosomes in phosphate buffer saline by AC magnetometer at selected frequencies 75-532 kHz and an amplitude up to 30 kA/m.

SAR values were higher than 2000 W/g at 532 kHz and 30 kA/m, which are higher than those reported for magnetic nanoparticles produced via chemical synthesis. In a ferrimagnet single domain nanoparticle two main mechanism are involved in the heating process. The first one it is due to the intrisic hysteresis losses of the ferrimagnet nanoparticle and the second one it is due to the physical rotation of the nanoparticle in a magnetic field, Brownian motion. In order to determine the role of each mechanism in the final SAR value, we compare the heating rates of the magnetosomes suspended in liquid medium, in which rotation of the magnetosomes must be taken into account and in gel where rotation is limited. The experimental data are compared with numerical calculations based on dynamic Stoner-Wolhfarth model.

In the cytotoxicity assays, the macrophages were incubated with magnetosomes (30 pg of magnetite per cell) and viability was measured over time by annexin-propidium staining and flow cytometer. Magnetosomes labeled with fluorescein isothiocyanate were used to track the internalization of the nanoparticles into the cells. No evident signs of cytotoxicity were found after 48 hours under the assayed conditions.

Finally, magnetosome-loaded macrophages were exposed to an applied magnetic field with frequency equal to 532 kHz and an amplitude of 25 kA/m during 40 minutes and the viability of the treated cells was evaluated as in the cytotoxicity assays. Further analysis need to be performed in order to obtain conclusive results.

Structure and magnetism of bioferrihydrite synthesized by ferritin protein.

Ferritins are iron-storage and detoxification proteins found in the three domains of life from microorganisms to humans. They are formed by similar or identical subunits self-assembled into a hollow globular shell that contains an iron core consisting of a ferric oxy-hydroxide mineral similar to the nanomineral ferrihydrite.

These proteins are of interest to researchers of different subjects: in biology because ferritin plays a dual role by storing an essential nutrient and protecting cells from the potentially toxic effects of free iron in excess at the same time; in medicine since it has been pointed out the role of ferritin in neurodegenerative diseases; in physics because of the complex structural and magnetic behavior associated to the nano ferric oxy-hydroxide biomineralized by the ferritin proteins, and in chemistry since the protein shell, called apoferritin, can serve as a cavity inside which inorganic nanostructures can be synthesized. Finally, there have been described potential applications of ferritins in the fields of electronics, catálisis and biomedicine. A good knowledge of the structure of the ferrihydrite biomineralizes by eukaryotic and prokaryotic ferritins has important implications in understanding the physical properties of the iron biomineral core. In particular, the magnetic properties are intimately related to the structure at the nanoscale, and a joint analysis combining magnetic and structural techniques can provide a global approach. With this aim, we have studied the structure and magnetic properties of the inorganic core biomineralized by three prokaryotic ferritins: bacterial ferritin (FtnA) and bacterioferritin (Bfr) of Escherichia coli and archaeal ferritin (PfFtn) of Pyrococcus furiosus and the results we compared with those of eukaritic ferritin, from horse soleen ferritin. The structure of the protein biomineral cores has been studied by means of X-ray Absorption Spectroscopy (XAS) on the Fe K-edge and High Resolution Transmission Electron Microscopy (HRTEM). The structural properties have been correlated with the magnetic

response, studied by means of the applied magnetic field and temperature dependencies of the magnetization.

SELECTED RESULTS

In this line, 5 researchers are involved, besides 1 postdoc and 1 PhD student. We have published papers and got 1 invited talks during 2014.

Nanostructured thin films and multilayers

Novel magnetic and electrical properties arise as dissimilar materials get into contact. Magnetic coupling and proximity effects yield exceptional features in thin films and multilayers. Moreover, geometric constrictions in patterned nanostructures induce unique spin configurations that are not possible in continuous films. We have investigated these effects in the following systems:

Antiferromagnet/Ferromagnet (AFM/FM)

- The exchange bias phenomenon (EB) mediated by a non magnetic spacer is currently being investigated in FeF2/spacer/Ni. Wedge-shaped samples for several spacers (Cu, Ag, Au, etc.) were deposited by electron beam evaporation. Surprising preliminary results were obtained, even for relative thick spacers.
- Competing magnetic correlation lengths were studied in FeF2/Ni for very thin Ni layers. X-ray magnetic circular dichroism and photoemission electron microscopy demonstrated that the AFM domain structure strongly depends on the competition between the AFM and FM length scales, which can be controlled with the FM thickness.
- Polycrystalline and epitaxial IrMn/FeCo bilayers were fabricated by sputtering and molecular beam epitaxy techniques, respectively. Different structural order yields a quite different magnetic behaviour in these two systems.
- Exchange bias of FeF2/FeNi bilayers presents a strong deviation from the well-known inverse proportionality law of EB with the FM thickness. This is due to the internal spin configuration in the FM layer. We have performed polarized neutron reflectivity (PNR) measurements to analyze the internal FeNi spin structure and its evolution with the temperature.

Perpendicular magnetic anisotropy (PMA)

- The thermal behaviour of Co/Ni multilayers with weak PMA was analyzed at room and low temperature by magnetometry measurements.

Patterned nanostructures.

- Circular dots of FeF2/FM (FM = Ni, NiFe) were patterned by electron beam lithography. This geometry will allow to investigate the competition between spin configurations induced by constraining effects in circular dots and the exchange coupling at the AFM/FM interface.
- Planar Hall effect and anisotropic magnetoresistance were explored in micrometer cross-shaped FeF2/Ni. Electrical measurements proved a method to determine the chirality of helical spin configurations in nanostructures. A theoretical calculation accounts for the experimental results.
- Circular and elliptical nanostructures of polycrystalline IrMn/FeCo were patterned by electron beam lithography to study the shape anisotropy effect on exchange bias.
- A positive tone photoresist was used to fabricate detachable nanostructures by interference lithography. However, this process is still challenging due to the resist hardening in oxygen plasma and the solvents required for the lift-off.

SELECTED RESULTS

1. "Antiferromagnetic/Ferromagnetic Nanostructures for Multidigit Storage Units", R. Morales, M. Kovylina, Ivan K. Schuller, A. Labarta, and X. Batlle. Applied Physics Letters 104, 032401 (2014).

We have explored three different fabrication routes for patterning Permalloy (Py) nanodiscs with magnetic vortex state, suitable for bio-medical applications: using templates of self-assembled latex spheres, filling nanoporous alumina films and using the technique denominated Hole-Mask Colloidal Lithography (HCL). The comparative analysis of the easiness, capabilities and results obtained leaded us to select HCL technique as the most promising. Intensive laboratory work in the clean room to determine the optimum preparation conditions has allowed us to successfully obtain Py nanodiscs with different dimensions in quantities suitable to

perform biomedical assays. According with what is expected, the nanodiscs present a magnetic vortex state depending on their aspect ratio. Discs between 30 to 50 nm thick and either 70 or 150 nm in diameter displays a cuasi-nominal vortex state, whereas thinner (<25 nm) discs present a mono-domain magnetic structure.

These just obtained results will be published shortly (manuscript in preparation). Preliminary results has been presented in selected conferences (ZING, DCINMA, Nanolito, and ICMAST).

Advanced Functional Materials

New materials with outstanding properties are continuously appearing (see for instance graphene) in all fields of activity. There is impossible to take care of all of them, but some representative fields of research are already running and will be developed in the first stages of the BCMaterials activity. Those are:

For Energy

This research line is focused on finding and design new materials capable of improving the performance of energy generation conversion and storage systems as chemical batteries (Li and Na-ion), fuel cells (SOFC, ITSOFC) or photovoltaic cells, as well as electromagnetic generators and motors. For this purpose, hybrid organic-inorganic compounds, metallic alloys and nanostructured composites are prepared by using low cost and ecoefficient synthesis processes.

WORK HAS BEEN CARRIED OUT IN THE FOLLOWING SUBJECTS

Three perovskites, LSFMC ((La0.8Sr0.2)0.95Fe0.6Mn0.3Co0.103), LNF (LaNi0.6Fe0.403-d) and LNC (LaNi0.6Co0.403-d) has been investigated as contact materials for IT-SOFC systems. The obtained contact resistance values are strongly influenced by the conductivity of the selected contact material, showing the lowest ASR (area specific resistance) for the {Crofer22APU/LNC/LSF} system. The stability of this system after treating at 800°C for 1000 h has been characterized using X-ray micro-diffraction (XRMD), scanning electron microscope equipped with an energy dispersive X-ray analyzer (SEM-EDX) and X-ray photoelectron spectroscopy (XPS)

techniques, showing that the LNC is a promising contact material for IT-SOFC.

A series of titanium perovskites with the general formula Li0.30(La0.50Ln0.50)0.567TiO3 (Ln=La, Pr,Nd) have been prepared by the glycine-nitrate method under different conditions in order to be evaluated as solid electrolyte for their application in lithium ion batteries. No secondary phases have been detected between these compounds and commercial cathode material (LiMn2O4) analysed by TDX measurements within a 30-1100°C temperature range. Slightly higher values of conductivity have been obtained for quenched samples and as the lanthanide size decreases.

The performance of MnV2O6 and its [{Mn(Bpy)}(VO3)2]·(H2O)1.16 and [{Mn(Bpy)0.5}(VO3)2]≈(H2O)0.62 hybrid derivative compounds was investigated against sodium and lithium counter electrodes. For MnV2O6 stable capacities of 850 mAh/g were achieved in lithium cells, the best value reported so far. The difference between Li and Na cell capacity most probably comes from the difference of standard potential of the two redox couples Li+/Li and Na+/Na of about ca. 0.3 V leading to an incomplete conversion reaction and thus lowers capacity in the case of Na cells.

