



# news

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The ALBA  
Synchrotron  
newsletter

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This Newsletter will be published at the end of a year which has meant for ALBA another step forward in its history.

On one side, the user operation has been consolidated, with more than one thousand users. Their results have had a significant impact in publications in international reviews covering both the public research and the proprietary area. A short selection of them is among the articles of this issue.

On another side, the beginning of the construction of Phase II beamlines means that the serious austerity period which we have suffered due to the critical general economic situation is coming to an end. The Infrared Spectroscopy and the ARPES communities will have access to two new instruments in ALBA in the next future, as you can read in this newsletter.

And, although it will be covered in more detail in the next issues, it is worth mentioning that the process of definition of the Phase III Beamline has started and has already produced proposals and conceptual designs thanks to the involvement of an active national community of scientists, present and future users of our instruments.

With my best wishes for a peaceful Christmas and for a New Year full of personal satisfactions and scientific advances,

**Caterina Biscari**  
ALBA Director



## ALBA launches an internship programme for undergraduate students

● In 2014, the ALBA Synchrotron has created a new programme to host university students to perform internships in its facilities.

Students may come from different academic profiles and apply to training projects in different areas of activity: physics and accelerators' technologies, scientific applications of synchrotron light, mechanics design, vacuum technologies, infrastructures, electronics, safety and radiological protection, communications and outreach and economy and public procurement, among others.

To participate in this programme, students must apply to one of the two calls per year opened by ALBA. They are evaluated according to the criteria established in the corresponding call.

In 2014, 20 students have started their internships in ALBA and 14 more have been selected so far to start their internships in 2015.

## New map of Spanish scientific infrastructures

● The Ministry of Economy and Competitiveness of the Spanish Government has recently updated the map of large scientific facilities (ICTS, *Infraestructuras Científico-Técnicas Singulares* in Spanish).

The new map includes 41 ICTS, one of which is the ALBA Synchrotron. These groups of ICTS can be single facilities, clusters or distributed facilities.

ICTS are considered by the Spanish Government as unique infrastructures open to the scientific and the industrial communities and as key players for the development of research and technology in Spain.

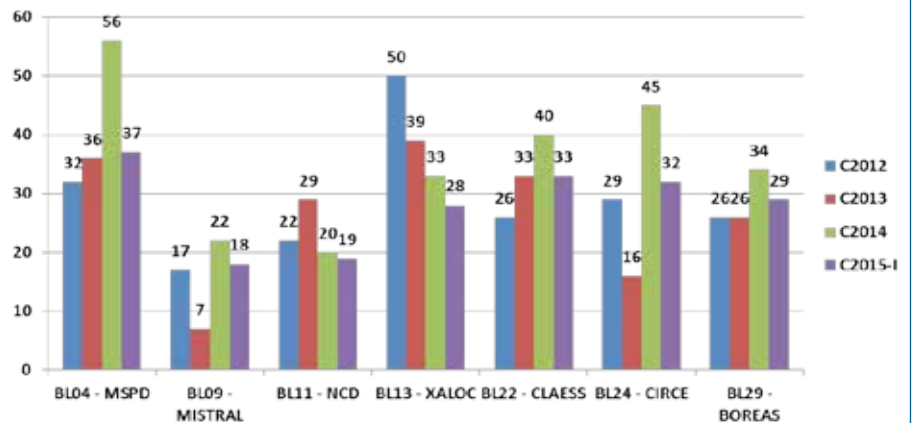
## Results of the 1<sup>st</sup> call for 2015 experiments

● Starting in 2015, the ALBA Synchrotron is opening two calls for experiments per year. The first call, which was open from June 5<sup>th</sup> till July 7<sup>th</sup>, received 196 proposals and will serve experiments from January till June 2015.

The Materials Science and Powder Diffraction (MSPD) beamline received the largest number of proposals (37). The highest number of shifts (607) was requested at the absorption and emission spectroscopy CLAES beamline.

It is also remarkable that this call has been the most international to date: 64% of proposals came from Spanish institutions, 31% from European institutions and 5% from other countries.