SELECTED RESULTS

1. Authors: A. Morán-Ruiz, K. Vidal, A. Larrañaga, M. I. Arriortua.
   Title: Effects of using (La0.8Sr0.2)0.95Fe0.6Mn0.3Co0.1O3 (LSFMC), LaNi0.6Fe0.4O3_d (LNF) and LaNi0.6Co0.4O3_d (LNC) as contact materials on solid oxide fuel cells.
   Journal: J. Power Sources, 248, 1067-1076 (2014)

2. Authors: A. Morán-Ruiz, K. Vidal, A. Larrañaga, M. A. Laguna-Bercero, J. M. Porras-Vázquez, P. R. Slater, M. I. Arriortua.
   Title: LaNi0.6Co0.4O3-d (LNC) dip-coated on Fe-Cr mesh as a composite cathode contact material on intermediate solid oxide fuel cells (IT-SOFCs).
   Journal: J. Power Source, 289, 509-519 (2014)

3. Authors: K. Vidal, L. Ortega-San-Martín, A. Larrañaga, R.I. Merino, A. Orera, M.I. Arriortua.
   Title: Effects of synthetic conditions on the structural, stability and ion conducting properties of Li0.30(La0.50Ln0.50)0.567TiO3 (Ln= La, Pr, Nd) solid

electrolytes for rechargeable lithium batteries.
Journal: Ceram. Int., 40, 8761-8768 (2014)

4. Authors: R. Fernández de Luis, A. Ponrouch, R. Palacín, M. K. Urtiaga, M. I. Arriortua.
Title: Electrochemical behavior of [{Mn(Bpy)}(VO3)2]≈(H2O)1.24 and [{Mn(Bpy)0.5}(VO3)2]≈(H2O)0.62 inorganic-organic Brannerites in lithium and sodium cells.
Journal: J. Solid State Chem., 212, 92-98 (2014)

In the field of Na-ion batteries we have worked on sodium vanadium fluorophosphates, which are considered very promising cathodes but need further studies to elucidate their electrochemical and structural behaviour. As the sodium carries the charge of this material during battery function it is important to know how it behaves crystallographically in order to better understand its evolution and thus try to design materials with, for example bigger crystallographic voids to accommodate more sodium. For this purpose in situ characterization of sodium-ion batteries are paramount. In this sense, we have determined in real-time during electrochemical cycling the reaction mechanism, lattice parameters, unit-cell volume, and sodium evolution of different Na3V2O2x(PO4)2F3-2x cathodes in fully functioning sodium-ion batteries. We have found that lattice and volume evolution is clearly dependent on the Na insertion/extraction mechanism, the sodium occupancy and distribution amongst the two crystallographic sites, and the electrochemical cycling history. Moreover, it has been proved that Na swaps from one site to the other during cycling, i.e. sodium site occupancy and mobility in the tunnels is interchangeable and fluid, a favourable characteristic for a cathode in a sodium-ion battery.

We have also continued our research to improve the properties of LiMn2O4 spinels as cathodes in Li-ion batteries. Due to its rapid Li extraction/insertion processes, low cost, low toxicity and safety characteristics, these materials are specially interesting for HEVs and EVs. However, some serious shortcomings remain to be solved, especially with regard to capacity decay. The capacity fading is mainly attributed to the Jahn-Teller distortion of the Mn is in the trivalent oxidation state and the progressive dissolution of Mn(III) in the Electrolyte. To address these problems, we have developed two different strategies: 1) to substitute a small portion of the Mn ions with divalent or trivalent cations, 2) to substitute a small amount of Mn(IV) by a tetravalent dopant.

The first solution decreases the amount of Mn(III) in the structure, and lessens the probability of producing a Jahn-Teller distorted tetragonal phase. The second strategy contributes to stabilize the spinel framework without decreasing the amount of the electroactive Mn(III) catión.

Finally, our studies revealed that both types of ion substitutions improve the stability of the bulk material but it is also necessary to minimize the amount of the active material-electrolyte interface for better cyclability. In fact, the concurrence of both differently substituted spinels in the electrode material has a synergistic effect favoring the electrochemical response of the cathode composite.

SELECTED RESULTS

1. "Modification of the electrochemical activity of LiMn1.95SiO.05O4 spinel via addition of phases with different physico-chemical properties". A. Iturrondobeitia, A. Goñi, L. Lezama, C. Kim, M. Doeff, J. Cabana and T. Rojo; Journal of Materials Chemistry A, (2014), 2, 3216-3222.
2. "Sodium Distribution and Reaction Mechanisms of a Na3V2O2(PO4)2F Electrode during Use in a Sodium-Ion Battery". N. Sharma, P. Serras, V. Palomares, H. E. A. Brand, J. Alonso, P. Kubiak, M. L. Fdez-Gubieda, and T. Rojo; Chemistry of Materials, (2014), 26, 3391-3402

A new field of High energy Permanent Magnets has started during 2014, by a collaboration with the University of Delaware (Prof. George Hadjipanayis), aiming to produce high coercivity Dy-free Nd-Fe-B magnets and explore ways to fabricate anisotropic Fe-Co(Ni) and MnBi alloy powders with high coercivity and magnetization that can be used for the development of rare earth-free high performance permanent magnets. This topic is a hot one as regards the substitution of critical raw materials, like rare earths, by other more abundant and available on the European Union or other western countries.

Work developed during 2014 has focused on grain boundary engineering of Nd-Fe-B magnets. Samples with composition Nd12Fe81B6Nb1 and Nd10Fe84B6 were melt-spun into amorphous ribbons and then nanocrystallized by annealing at temperatures in the range of 650-700 oC to obtain the optimal nanostructure for high coercivity. Optimally annealed samples were used for diffusion

experiments with Pr68Cu32 eutectic alloys that provide a boundary layer in the nanograined magnets and increased the coercivity to above 11 kOe (1.1 Tesla)

Preliminary results were presented in the Workshop on Rare Earth Permanent Magnets 2014, Annapolis, MD, USA; 17-22 August 2014, and a more elaborated work has been accepted for oral presentation at the 144th TMS annual meeting and exhibition, to take place in Orlando, FL, USA, in March 14-19, 2015.

People involved in this area are: 5 senior researchers, 2 postdocs, and 2 PhD students. There exist collaborations with the University of Birmingham, the University of New South Wales, the University of Delaware and the Lawrence Berkeley National Laboratory. 10 papers have been published in high impact scientific journals during 2014.

For Sensors

Approach to molecular sensors

Work has been carried out in the following subjects: The synthesis and characterization of nano-structured silver vanadium oxides hydrogels have been carried out by different techniques. These interesting materials are formed by micrometric length ribbons decorated with silver nano-particles. The hydrogels can be easily processed as aerogels or film dried samples. Moreover, the abortion experiments against several dyes reveals absorption capacities as high as 600mgr of dye per gr of adsorbent.

In a second approach to develop molecular sensors, high porous metal organic frameworks based on the azide ligand has been synthesized. This coordination network contains two removable tetrachloroethane molecules within its pores, approximately the 40% of formula weight. Future work will be focused on the absorption properties of this material.

SELECTED RESULTS

1. Authors: R. Fernández de Luis, J. Orive, E.S. Larrea, M.K. Urtiaga, M. I. Arriortua.
   Title: Hybrid Vanadates Constructed from Extended Metal Organic Arrays. Crystal Architectures and Properties.
   Journal: CrystEngComm., 16(45), 10332-10366 (2014)

2. Authors: R. Fernández de Luis, J. Orive, E.S. Larrea, M.K. Urtiaga, M. I. Arriortua.
   Title: Reversible solid-state transformation in {Ni2(H2O)2(Bpa)2}(V6O17) proved by synchrotron radiation: color and magnetic properties change.
   Journal: Cryst. Growth and Desing., 14(2), 658-670 (2014)

3. Authors: F. Llano-Tomé, B. Bazán, M.K. Urtiaga, G. Barandika, M.I. Arriortua.
   Title: Herringbone planes in CuII-PDC-bpe systems based on three-connected nodes: mapping this type of compounds.
   Journal: CrystEngComm., 16, 8762-8735 (2014)

4. Authors: A. Fidalgo-Marijuan, G. Barandika, B. Bazán, M.K. Urtiaga, E. Serrano-Larrea, M. Iglesias, M.I. Arriortua.
   Title: Biomimetic catalysts based on metalloporphyrin MOFs.
   Journal: J. Biol. Inorg. Chem., 19, S864-S864 (2014)

5. Authors: R. Fernández de Luis, A. Ponrouch, R. Palacín, M. K. Urtiaga, M. I. Arriortua.
   Title: Electrochemical behavior of [{Mn(Bpy)}(VO3)2]≈(H2O)1.24 and [{Mn(Bpy)0.5}(VO3)2]≈(H2O)0.62 inorganic-organic Brannerites in lithium and sodium cells.
   Journal: J. Solid State Chem., 212, 92-98 (2014)

Magnetic functional materials

Magnetic functional materials for sensors include soft magnetic materials in different configurations (thin films, multilayers, amorphous ribbons) for high performance magnetic sensors based on magneto-impedance and other coupled phenomena (as magnetoelasticity and/or magneto-electric). Also magnetic polymer/nanoparticles composites of special interest for protection and enhancement of functional parameters of sensors in the form of films or multilayers are studied.

Magnetoelectric and multiferroic composites

Work has been carried out in the following subjects:

- Magnetoelectric response in devices with the shape of laminated composites: Fe59Co16Si15B10 / PVDF / Fe59Co16Si15B10 has been analysed. Laminates with 4, 3, 2, 1 and 0,5 cm lengths show a working resonant frequency of 56, 76, 113, 229 and 402 kHz , respectively. While for the longest device we obtained a field

sensitivity of 5200 V/T (that translates to a sensitivity of 200 pT), for the 0,5 cm shortest one we still have obtained 475 V/T , equivalent to 2 nT magnetic field sensitivity. The results obtained for the smallest magnetoelectric laminate are of special relevance since the working frequency already locates at the LFR (Low Radio Frequency range), allowing for the development of devices working in near-field communication systems or under environmental electromagnetic radiation conditions.

- Nanoparticle dispersion is assumed to be one of the key factors in composites performance and, in particular, in improving magnetoelectric (ME) coupling in polymer composites. In this way, the dispersion of cobalt ferrite ($CoFe_2O_4$) nanoparticles in poly(vinylidene fluoride)-trifluorethylene, P(VDF-TrFE), matrix and its role in the piezoelectric, magnetic and magnetoelectric properties of the nanocomposite has been studied by preparing samples via two alternative dispersion routes: ultrasound and citric acid nanoparticle surfactation. No substantial differences have been detected in the ferroelectric, piezoelectric, magnetic and magnetoelectric response of samples prepared with and without surfactants, leading to a simplified large-scale production.