Total submitted Proposals per beamline from CALL 2012 to CALL 2015



## ALBA will host MEDSI 2016

● ALBA has been chosen to host and organize the 9th edition of the Mechanical Engineering Design of Synchrotron Radiation Equipment and Instrumentation (MEDSI) conference. ALBA was selected in the last conference held in Melbourne in October 2014.

Around 150 delegates from synchrotron facilities all over the world will participate in MEDSI 2016 to showcase and exchange cutting-edge developments in mechanical design and engineering of synchrotron-based instrumentation. We hope to see you all in Barcelona!

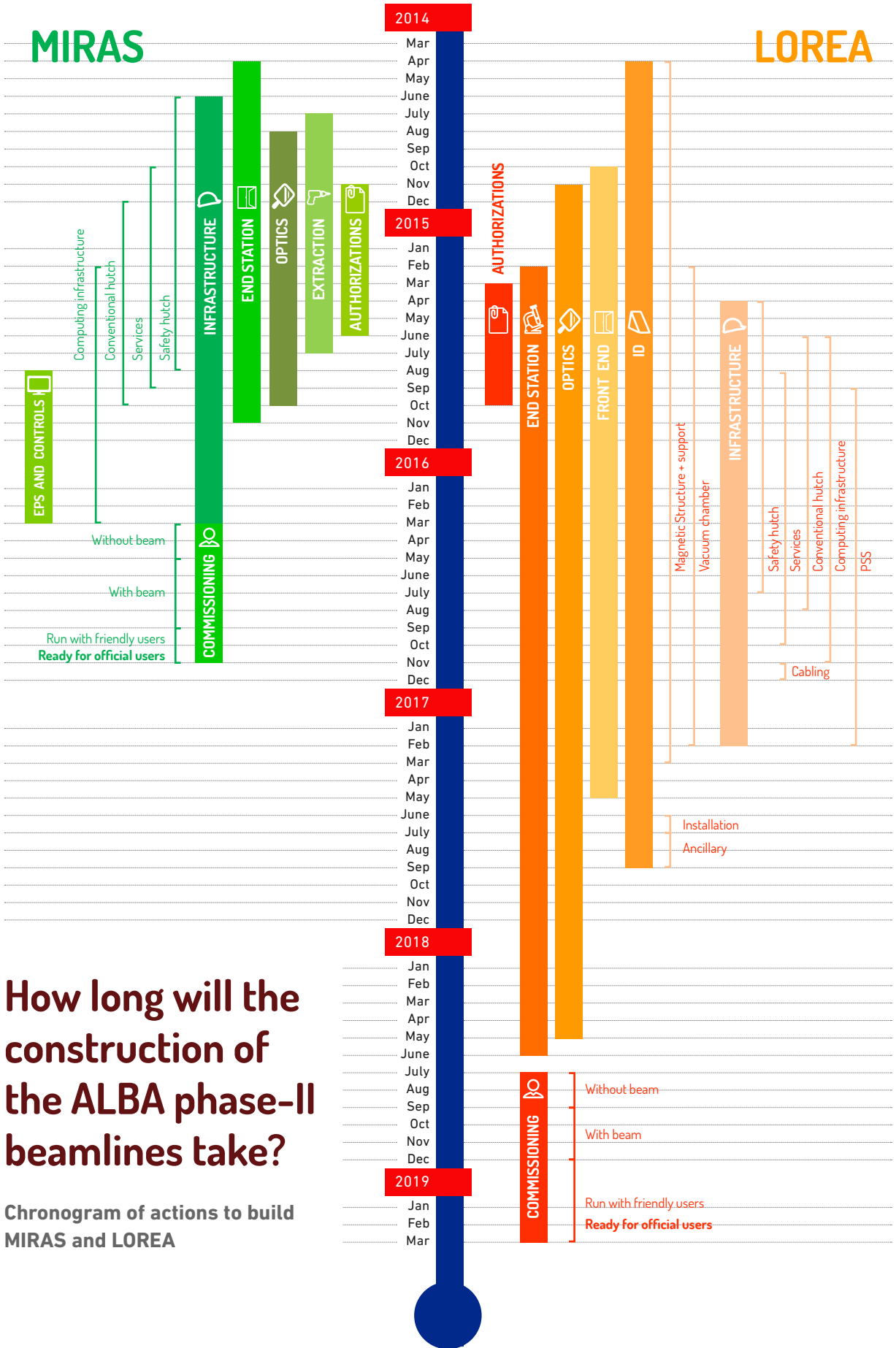


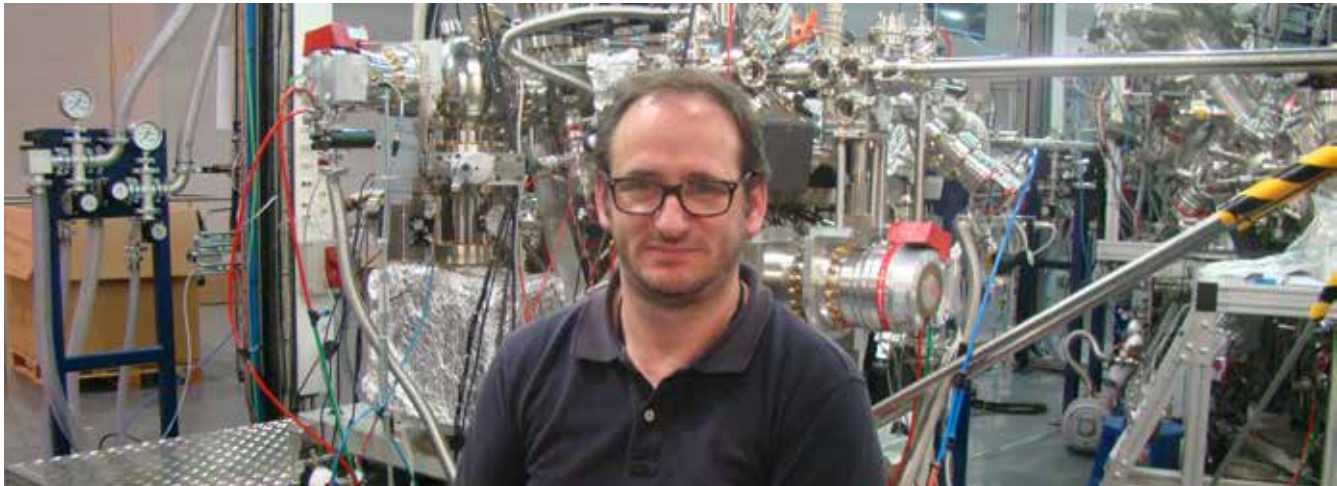


## Focus on phase-II beamlines

**ALBA phase-II beamlines - MIRAS and LOREA - have started its construction in 2014. The infrared microspectroscopy beamline (MIRAS), dedicated to infrared spectro-imaging, will be a valuable tool for the study of molecules and functional groups. The low-energy ultra-high resolution angular photoemission beamline (LOREA) will let us understand the electronic structure of graphene-based material, topological insulators and other advanced materials.**

This report gives us new insights about the status and complexity of both projects. An infographic with a timeline of the MIRAS and LOREA projects shows the different steps of their construction. An interview with Josep Nicolas offers us complementary information about the project management of phase-II beamlines. A complete report by Professor Enrique García Michel describes the characteristics and capabilities of the ARPES technique, which will be available in the future LOREA beamline.





## How to manage the construction of a new beamline

We have talked to Josep Nicolas, head of the Optics, Metrology and Support section of the ALBA Synchrotron, and one of the persons responsible for the phase-II beamline project management. During the conversation, we have discovered more about how to manage the construction of a new beamline inside a synchrotron facility.

### **Building a beamline seems quite complicated... Can you briefly explain the different steps?**

The first thing is to have a clear definition of what beamline you want. Based on the users' proposal, you have to define the goals of the beamline, design the optics and endstation, build the beamline (where we distinguish three different parts: the source and front-end, the beamline optics and the endstation), do the call for tenders and then, mechanical design, manufacture and testing. In the meantime, you are also building the infrastructure around these beamline components: hutch, media supplies, control system, computer, racks, cables,... Once the installation is completed, commissioning starts and, finally, friendly users arrive. They are users who bring samples to do experiments to check the limits and initial performances of the beamline, but without guarantee that things will work.

### **How long does the process of constructing a new beamline take?**

Between three and four years, from the beginning till the commissioning.

### **How many people can be involved in this process?**

The beamline normally takes 13 work packages (inside each of them there are three or four people). So there are about 40-50 people from the ALBA staff working in the project (although not full time). Apart from them, we also have to take into account the external suppliers and collaborators that may contribute to the beamline development. The beamline scientist should be involved in the very beginning to better define the technical requirements of the beamline (which source is needed, what kind of optics are wanted and details of everything)

### **Is it very expensive to build a beamline?**

Yes, but it depends on what you compare it with... This is high quality, high precision elements, high technology.

Scientific equipment is normally expensive and a beamline is a very big lab. Besides, there are not many supplying companies and prices are always high.

### **When constructing a beamline, are there many internal developments?**

For phase I beamlines there were some internal developments but main beamline components were essentially bought from companies. This is not going to be the case for phase-II beamlines because the ALBA staff have more experience. Now we can really assume the installation, the integration, the vacuum conditioning,... because our staff have good experience. For example, MIRAS has been designed profiting from a strong collaboration with Soleil and a group of users from ICTP (CSIC). It is now being constructed with internal resources. Only the endstation will be bought and then integrated by us.

### **In your opinion, which are the main difficulties or challenges of a project like this?**

When building beamlines in the situation that we have at ALBA, the main problems are cost and time and we have a limited budget. The main risk always is delay because we are short of human power and it's easy to get delays. At the beginning you have to be careful not to make fundamental errors that will invalidate everything. This is why it's important to get some advice from outside. On the details you can make mistakes, but these are easier to solve. Every beamline has its own story and you can hardly foresee what is going to happen in detail.

### **Constructing a beamline means putting in common people from different disciplines: scientists, engineers, computer engineers,... Is that the most difficult part?**

It's a difficult part but we have learned a lot. Beamline scientists need to understand the language of engineers and vice versa.

# ARPES: watching the electronic bands



**Enrique Garcia Michel**  
Department of Condensed Matter Physics  
Universidad Autónoma de Madrid

The Angle Resolved Photoemission Spectroscopy (ARPES) technique is based on the photoelectric effect originally described by Hertz in 1887. In the photoelectric effect, a metal surface emits electrons when light shines upon it. These electrons are usually called “photoelectrons”. Einstein explained the photoelectric effect in one of his famous articles published in 1905 as a manifestation of the quantum nature of light. His idea was that an electron can absorb a quantum of light (a photon) and use this energy to escape the solid. He was awarded the 1921 Nobel Prize in Physics for this discovery. The physical properties of the photoelectron outside the solid (and in particular the energy) carry information on the values of these properties inside the solid. This is the main idea behind Photoemission Spectroscopy: by analysing the photoelectrons, we can learn about the properties that the electrons had before the photoemission process. The first magnitude that can be measured is the electron kinetic energy. The electron energies involved vary in the range from a few eV (for electrons in the shallow levels of the valence band) to several hundreds or even thousands of eV (for electrons belonging to the deeper core levels of the atoms). In general, the analysis of the kinetic energies of the photoelectrons will require a resolution in the range of tenths of eV for the valence band, and the whole measurement process has to be performed in vacuum, as otherwise the mean free path of photoelectrons would be too short.

The most common electron spectrometer able to perform this energy analysis is a hemispherical analyser. Two metallic hemispheres are used as an energy filter, so that

only electrons with a selectable kinetic energy can travel across the hemispheres and reach a detector. Modern Photoelectron Spectroscopy started by the late 60s of the 20th century as the first analysers of this kind became commercially available, following the work of Siegbahn for core-level electrons (who was awarded the 1981 Nobel Prize in Physics), of Spicer for the valence band and of Turner for gas phase photoemission.