SELECTED RESULTS

1. Radiofrequency magnetoelastic resonators for magnetoelectric applications, A. Lasheras, J. Gutiérrez, A. Balza, J.M. Barandiarán and A. Rodríguez Pierna, Journal of Physics D: Applied Physics 47 , pp.315003-1 (7 pages)
2. Effect of the filler dispersion and dispersion method on the piezoelectric and magnetoelectric response of $CoFe_2O_4$/P(VDF-TrFE) nanocomposites, P. Martins, R. Gonçalves, S. Lanceros-Méndez, A. Lasheras, J. Gutiérrez and J.M. Barandiarán, Applied Surface Science 313, pp. 215-219

Magnetoimpedance based devices

A new record in performance has been established for thin-film based MI structures: 350 % of impedance ratio and 300 %/Oe field sensitivity. This has been achieved by combining in a new series of multilayered sandwiched structures the knowledge gained in previous years about material properties and the influence of the geometry on the sensitivity.

GMI element surrounded by a micro-patterned coil for developing sensors based on the off-diagonal MI effect.

From the point of view of sensor development, we have established the process flow and parameters for fabricating micro-patterned off-diagonal GMI sensors based on multilayered Permalloy (Py) sandwiched structures surrounded by a pick-up coil (see figure). Intense research is being performed to optimize the properties of the insulating layers between the coil and the GMI samples. They need to be prepared at low temperature (below 300°C) to avoid depredating the magnetic properties of the Permalloy-based multilayered structure. Sputtered SiO2 and Al2O3 layers, deposited by RF-sputtering do not provide the needed electrical insulation. Research has begun to grow Al2O3 layers from anodization of Al layer either by electrochemical or Reactive ion etching process.

SELECTED RESULTS

1. Comparative study of magnetic, microwave properties and giant magnetoimpedance of FeNi-based multilayers with different structure, G.V. Kurlyandskaya, E. Fernández, A. García-Arribas, V.N. Lepalovsij, and S.O. Volchkov Journal of Alloys and Compounds 615 (2014) S296–S299
2. Magnetoimpedance of thin film meander with composite coating layer containing Ni nanoparticles, K. J. Lodewijk, E. Fernández, A. García-Arribas, G. V. Kurlyandskaya, V. N. Lepalovskij, A. P. Safronov, and B. J. Kooi Journal of Applied Physics 115, 17A323 (2014)

This line includes 4 main researchers, 1 postdocs and 1 PhD student. Has published 16 papers (6 are Q1 qualification) and got 2 Invited + 9 Contributed talks during 2013 and 2014.

AGREEMENTS WITH SPANISH AND FOREIGN RESEARCH INSTITUTIONS

The most important agreement during 2014 is:

Karlsruhe Institute of Technology.
Institute of Microstructure Technology (Prof. Manfred Kolh)

"Development of FSMA-based actuators for nanoscale applications" (2014 – 2016)

ID: 2014-012-004332Project applications during 2013

RESEARCH PROJECTS

On going Project during 2014

Development of Dy-free and Rare Earth-free High Energy Magnets Using Nanotechnology

Reference: AYD-000-195
Partners: UNIVERSITY OF DELAWARE. DPT. OF PHYSICS / BCMaterials
Visitor Researcher: PROF. GEORGE HADJIPANAYIS
Financial Backers: BIZKAIA:TALENT
Awarding program: PROGRAMA DE AYUDAS A LA INVESTIGACIÓN
Awarding period: 2014-2015 Starting year: 2014

SUMMARY

The project presents viable and innovative approaches to produce high coercivity Dy-free Nd-Fe-B magnets and explore ways to fabricate for the first time anisotropic Fe-Co(Ni) and MnBi alloy powders with high coercivity and magnetization that can be used for the development of rare earth-free high performance permanent magnets. High coercivity Dyfree magnets will be synthesized by using die-upset nanocrystalline ribbons and Nd2Fe14B nanoparticles. Liquid chemistry procedures will be used to synthesize the Fe-Co(Ni) nanoparticles in the tetragonal (L10) or hexagonal (L12) phase, and MnBi nanoparticles in the high anisotropy hexagonal LTP phase. If Fe-Co alloys can be synthesized

with the anisotropic L1O structure, they can have an energy product three times higher than that of current Nd-Fe-B. The development of new Dy-free and rare earth free high energy magnets will lead to lower cost more efficient energy and power dense devices and most importantly it will result in a substantial reduction in our dependence upon the critical rare earth elements in permanent magnets.

Polymer/Polyoxometalate Smart Coatings for Chemical and Biological Applications (POCHBA)

Reference: MAT 2013-48366-C2
Partners: UNIVERSIDAD DEL PAÍS VASCO / EUSKAL HERRIKO UNIBERTSITATEA - UPV/EHU / BCMaterials
Financial Backers: MINISTERIO DE ECONOMÍA Y COMPETITIVIDAD - GOBIERNO DE ESPAÑA
Awarding program: PLAN ESTATAL DE INVESTIGACIÓN CIENTÍFICA Y TÉCNICA Y DE INNOVACIÓN – PROGRAMA DE FOMENTO DE LA ICT DE EXCELENCIA. SUBPROGRAMA DE GENERACIÓN DE CONOCIMIENTO
Awarding period: 2014 - 2016 Starting year: 2014

SUMMARY

Combination of organic and inorganic components can lead to hybrid materials with unusual structures and properties originating from synergism between fractions. The appropriate choice of components can allow the resulting material to be processed as smart, stimuli-responsive coatings for applications as sensors-actuators in microelectronics, electrooptics and/or biotechnology, among others.

In this project we propose to prepare smart hybrid surfaces using polymers able to immobilize polyoxometalates (POMs).

Such surfaces will be synthetized onto magnetic substrates, either nanoparticles or magnetoelastic resonators, that allow to contactless monitor the system about its mass, so it will be possible to know the evolution of the surface during the synthesis as well as in the interaction with other molecules/systems and evaluate, therefore, their performance as sensors.

Hybrid materials will be prepared by anchoring POMs onto smart functionalized polymer surfaces having inter-polymer complexes. We

pretend to prepare surfaces and coatings based on polystyrene and an amphiphilic diblock copolymer:

PS-b-PX (PX= PEG, PLys or PAA). These polymeric surfaces can be formed or destroyed depending on environmental conditions and interact with other polymers or chemical species, and in particular, with POMs. Those are anionic metaloxo clusters that have been regarded as remarkable inorganic building blocks because of their multifunctional nature and have the possibility of anchoring on the above polymeric surfaces either through the coordination sphere of one of their metals, or through formation of extensive hydrogen-bonding networks.

The so prepared devices will be tested as drug delivery systems (isoPOMs grafted on PX = PAA or PLys functions) or as chemical sensors (heteroPOMs grafted on PX = PAA, PLys or PEG functions) after deposited onto Magnetic Nanoparticles or Magnetoelastic cantilever resonators, that act as vectors or detectors of the specific actuation respectively. The final aim of the project is to develop a demonstrator of a sensor based on these compounds.

Development of smart materials, functional materials and materials for advanced processes (ACTIMAT)

Reference: IE13-380
Partners: UNIVERSIDAD DEL PAÍS VASCO / EUSKAL HERRIKO UNIBERTSITATEA - UPV/EHU / IK4 / BASQUE CENTER FOR MATERIALS, APPLICATIONS AND NANOSTRUCTURES / MAIER TECHNOLOGY CENTRE, S. COOP / TECNALIA / MONDRAGON GOI ESKOLA POLITEKNIKOA S. COOP. / CENTRO DE TECNOLOGÍAS AERONÁUTICAS
Financial Backers: DEPARTAMENTO DE DESARROLLO ECONÓMICO Y COMPETITIVIDAD – GOBIERNO VASCO
Awarding program: ETORTEK. PROYECTOS DE INVESTIGACIÓN ESTRATÉGICA
Awarding period: 2013-2014 Starting year: 2013

SUMMARY

The new ACTIMAT project 2013 – 2014 is a continuation of the former road map of strategic research in materials established in 2010, but has undergone a drastic revision aiming to get more concrete, concentrate and focused. In 2013-14 continuity was kept in:

- Development of Smart Materials. Centred in the development of materials, while devices, like sensors or actuators are derived to other fields of strategic areas.
- Development of Functional Materials. As in the previous area, the project is centred on materials development, characterization and performance, leaving other aspects, as design and transformation, to other research projects.

New Proposals during 2014

Novel magnetic nanostructures for biomedical applications (Accorded)

Reference: AYD-000-266
Partners: FUNCTIONAL MATERIALS LABORATORY, UNIVERSITY OF SOUTH FLORIDA (TAMPA, FLORIDA, USA) / BCMaterials
Visitor Researcher: PROF. HARIHARAN SRIKANTH
Financial Backers: BIZKAIA:TALENT
Awarding program: PROGRAMA DE AYUDAS A LA INVESTIGACIÓN
Awarding period: 2015-2016 Starting year: 2015

SUMMARY

This project presents viable and innovative approaches to produce novel magnetic nanoparticles with improved properties that overcome the limitations in commonly employed Fe-oxide nanoparticles used in nanomedicine. We will focus on exploiting Au/Fe-oxide and Fe(FeCo)/Fe-oxide nanoparticles with different morphologies of core/shell, hollow, and dumbbell, and tuned aspect ratio for use in hyperthermia and drug delivery. High quality nanoparticles with controlled shape, size, and morphology will be synthesized, using non-hydrolytic synthesis methods, and characterized at the Functional Materials Laboratory (Tampa, Florida, USA), which will then be functionalized and tested for biomedical applications at the BCMaterials (Bilbao, Spain). Our combined efforts will help accelerate the practical use of these functional magnetic nanoparticles as an inexpensive cutting-edge nanotechnology tool in the emergent era of personalized medicine.