Besides the energy, several other properties of the photoelectrons are interesting. The most important one is the momentum (Fig. 1). Photoelectrons excited inside the solid move in different directions. As the momentum carried by the photon is very small compared to the momentum of the electron inside the solid, the momentum of the photoelectron carries information on the momentum of the electron in its initial state. This is a relevant property, as the electron energy states inside the solid can be classified according

to their momentum, i.e. momentum is a good quantum number, and energy vs. momentum representations of electronic states (electronic band structure) provide all the information required to understand the electronic behaviour of a certain material. In order to measure the photoelectron momentum, the emission direction has to be determined. Thus, it is necessary to measure how many photoelectrons leave the solid along any possible direction, and with which kinetic energy. From the technical point of view, it is sufficient to design a hemispherical analyser with a narrow acceptance cone, and to displace it facing the surface, so that only photoelectrons emitted along a particular direction are detected. This approach was broadly used in the 70s and 80s, but its main limitation is that, in order to obtain a good angular resolution (in the range of 1-2° at that time), a narrow emission cone needs to be selected. This decreases the number of available photoelectrons accordingly, setting a limit to the

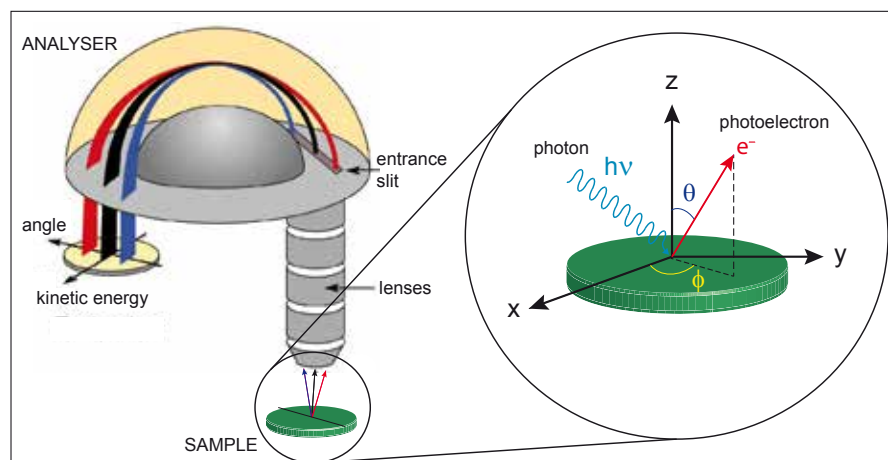


Figure 1: Scheme of a two-dimensional electron analyzer. The angle resolution is achieved by a slit and a two-dimensional detector. The zoom shows the sample with incident photons and the outgoing photoelectrons, whose properties are measured in the experiment.



energy resolution in the range of 20-40 meV, so the use of an intense photon source is critical. The advent of dedicated synchrotron radiation sources since the 70s was the first crucial step in the development of ARPES as a standard method. The use of synchrotron radiation has other advantages. As the light is polarized, the symmetry (parity) of the electron initial state in the solid can be determined. Finally, ARPES can be combined with spin detection to provide information on the electron spin. We may say that ARPES can experimentally determine all the quantum numbers of electrons in solids (energy, momentum, parity and spin) and is thus the most powerful technique to probe the electronic structure of materials.

A new technical improvement in the 90s, the use of two-dimensional detectors, transformed ARPES into the versatile and powerful technique that it is nowadays. A two dimensional detector (multichannel plate) placed behind the hemispherical analyser permits a much faster data acquisition and enables electronic angular resolution, i.e. the hemispherical analyser is fixed and electrons leaving the sample with different angles reach the detector at different places (Fig. 1). This approach allowed to easily reach resolutions of 0.2°/2-10 meV, and a signal to noise ratio that permitted the representation of data as a grey scale image (Fig. 2a). Further developments have significantly improved these standards. These developments coincided with the investigation of several scientific topics where the analysis of the electronic band structure and of the Fermi surface, as provided by ARPES, was crucial to understand the physical behaviour.