Cost-effective magnetoelastic resonance system for improved clinical diagnostics: biomarkers in emergency services

Call: H2020-PHC-2014 -TWO-STAGE- SECOND STAGE
Topic: PHC-10-2014 Type of action: RIA
Proposal acronym: MERIADOC
Partners: GAIKER , BCMaterials (UPV/EHU), VAC, ACREO, VTT, OSAKIDETZA, MKFLUID

Magnetic Microtools for Cell Therapy and Regenerative Medicine

Call: H2020-PHC-2015 -TWO-STAGE-
Topic: PHC-16-2015 Type of action: RIA
Proposal number: 667064-1
Proposal acronym: MAGCELTHER
Partners: BCMaterials (UPV/EHU), ACHUCARRO (UPV/EHU), INSTITUTE OF PHYSICS (ASCR), INSTITUTE OF EXP. MEDICINE (ASCR), NEEL INSTITUTE (CNRS), DEPT. OF BIO-MEDICAL ENG (UNIVERSITY OF FLORIDA), HISTOCELL, RESONANT CIRCUIT LTD., RHEFOR GmdH.

Assembly and test of Symmetric Solid Oxide Fuel Cells

Call: H2020-MSCA-IF-2014
Topic: MSCA-IF-2014-EF Type of Action: MSCA-IF-EF-ST
Proposal Acronym: SYMFCT

Magnetic biosensors based on smart surfaces

Call: H2020-MSCA-IF-2014
Topic: MSCA-IF-2014-EF Type of Action: MSCA-IF-EF-ST
Proposal Acronym: MABIOSS

Nanopatterned surfaces for sensors based on magnetic nanoparticles

Call: H2020-MSCA-IF-2014
Topic: MSCA-IF-2014-GF Type of Action: MSCA-IF-EF-ST
Proposal Acronym: NANOSMAG

Nanopatterned surfaces for sensors based on magnetic Functional properties and non-equilibrium processes in shape memory alloys and related ferroic materials

Call: H2020-MSCA-IF-2014
Reference: MAT2014-56116-C4
Acronym: FUNSAFE
Funding Body: MINISTERIO DE ECONOMÍA Y COMPETITIVIDAD - GOBIERNO DE ESPAÑA
Awarding program: PROGRAMA ESTATAL DE I+D+I ORIENTADA A LOS RETOS DE LA SOCIEDAD.
Partners: UNIV. ILLES BALEARS (COORDINATOR), UPV/EHU, BCMaterials, UNIV. OF OVIEDO

BCMaterials 2014

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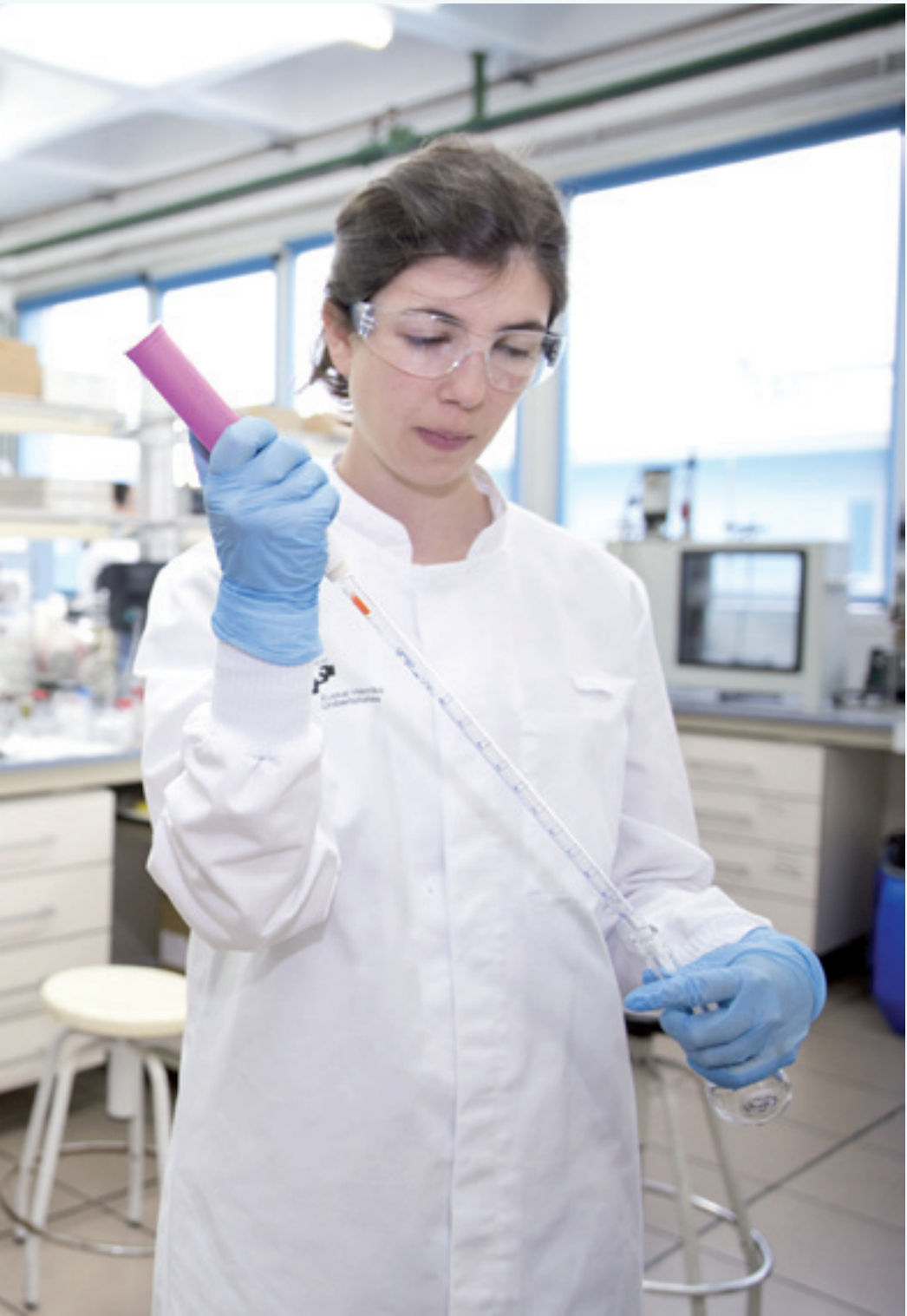
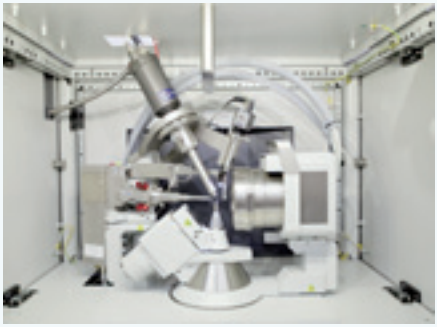
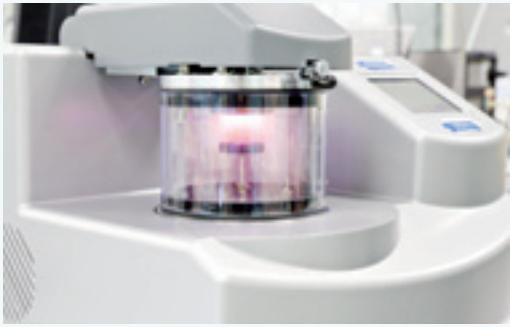
BCMaterials 2014

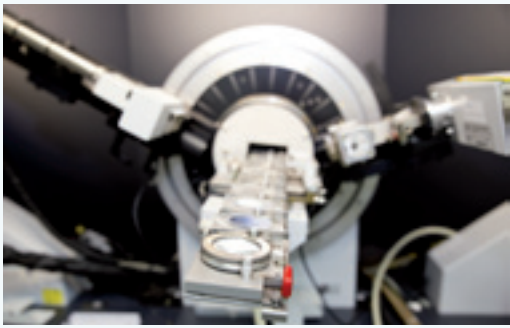
FACILITIES

"THE INTERNATIONAL QUALITY RESEARCH IN THE AREA OF MATERIAL SCIENCE IS ONE OF THE KEY STRATEGIC PRIORITIES OF EUROPEAN, SPANISH AND BASQUE RESEARCH STRATEGIES"









DISSEMINATION OF RESULTS

In order to make the center visible to the local research community and to present it in other audiences, a number of talks, seminars and conferences have been organized. In addition we tried to participate in conferences already organized by other institutions by organizing symposia within them or promoting the presence of BCMaterials members in the Committee.

PUBLICATIONS

I.

Cation-Directed Dimeric versus Tetrameric Assemblies of Lanthanide-Stabilized Dilacunary Keggin Tungstogermanates.

B. Artetxe, S. Reinoso, L. San Felices, L. Lezama, J M. Gutiérrez-Zorrilla, J. Á. García, J. R. Galán-Mascarós, A. Haider, U. Kortz and C. Vicent.

Chemistry – A European Journal, 2014, 20 (38), 12144–12156

II.

Magnetic Properties and Magnetic Entropy Change in Gd/Ti Multilayers.

A. Svalov V. O. Vas'kovskiy, K. G. Balymov, J. Alonso M. L. Fernández-Gubieda Galina V. Kurlyandskaya.

IEEE Transactions on Magnetics, 2014, 50 (11)

III.

Magnetic moment distribution modeling in non stoichiometric Ni-Mn-Ga ferromagnetic shape memory alloys.

P. Lázpita, J. Gutierrez, J. M. Barandiarán, V. Chernenko C. Mondelli, L. Chapon.

Journal of Physics: Conference Series, 2014, 549, 012016

IV.

Thermal behaviour of H-bonded interpolymer complexes based on polymers with acrylamide or lactame groups and poly(acrylic acid): Influence of N-alkyl and a-methyl substitutions.

L. Ruiz-Rubio, J. L. Vilas, M. Rodríguez, L. M. León

Polymer Degradation and Stability, 2014, 109, 147–153

V.

Comparative study of magnetic, microwave properties and giant magnetoimpedance of FeNi-based multilayers with different structure.

G. V. Kurlyandskaya E Fernández, A. García-Arribas, V. N. Lepalovskij, S. O. Volchkov.

Journal of Alloys and Compounds, 2014, 615 (1), S296-S299

VI.

Tailoring the Exchange Bias in FeNi/FeMn Bilayers by Heat Treatment and FeMn Surface Oxidation.

A. Svalov, P. A. Savin V. N. Lepalovskij A. Larrañaga V. O. Vas'kovskiy A. García-Arribas Galina V. Kurlyandskaya.

IEEE Transactions on Magnetics, 2014, 50, 4

VII.

Hybrid vanadates constructed from extended metal-organic arrays: crystal architectures and properties.

R. Fernández, J. Orive, E. S. Larrea, M.-K. Urtiaga, M. I. Arriortua.

CrystEngComm, 2014, 16, 10332–10366

VIII.

Synthesis and characterization of novel piezoelectric nitrile copolyimide films for high temperature sensor applications.

A. Maceiras, P. Martins, M. San Sebastián, A Lasheras, M. Silva J.M. Laza, J. L. Vilas, J. Gutierrez S. Lanceros-Mendez, J. M. Barandiarán, L. M. León.

Smart Materials and Structures, 2014, 23 (10), 105015

IX.
X phase of MnWO4.
I. Urcelay, E. Ressouche, A. A. Mukhin, V. Yu. Ivanov A. M. Kadomtseva, Yu. F. Popov, G. P. Vorob'ev, A. M. Balbashov, J. L. García-Muñoz, V. Skumryev.
Physical Review B, 2014, 90, 024408

X.
Radiofrequency magnetoelastic resonators for magnetoelectric applications.
A. Lasheras J. Gutierrez A. Balza J.M. Barandiarán A. Rodríguez Pierna.
Journal of Physics D: Applied Physics, 2014, 47, 315003

XI.
A Novel Micro- and Nano-Scale Positioning Sensor Based on Radio Frequency Resonant Cavities.
E. Asua V. Etxebarria A. García-Arribas J. Feuchtwanger J. Portilla, J. Lucas.
Sensors (MDPI), 2014, 14 (6), 9615-9627

XII.
Effect of filler dispersion and dispersion method on the piezoelectricand magnetoelectric response of CoFe2O4/ P(VDF-TrFE)nanocomposites.
P. Martins R. Gonçalves S. Lanceros-Mendez A. Lasheras J. Gutierrez, J. M. Barandiarán.
Applied Surface Science, 2014, 313, (215-219)

XIII.
Reversible Dehydration in Polyoxometalate-Based Hybrid Compounds: A Study of Single-Crystal to Single-Crystal Transformations in Keggin-Type Germanotungstates Decorated with Copper(II) Complexes of Tetradentate N-Donor Ligands.
A. Iturrospe , L. San Felices, S. Reinoso B. Artetxe, L. Lezama, J. M. Gutiérrez-Zorrilla.
Crystal Growth & Design, 2014, 14 (5), 2318–2328

XIV.
Polymer–polymer complexes of poly(N-isopropylacrylamide) and poly(N,N-diethylacrylamide) with poly(carboxylic acids): a comparative study.
L. Ruiz-Rubio J.M. Laza, L. Pérez, N. Rioja, E. Bilbao.
Colloid and Polymer Science, 2014, 292 (2), 423–430

XV.
Sodium Distribution and Reaction Mechanisms of a Na3V2O2(PO4)2F Electrode during Use in a Sodium-Ion Battery.
N. Sharma P. Serras,V. Palomares H. E. A. Brand, J. Alonso P. Kubiak M. L. Fernández-Gubieda, T. Rojo.
ACS Chemistry of Materials, 2014, 26 (11), 3391–3402

XVI.
First-principles calculation of the instability leading to giant inverse magnetocaloric effects.
D. Comtesse M. E. Gruner M. Ogura V. V. Sokolovskiy V. D. Buchelnikov A. Grünebohm R. Arróyave N. Singh T. Gottschall O. Gutfleisch, V. Chernenko F. Albertini S. Fähler P. Entel.
Physical Review B, 2014, 89, 184403

XVII.
Study of the effect of gamma irradiation on a commercial polycyclooctene I. Thermal and mechanical properties.
N. García J.M. Laza, J.M. Cuevas, J. L. Vilas E. Bilbao, L. M. León.
Radiation Physics and Chemistry, 2014, 102, 108–116

XVIII.

Hysteretic and anhysteretic tensile stress-strain behavior of Ni-Fe(Co)-Ga single crystal: Experiment and theory.

A.Kosogor V. A. L'vov, V. Chernenko, E. Villa, J. M. Barandiarán, T.Fukuda, T.Terai. T. Kakeshita.

Acta Materialia, 2014, 66, 79–85

XIX.

Pulsed-mode operation and performance of a ferromagnetic shape memory alloy actuator.

E. Asua A. García-Arribas V. Etxebarria J. Feuchtwanger.

Smart Materials and Structures, 2014, 23 (2), 025023

XX.

Magnetic properties of Ni40+xMn39-xSn21 (x = 0, 2, 4, 6 and 8 at.%) Heusler alloys.

P. Lázpita J. M. Barandiarán, V. Chernenko B. Valle García, E. Díaz Tajada T. Lograsso D.L. Schlagel.

Journal of Alloys and Compounds, 2014, 594, 171-174

XXI.

Impact Damping in NiMnGa/Polymer Composites.

J. Feuchtwanger J. Aurrekoetxea J. Zurbitu, J. L. Vilas, L. M. León, J. M. Barandiarán, H. Hosoda V. Chernenko.

Materials Transactions, 2014, 55 (3), 629-632

XXII.

Magnetostructural transition and magnetocaloric effect in MnNiGe1.05 melt-spun ribbons.

G. Daniel-Pérez J. L. Sánchez Llamazares A. Quintana-Nedelcos, P. Álvarez-Alonso R. Varga V. Chernenko.

Journal of Applied Physics, 2014, 115, 17A920

XXIII.

Interplay between microstructure and magnetism in NiO nanoparticles: breakdown of the antiferromagnetic order.

N. Rinaldi-Montes, P. Gorría D. Martínez-Blanco A. B. Fuertes, L. Fernández Barquín, J. Rodríguez Fernández, I. de Pedro M. L. Fernández-Gubieda, J. Alonso L. Olivi G. Aquilanti J. A. Blanco.

Nanoscale, 2014, 6, (457-465)

XXIV.

Martensitic transformation in Ni-Mn-Ga/Si(100) thin films.

I. R. Aseguinolaza, I. Orue, A. Svalov K. Wilson P.Müllner, J. M. Barandiarán, V. Chernenko.

Thin Solid Films, 2014, 558, (449-454)

XXV.

Sensor applications of soft magnetic materials based on magneto-impedance, magneto-elastic resonance and magnetoelectricity.

A. García-Arribas, J. Gutierrez, G. V. Kurlyandskaya, J.M. Barandiarán, A. Svalov, E. Fernández A. Lasheras, D. de Cos, I. Bravo-Imaz.

Sensors (MDPI), 2014, 14 (5), (7602-7624)

XVI.

CuII–PDC-bpe frameworks (PDC = 2,5-pyridinedicarboxylate, bpe = 1,2-di(4-pyridyl)ethylene): mapping of herringbone-type structures.

F. Llano-Tomé, B. Bazán M.K. Urtiaga, G. Barandika, L. Lezama, M. I. Arriortua.

CrystEngComm, 2014, 16, 8726–8735

XVII.

Fe3O4 nanoparticles prepared by the seeded-growth route for hyperthermia: electron magnetic resonance as a key tool to evaluate size distribution in magnetic nanoparticles.

I. Castellanos-Rubio, M. Insausti, E. Garaio publication-author 3606 F. Plazaola T. Rojo, L. Lezama.

Nanoscale, 2014, 6, (7542-7552)

XVIII.

Shape memory effect for recovering surface damages.

N. García, J.M. Laza, J.M. Cuevas, B. Gonzalo, J. L. Vilas, L. M. León.

Journal of Polymer Research, 2014, 21 (6), 481

XXIX.

Antiferromagnetic/ferromagnetic nanostructures for multidigit storage units.

R. Morales , M. Kovylina , I. K. Schuller, A. Labarta , X. Batlle.

Applied Physics Letters, 2014, 104, 032401

XXX.

A multifrequency electromagnetic applicator with an integrated AC magnetometer for magnetic hyperthermia experiments.

E. Garaio, J. M. Collantes, F. Plazaola, J. A. Garcia, J. Castellanos-Rubio.

Measurements Science & Technology, 2014, 25, 115702

XXXI.

A wide-frequency range AC magnetometer to measure the specific absorption rate in nanoparticles for magnetic hyperthermia.

E. Garaio, J. M. Collantes, J. A. Garcia, F. Plazaola, S. Mornet, F. Couillaud, O. Sandre.

Journal of magnetism and magnetic materials, 2014, 368, 432

XXXII.

Detection of atomic scale changes in the free volume void size of three-dimensional colorectal cancer cell culture using positron annihilation lifetime spectroscopy.

E. Axpe, T. Lopez-Euba, A. Castellanos-Rubio, D. Merida, J. A. Garcia, L. Plaza-Izurieta, N. Fernandez-Jimenez, F. Plazaola, J. R. Bilbao.

PLOS ONE, 2014, 9, e83838

XXXIII.

Vacancy dynamic in Ni-Mn-Ga ferromagnetic shape memory alloys.

D. Merida, J. A. Garcia, V. Sanchez-Alarcos, J. I. Perez-Landazabal, V. Recarte, F. Plazaola.

Applied Physics Letters, 2014, 104, 231905

ORGANIZATION OF CONFERENCES

New Materials for a Better Life!

The third edition of the Workshop on "New Materials for a Better Life!" took place on June the 12th, 2014 at the "Paraninfo" of the Faculty of Science & Technology, UPV/EHU, Leioa Campus, Biscay. This year it was devoted to new materials for sensors and biosensors, covering a large range of applications, lectured by first order speakers from all around the world, including a round table about Sensors for Industry and Society, with researchers, as well as industrial and government representatives.

PROGRAMME

- Plenary Talk: Luis Liz-Marzan (BioMagune, Donostia): "Metal Nanoparticles for Plasmonic Sensing".
- Lecture 1: Senen Lanceros, (Univ. do Minho and International Nanotechnology Laboratory, Braga, Portugal): "Electroactive polymers for sensing".
- Lecture 2: Ioanna Giouroudi (Technical University Wien, Austria): "Magnetic Microfluidics for Biosensors".
- Lecture 3: Alejandro Baeza (Univ. Complutense, Madrid): "Self-propelled Silica nanoparticles for DNA sensing".
- Lecture 4: Marina Díaz Michelena, (Instituto de Tecnología Aero Espacial, INTA, Madrid): "Compact miniaturized aerospace magnetometers".
- Lecture 5: George Hadjipanayis, Department of Physics and Astronomy, Univ. of Delaware, Newark (USA): "Magnets for Energy-related Applications".
- Lecture 6: Kiyonory Suzuki (Monash University, Australia): "Magnetic particles with a hyperthermia switch".
- Lecture 7: Del Atkinson, (Univ. Durham, UK) "Printable sensor materials and technologies".

ROUND TABLE: SENSORS FOR INDUSTRY AND SOCIETY

ESTÍBALIZ HERNÁEZ,
Deputy Minister for Technology, Innovation and Competitiveness.

NURIA GISBERT,
General Manager of CIC Microgune.

FRANCISCO JAVIER CÁCERES,
General Manager of Ineustar, Asociación Española de la Industria de la Ciencia.

JOSÉ MANUEL BARANDIARÁN,
BCMaterials Scientific Director.

FERNANDO PLAZAOLA,
Vice-Rector for Research.

4th International Conference on Materials and Applications for Sensors and Transducers (ICMAST)

Held in Bilbao on June 8-11. Organized by IDAS, BCMaterials and UPV/EHU. Chaired by Jon Gutierrez and Jose Manuel Barandiaran, members of BCMaterials.

The conference aims at creating a forum in the field of materials research and their applications, especially for those materials that are used for sensors and devices that transduce physical properties.

INVITED SPEAKERS

PROF. MASAAKI FUTAMOTO
"Improvement of Magnetic Force Microscope Performance by Tuning the Coating Material of Sensor Tip".
CHUO UNIVERSITY, TOKYO, JAPAN, N.49

PROF. CHRISTOPHE DOLABDJIAN
"Required magnetic material performances for low noise Magnetometer development".
UNIVERSITÉ DE CAEN BASSE-NORMANDIE, CAEN, FRANCE

PROF. JOSE M.G. MERAYO
"Magnetic Materials for Magnetometric Applications"
DTH, COPENHAGEN, DENMARK

PROF. SENÉN LANCEROS MÉNDEZ
"Recent developments and future challenges on piezoelectric polymers and polymer composites for sensor and actuator applications"
UNIVERSIDADE DO MINHO-INL, BRAGA, PORTUGAL

DR. DEL ATKINSON
"*A Printable Pressure Sensitive Conductive Ink for Sensors and Touch Interfaces"*
UNIVERSITY OF DURHAM, GREAT BRITAIN, N.51

DR. GALINA KURLYANDSKAYA
"*Magnetic Materials for Biosensors Applications"*
UPV/EHU, LEIOA, SPAIN

DR. IOANNA GIOUROUDI
"*Biomedical Applications of Magnetic Nanoparticles"*
TECHNICAL UNIVERSITY OF WIEN, WIEN, AUSTRIA

DR. HARVEY AMORÍN
"*Magnetoelectric Ceramic Composites"*
INSITUTO DE CIENCIA MATERIALES-CSIC, MADRID, SPAIN

DR. MARINA DIAZ MITXELENA
"*Compact magnetic sensors for planetary magnetic mineralogy"*
INTA, SPAIN

XXIV Simposio del GE3C. Grupo Especializado de Cristalografía y Crecimiento Cristalino.

Cristalografía y Sostenibilidad, was held in Bilbao on June 23-26 by GE3C, Organized by BC Materials and UPV/EHU. Chaired by Maribel Arriortua member of BC Materials

The conference was devoted to Crystallography and Sustainability.

10th European Conference on Magnetic Sensors and Actuators (EMSA)

The conference was held in Vienna, Austria during July 6-9, 2014. BCMATERIALS contribution consisted in the role of Dr. Alfredo Garcia Arribas as Conference Proceedings Editor, in a special issue of the IEEE Trans on Magn.

SELECTED CONFERENCE CONTRIBUTIONS

1. *"Neutron and synchrotron studies in Magnetic Shape Memory Alloys"*, TMS 2014, San Diego, CA, USA, Feb. 16-20, 2014 (Invited). M Barandiaran
2. *"Growth of Co/Ni Multilayers with Perpendicular Magnetic Anisotropy".* APS-March Meeting 2014. Denver, Colorado (USA). March 3-7, 2014, R Morales
3. *"Structure of Imidazolium-based Ionic Liquids",* American Physical Society Meeting, Denver, CO, USA, 4 March 2014 (Invited) M. L. Saboungi
4. *"Superparamagnetic Nanoplatforms for Theragnostic Applications: a Structural Investigation"* (with I. Milosevic), American Physical Society Meeting, Denver, CO, USA, 5 March 2014 (Invited) M. L. Saboungi
5. *"Magnetic nanoparticles from magnetotactic bacteria: the process of biomineralization"* Nanobiomaterials. Zing Conference, Nerja (Spain) April 2014. (Invited) M. L. Fernández-Gubieda
6. *"Magnetic oxide and metal nanoparticles".* Zing Conference on Nanobiomaterials. 6-9 April 2014, Nerja, Spain. (Invited) M Barandiaran
7. *"Magnetite Nanoparticles from Bacteria".* 4th International conference on superconductivity and magnetism. 29 April-5 May, Antalya, Turkey. (Invited) M Barandiaran
8. *"Thin films of ferromagnetic shape memory materials".* International 4th International conference on superconductivity and magnetism. 29 April-5 May, Antalya, Turkey. (Invited) VA Chernenko
9. *"Topological defects in the magnetic stripe domain pattern as a tool to control magnetization reversal in magnetic lateral multilayers with perpendicular magnetic anisotropy".* IEEE International Magnetics Conference (Intermag14). Dresden, Germany. May 4-8, 2014, R Morales
10. *"Direct observations of spin configurations in exchange-biased Ni/FeF2 nanostructures".* IEEE International Magnetics Conference (Intermag14). Dresden, Germany. May 4-8, 2014, R Morales

11. *"Determination of the out-of-plane anisotropies (first and second order) of amorphous NdCo5 thin films by micromagnetic simulations".* IEEE International Magnetics Conference (Intermag14). Dresden, Germany. May 4-8, 2014, R Morales

12. *"Effects of Water on the Structure and Dynamics of Ionic liquids",* Seminar at J-PARC, Tokai, Japan, 5 June 2014 (Invited) M. L. Saboungi

13. *"Neutron and synchrotron studies of Structure and Magnetism of Shape Memory Alloys".* VII Reunión de la SETN, 22-25 June 2014, Pamplona, Spain. (Invited) M Barandiaran

14. *"Radio-frequency range magnetoelectric effect measured at high temperature",* oral contribution given at the 10th European conference on magnetic sensors and actuators (EMSA), Vienna, Austria, 6-9 July, 2014, A. Lasheras

15. *"Giant tensile superelastic behavior in ferromagnetic shape memory alloys. Experiment and theory".* ICOMAT 2014, July 6-13, Bilbao. (Invited) VA Chernenko

16. *"Fabrication of Patterned Ferromagnetic Shape Memory Thin Films",* XXIII International Materials Research Congress. 17-21 August, 2014, Cancun, Mexico (Invited) VA Chernenko

17. *"Thin Films of Ferromagnetic Shape Memory Materials".* XXIII International Materials Research Congress. 17-21 August, 2014, Cancun, Mexico (Invited) VA Chernenko

18. *"Water and Ionic Liquids",* EMLG - JMLG Annual Meeting 2014, University Roma Tre, Rome, Italy, 10 September 2014 (Invited) M. L. Saboungi

19. *"Biomineralization process of Magnetospirillum gryphiswaldense by X-ray absorption spectroscopy".* 4th International Meeting on Magnetotactic Bacteria. 15-18 September 2014. Centro Brazileiro de las Pesquisas Físicas (Rio de Janeiro - Brazil). (Invited) M. L. Fernández-Gubieda

20. *"Magnetic shape memory materials".* Smart functional materials for shaping our future, 19- 20 September, 2014, Debrecen, Hungary. (Invited) VA Chernenko

21. *"Magnetic Biocompatible Hybrid Nanoparticles",* Nanoandes School, Merida, Venezuela, 22nd October 2014 and NanoSur Conference, Caracas, Venezuela, 29th October 2014 (Invited) M. L. Saboungi

22. *"Magnetization reversal in exchange biased cross junctions revealed by magnetoresistance and planar Hall effect".* 59th Annual Conference on Magnetism and Magnetic Materials (MMM 14). Honolulu, Hawaii (USA). Nov. 3-7, 2014. (Invited) R Morales

23. *"FeCo nanowires for enhanced magnetic hyperthermia",* and *"From core/shell to hollow Fe/γ-Fe2O3 nanoparticles: evolution of the magnetic behaviour".* 59th Annual Magnetism & Magnetic Materials Conference (MMM2014). Honolulu (USA) November 2014, J Alonso

24. *"Ferromagnetic shape memory effect: underlying physics and practical importance",* 2014 Energy, Material and Nanotechnology Fall Meeting, EMN-2014, November 22-25, 2014, Orlando, Florida, USA. (Invited) VA Chernenko

25. *"Magnetization dynamics and collective phenomena in magnetic nanostructures",* EMN Fall Meeting 2014. Orlando (USA) November 2014, J Alonso

PhD THESIS DEFENDED IN 2014

1. Arkaitz Fidalgo. (March 2014) *"MOFs basados en metaloporfirinas: diseño estructural orientado a la biomimetización de sus propiedades naturales",* Outstanding, "Cum Laude"

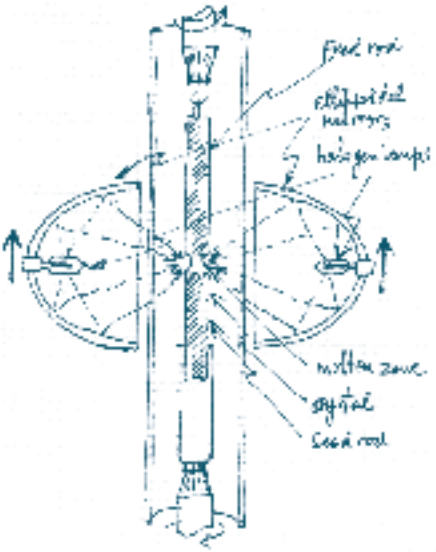
2. Ivan Rodriguez-Aseguinolaza (May 2014). *"Thin Films of Ferromagnetic Shape Memory Alloys",* Outstanding, "Cum Laude" and "International Doctor".

3. Beñat Artetxe. (June 2014). *"Systematic studies on 3d- and 4f-metal containing polyoxometalates suitable for organic derivatization",* Outstanding, "Cum Laude" and "International Doctor"

4. Aroa Pache (June 2014). *"Hybrid compounds based on the interaction between heteropolyoxotungstates and copper(II) complexes of N,O-chelating ligands",* Outstanding, "Cum Laude" and "International Doctor".

5. Paula Serras. (July 2014): *"High voltage cathodes for Na-ION batteries: Na3V2O2x(PO4)2F3-2X system",* Outstanding, "Cum Laude" and "International Doctor".

FLOATING ZONE FURNACE
FOR SINGLE CRYSTAL
GROWTH



IMPURITY CONCENTRATION ALONG THE CRYSTAL

$$C_S(x) = C_0\left(1-(1-k_0)e^{-\frac{k_0 x}{l}}\right)$$

C_0 : initial impurity concentration
k_0 : segregation coeff.
l : zone length

TALKS AND SEMINARS

1. 15/01/2014 *"Orden magnético y ferroelectricidad en la familia de multiferroicos de tipo II Mn(1-x)CoxWO4"*, Irene Urcelay (BCMaterials Fortnightly Seminars).
2. 15/01/2014 *"Propiedades Físicas de Óxidos de Metales de Transición"*, Daniel Salazar (BCMaterials Fortnightly Seminars).
3. 29/01/2014 *"Poliamidas aromáticas"*, Maria San Sebastian (BCMaterials Fortnightly Seminars).
4. 29/01/2014 "*Síntesis y encapsulamiento de nanopartículas magnéticas de base ferrita"*, Xabier Lasheras (BCMaterials Fortnightly Seminars).
5. 11/02/2014 *"Electroactive Smart Materials"*, Senen Lanceros-Mendez (BCMaterials Fortnightly Seminars).
6. 26/02/2014 *"Magnetoelectric effect in new laminated composites"*, Andoni Lasheras (BCMaterials Fortnightly Seminars).
7. 26/02/2014 *"Nitrile Containing Copolyimides for Sensor Applications"*, Alberto Maceiras (BCMaterials Fortnightly Seminars)
8. 18/03/2014 *"Fabrication of magnetic nanodisks by self-assembled templates"*, Maite Goiriena (BCMaterials Fortnightly Seminars).
9. 18/03/2014 "*Microstructural Characterization of Topological Insulators and Shape Memory Alloys Using Nuclear Techniques"*, Iraultza Unzueta (BCMaterials Fortnightly Seminars).
10. 25/03/2014 *"Immobilization of Hybrid Polyoxometalates on Tailored Polymeric Surfaces and Applications"*, Jagoba Martin (BCMaterials Fortnightly Seminars).
11. 25/03/2014 *"Shape Memory Effect for Recovering Surface Damages on Polymer Substrate"*, Nuria Garcia (BCMaterials Fortnightly Seminars).
12. 01/04/2014 *"Development of Polymeric Multilayers for Biomedical Applications"*, Ariane Sagasti (BCMaterials Fortnightly Seminars).
13. 01/04/2014 *"MOF design and synthesis"*, Laura Bravo (BCMaterials Fortnightly Seminars).

14. 15/04/2014 *"Physico-chemical studies of complex silver-magnetite nanostructures"*, Diego Muraca (BCMaterials Fortnightly Seminars).

15. 15/04/2014 *"Latest results on thin film magnetoimpedance micro-structures"*, Eduardo Fernandez (BCMaterials Fortnightly Seminars).

16. 17/06/2014 *"Silver Vanadium Oxides for Battery Applications"*, Roberto Fernández (BCMaterials Fortnightly Seminars).

17. 19/11/2014 *"Effect of Fe on the martensitic transformation in NiMnSn produced by melt-spinning"*, Christian Omar (BCMaterials Fortnightly Seminars).

18. 19/11/2014 *"Películas delgadas de aleaciones ferromagnéticas con memoria de forma"*, Iván Rodríguez (BCMaterials Fortnightly Seminars).

19. 03/12/2014 *"Nanocomposites Nd0.05Bi0.95Fe0.95Co0.05O3/PVDF & Nd0.05Co0.95Fe2O4/PVDF by bubble and traditional electrospinning method"*, Netzahualpille Hernández (BCMaterials Fortnightly Seminars).

20. 03/12/2014 *"High voltage cathodes for Na-ion batteries: Na3V2O2x(PO4)2F3-2x system"*, Paula Serras (BCMaterials Fortnightly Seminars).

21. 10/04/2014 *"Exchange bias and magnetization dynamics in nanostructures"*, H. Srikanth (FCyT).

22. 10/04/2014 *"Materiales magnéticos nanoestructurados como adsorbentes en el rango de microondas"*, G.V. Kurlyandskaya (FCyT).

23. 11/04/2014 *"Functional polymer nanocomposites for tunable high frequency device applications"*, H. Srikanth (FCyT).

24. 29/05/2014 *"Magnetic Materials in Medicine: Applications in Diagnosis, Management and Treatment of Disease"*, Tim St. Pierre (FCyT).

25. 15/07/2014 *"Quantitative theory of the entropy change at the martensitic transformation"*, V. A. L'vov (FCyT).

26. 23/07/2014 *"Nuevos materiales magnéticos con aplicaciones biomédicas"*, José Manuel Barandiaran (Berripills, Hospital de Cruces).

27. 17/10/2014 *"Design of magnetic nano-architecture"*, Davide Peddis (FCyT).

28. 24/10/2014 *"Caracterización de fases magnéticas por resonancia de espín (Electron Spin Resonance, ESR)",* Raúl Valenzuela (FCyT).

29. 05/11/2014 *"Nanobiomagnetismo: magnetismo en bacterias y otros seres vivos",* María Luisa Fernández-Gubieda (FCyT).

HIGH LEVEL EDUCATION

BCMaterials is deeply involved in postgraduate programs on Materials, at Master and Doctorate level, committed to motivate and prepare graduates in Physics, Chemistry and Engineering for research at the cutting edge.

Master on New Materials. Academic year 2014-15.

This is an inter-University Master UPV/EHU- University of Cantabria, running already for 5 academic years. The Master is intimately linked to BCMaterials: The academic responsible of the Master is Prof JM Barandiaran, Scientific director of BC Materials. Most BCMaterials academic members (faculty) do lecture in the Master and some researchers supervise Master Thesis in New Materials.

After an intense campaign of advertising, the Master got 64 pre-registrations at the UPV/EHU. From those 23 candidates were selected, and finally 17 started the Master Degree. Adding 4 more from the University of Cantabria this year we have 21 full time students.

We offered 4 fellowships to develop the Master Thesis at BCMaterials, All BCMaterials students are already working in the different research lines.

The Master was verified in November 2014 by the UNIBASQ and ANECA agencies for another 4 years operation.

http://www.ehu.eus/es/web/nuevosmateriales/aurkezpena

Lectures of BCMaterial members in other Masters

In addition, several academic members and researchers of BCMaterials are lecturing in other Master degrees, such as:

- Master in "Ingeniería Física": Jon Gutiérrez, M. Luisa Fernández-Gubieda, Alfredo García Arribas, José Manuel Barandiaran.
- Master in "Análisis Forense": Maribel Arriortua, Begoña Bazán.
- Master in "Cuaternario": Maribel Arriortua, Begoña Bazán.

- Master in "Química Sintética e Industrial": Maribel Arriortua, Begoña Bazán.

New PhD program: Doctorate in Science and Technology of Materials

http://www.ehu.eus/es/web/estudiosdeposgrado-graduondokoikasketak/doktorego-eskaintza-ikasle-berriak?p_cod_idioma=CAS&p_propuesta=1796&p_menu=1&requestTime=1425369464807

New PhD programs have been put forward in the UPV/EHU, following the Bologna process, to adequate national qualifications frameworks to the future European Qualifications Framework. In this way, they aim to motivate and prepare PhD candidates as to:

- Carry out research independently,
- Analyse scientific events with wide and deep perception,
- Determine necessary steps to reach new synthesis,
- Make original contributions to science.

The new PhD program in Science and Technology of Materials was designed, and is now directed, by Prof. L León, member of BCMaterials, and will incorporate all new PhD students from BCMaterials.

The program was evaluated positively by UNIBASQ in 2013 and by ANECA in 2014.

It started operation in September 2014. In his first year, the PhD program got 20 pre-registered students, from which 17 were accepted.

OTHER ACTIVITIES

LARGE FACILITIES PROPOSALS AND MEASUREMENTS

"Determination of Removal/Uptake of Crystallization and Coordination Water Molecules Mechanism for Two Supramolecular Metal-Organic Frameworks"

DATES: FROM JANUARY 21ST TO JANUARY 25TH, 2014 (12 SHIFTS - 96 H)
EXPERIMENT: DETERMINATION OF REMOVAL/UPTAKE OF CRYSTALLIZATION AND COORDINATION WATER MOLECULES MECHANISM FOR TWO SUPRAMOLECULAR METAL-ORGANIC FRAMEWORKS.

LINE/BEAMLINE: BM25A: The Spanish CRG Beamline for Absorption and Diffraction. ESRF Grenoble, France.

PROPOSERS: M.K. Urtiaga, M.I. Arriortua, B. Bazán, A. Calderón, R. Fernández de Luis, F. Llano, G. Barandika

RESULTS: Reversible crystal to crystal transformation upon desorption/adsorption of water molecules has been studied by means of X-ray diffractometry and (TDX) and synchrotron radiation. The compound 1 exhibit the general formula [Cu(PDC)(dpk)(H2O)], and for the dehydrated phase 2 is [Cu(PDC)(dpk)]. Single crystals of compound 1 were obtained through agarose gel synthesis, being characterized by single-crystal X-ray diffraction. In the case of the phase dehydrated 2, the crystal structure shows a readjustment upon desorption of the coordination water molecules from 1, breaking the connection along the [100] direction, and giving rise to a 1D supramolecular network. A reversible solid state transformation in powder sample takes place between 1 and 2 upon a heating treatment, and the crystal structure of 2 has been solved via Rietveld refinement. The X-ray pattern at 130°C of compound 2 has been collected in the BM25B SpLine of the ESRF (European Synchrotron Radiation Facility).

“Temperature dependence of the ferromagnetic domain configurations and exchange bias effects in antiferromagnetic / ferromagnetic systems”.

PROPOSAL NUMBER: CRG-2130
DATES: FROM DECEMBER 16TH TO DECEMBER 21ST, 2014.
EXPERIMENT: TEMPERATURE DEPENDENCE OF THE FERROMAGNETIC DOMAIN CONFIGURATIONS AND EXCHANGE BIAS EFFECTS IN ANTIFERROMAGNETIC/ FERROMAGNETIC SYSTEMS.

LINE/BEAMLINE: SuperADAM. ILL Grenoble, France.

PROPOSERS: D. Navas, R. Morales, C. Redondo, F. Batallán, B. Toperverg, I. K. Schuller.

RESULTS: Polarized neutron reflectivity (PRN) measurements were obtained for FeF2/NiFe bilayers in the temperature range from 10 K to 70 K. Magnetization reversal curves show that the NiFe domain structure strongly depends on the temperature. PRN curves at different magnetic fields and temperatures confirm distinctive features that must be analysed.

“In situ study of the microstructural recovery processes in NiMnGa and NiMnIn metamagnetic chape memory alloys”.

PROPOSAL NUMBER: CRG-2158
DATES: FROM SEPTEMBER 26TH TO SEPTEMBER 29TH, 2014.
EXPERIMENT: IN SITU STUDY OF THE MICROSTRUCTURAL RECOVERY PROCESSES IN NIMNGA AND NIMNIN METAMAGNETIC CHAPE MEMORY ALLOYS.

LINE/BEAMLINE: D1b and D2b. ILL Grenoble, France.

PROPOSERS: J. I. Pérez Landazabal, V. Recarte, V. Sánchez, I. Unzueta.

“Atomic scale properties of magnetic Mn-based alloys probed by emission Mössbauer spectroscopy”.

PROPOSAL NUMBER: IS578
DATES: FROM AUGUST 26TH TO SEPTEMBER 13TH, 2014.
EXPERIMENT: ATOMIC SCALE PROPERTIES OF MAGNETIC MN-BASED ALLOYS PROBED BY EMISSION MÖSSBAUER SPECTROSCOPY.

LINE/BEAMLINE: ISOLDE14. CERN Geneva, Switzerland.

PROPOSERS: R. Mantovan, H. P. Gunnlaugsson, A. Zenkevich, J. H. Zhao, F. Plazaola, K. Johnston, H. Masenda, T. E. Mølholt, D. Naidoo, M. Ncube, K. Bharuth-Ram, M. Fanciulli, J. A. Garcia, H. P. Gislason, G. Langouche, J. Lu, S. Ólafsson, I. Perez Landaluze, V. Rylkov.

RESULTS: 57Fe Emission Mössbauer spectroscopy measurements were obtained for Ni50Mn25In25, Ni50Mn34In16 and Ni50Mn37Sn13 in the temperature range from 111 K to 724 K. Mössbauer spectra at room temperature show that there are two inequivalent positions for implanted Fe atoms with the same area, indicating that Fe atoms locate in Ni and (Mn, Sn/In) positions randomly. In high temperature measurements a new subspectrum appears at the Curie temperature. Low temperature spectra show different behaviours that must be analysed.

“EXAFS study on the magnetite biosynthesis of Magnetospirillum gryphiswaldense”

PROPOSAL NUMBER: LS-2276
EXPERIMENT: BEAMLINE BM23, SYNCHROTRON ESRF. GRENOBLE (FRANCIA)
DATES: FROM 14-05-2014 TO 19-05-2014 (4 DAYS)

TITLE: EXAFS study on the magnetite biosynthesis of Magnetospirillum gryphiswaldense

PROPOSERS: Ana García Prieto, Alicia Muela, J. Alonso, M. Luisa Fernandez-Gubieda

This proposal was a continuation of a previous XANES experiment, in which we were able to follow the process by which the magnetotactic bacteria M. gryphiswaldense form the magnetite nanoparticles. The previous experiment led us to conclude that the bacteria use ferrihydrite as a source of Fe ions to form magnetite particles.

With this experiment we have performed EXAFS on the Fe K-edge in a time-resolved study. EXAFS has allowed us to probe the local surroundings of the Fe atoms, in particular the nearest neighbours' distances and the occupation states, and to follow the transformation of the ferrihydrite phase to the magnetite as the biomineralization process evolves.

"Microstructural study of ferritin-like protein cores in prokaryotes"

PROPOSAL NUMBER: 20140081
EXPERIMENT: BEAMLINE XAFS, SYNCHROTRON ELETTRA. TRIESTE (ITALIA)
DATES: FROM 23-07-2014 TO 27-07-2014 (4 DAYS)

TITLE: Microstructural study of ferritin-like protein cores in prokaryotes

PROPOSERS: Ana García Prieto, Alicia Muela, J. Alonso, M. Luisa Fernandez-Gubieda

Even though the microstructure and magnetic properties of mammalian ferritin cores have been extensively studied over the years, this is not the case for ferritin-like protein cores of prokaryotes. This could be attributed to their lack of long-range order, usually ascribed to the higher phosphorous content as compared to mammalian cores, which makes most common structural techniques such as XRD and/or TEM unable to determine the microstructure in detail. Fe K-edge EXAFS is a necessary technique to probe the microstructure of the ferritin-like protein cores of prokaryotes because it does not require long-range order and allows probing the microstructure locally. By means of Fe K-edge EXAFS measurements we have studied the microstructure of mammalian, bacterial (bacterioferritin and bacterial ferritin) and archaeal ferritin-like protein cores aimed to understand i) the role of the phosphorous content on the microstructure and magnetic properties of the mineral cores, and ii) the microstructural changes occurring during the ferritin-like protein core biomineralization process.

“Influence of Fe addition on structural and magnetic properties of Mn49Ni42-xFexSn9 (x=0, 2, 3, 4, 5 and 6) Heusler alloys”.

PROPOSAL NUMBER: CRG-2146
EXPERIMENT: NEUTRON DIFFRACTION STUDY TO DETERMINE THE ROLE OF THE FE ADDITION ON THE MARTENSITIC TRANSFORMATION AND THE MAGNETIC INTERACTIONS IN A SERIES OF MN-NI-FE-SN METAMAGNETIC SHAPE MEMORY ALLOYS.
DATES: FROM DECEMBER 2ND TO DECEMBER 3RD, 2014.

LINE/BEAMLINE: D1B, ILL Grenoble, France.

PROPOSERS: P. Lázpita, V. A. Chernenko, J. M. Barandiarán, J. Gutiérrez, I. Urcelay, J. A. Rodríguez-Velamazán

RESULTS: The diffraction patterns for different compositions were measured as function of temperature in the cooling and heating processes, between 50 K and 350 K, showing different behaviors depending on the Fe content. The alloys which their compositions present less than the 4 % Fe show a structural transformation, from a high symmetry in the austenite phase, cubic, to a lower one in the martensitic phase, orthorhombic. Depending on the composition, the structural transformation occurs at different temperatures which decrease with the increment of the Fe content as was expected. This group of alloys presents a partial structural transformation, and an important amount of the austenite phase remains still at 50 K (a temperature far from their martensitic transformations). In the other hand, the high content of Fe in the 5 and 6 % Fe samples prevents the structural transformation, showing a high symmetry cubic structure invariant unless until 50 K. A deeply analysis of the data, which will allow determining the magnetic order of the different phases and the influence of the atomic order in the martensitic transformation, is now in progress.

“Study of lattice instability and magnetic states in Ni-Fe-Ga-Co FSMA thin film by hard X-ray photoelectron spectroscopy”

DATES: FROM JANUARY 15TH TO JANUARY 18TH, 2014

LINE/BEAMLINE: BL47XU: Japan Synchrotron Radiation Research Institute/SPring-8, Kouto, Sayo-cho, Sayo-gun, Hyogo 679-519,Japan.

PROPOSERS: A. Kimura, K. Sumida, K. Shirai, M. Taniguchi, J. M. Barandiaran, V. A. Chernenko.

RESULTS: The temperature evolution of the electronic structure of a Ni-Fe(Co)-Ga/MgO(100), Heusler-type, ferromagnetic shape memory alloy thin film has been followed by a bulk-sensitive hard X-ray photoelectron spectroscopy, element-selective soft X-ray magnetic circular dichroism and first-principles calculation. The reversible changes of the electronic states near the Fermi energy show a hysteresis associated with the martensitic phase transition (MPT), where the pseudo-gap opens on cooling and closes again on warming. In addition, the Ni 3d spin magnetic moment increases approximately 2 times across the MPT, whereas the change of Fe 3d moment is moderate. By comparing the experimental results with the calculated spin-resolved density of states, we conclude that the band Jahn-Teller effect of Ni 3d and Fe 3d orbitals is responsible for MPT.

VISITS TO FOREIGN LABORATORIES

RAFAEL MORALES, Center for Advanced Nanoscience, University of California San Diego. USA
01/01/2014 - 26/06/2014 DURATION: 26 WEEKS

JON GUTIERREZ, Depto Física, Universidade do Minho, Braga, Portugal
25/01/2014 - 25/07/2014 DURATION: 24 WEEKS

ANDONI LASHERAS, Depto Física, Universidade do Minho, Braga, Portugal
02/05/2014 - 30/07/2014 DURATION: 12 WEEKS

NASTASSIA SORIANO,
Universidade Estadual de Campinas, Brasil
31/07/2014 – 02/10/2014 DURATION: 9 WEEKS

BEATRIZ MORA,
Centro Atómico Bariloche, San Carlos de Bariloche, Argentina
14/09/2014 – 13/12/2014 DURATION: 13 WEEKS

RAFAEL MORALES,
Centro de Desenvolvimento da Tecnologia Nuclear, Belo Horizonte, Brazil
30/10/2014 - 14/12/2014 DURATION: 6 WEEKS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