We may first mention the emergent physical phenomena in complex quantum matter, including high temperature superconductivity and colossal magnetoresistance. ARPES results have had a crucial impact on the study of the superconducting cuprates. These compounds belong to the perovskite family. The undoped parent compounds are anti-ferromagnetic insulators and the

doped compounds have the highest known superconducting transition temperatures, a phenomenon not yet understood. An analysis of the Fermi surface of these materials with ARPES provided the necessary information to understand many of the properties of these complex materials, notably we may highlight that it revealed that their pairing symmetry was different from the symmetry so far observed in other superconductors. This research has extended to pnictides, iridates, nickelates, and several other materials exhibiting a complex physical behaviour. Fig. 2c shows a representation of reciprocal space (including the electron band structure and the Fermi surface) and of its experimental determination with ARPES.

In recent years, graphene has deserved ample attention in the ARPES community. Its characteristic band structure shows a linear energy vs. momentum dispersion (Fig. 2b). As the velocity of the carriers is related to the band curvature, a linear dispersion relationship implies a constant velocity, independent of the kinetic energy, as found in a massless relativistic particle. This finding indicates that the carriers indeed

behave as Dirac fermions.

Topological insulators are a new class of materials with unexpected electronic properties: they are bulk insulators, but exhibit electronic states localized at their surface, which are protected by the symmetry of the material ("topologically protected"). This protection makes them robust against perturbations like defects. ARPES has demonstrated the experimental realization of topological insulators and has provided ample information of the protected surface states. Current research focuses on topics like the nature of the interaction of these states with magnetism and on the properties of the interfaces formed between topological insulators and superconductors, which are expected to exhibit unconventional properties.

ARPES is now a vigorous, well-established technique, but it is also improving its technical features. New technical developments, like time-of-flight detection, the use of lasers as exciting source, and the improvement of spin detection methods, ensure that ARPES will continue providing exciting new physical information on the electronic properties of solids for a long time.

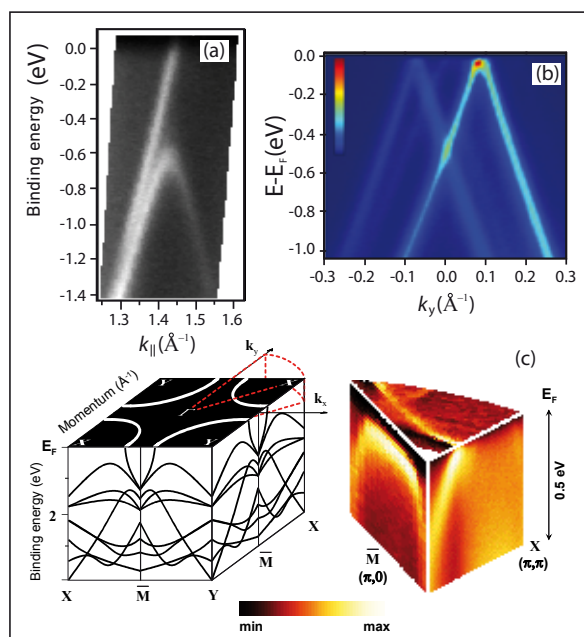


Figure 2: (a) Electron band structure of Sn/Cu(100) in the charge density wave (CDW) state. The band folding observed near 1.45 Å<sup>-1</sup> is due to the formation of a CDW. (adapted from Phys. Rev. B 72, 041401 (2005)). (b) ARPES measured band structure of an 11-layer graphene film grown on SiC. The graphene layers are electronically decoupled, so that each Dirac cone corresponds to an individual macroscale graphene sheet (adapted from Phys. Rev. Lett. 103, 226803 (2009)). (c) Theoretical and experimental electron band structure and Fermi surface of Bi<sub>2</sub>Sr<sub>2</sub>CaCuO<sub>8-δ</sub> cuprate superconductor. The left panel shows the distribution of electron bands in reciprocal space. The red dashed lines highlight the portion of reciprocal space probed with ARPES in the right panel (adapted from Phys. Rev. B 64, 094513 (2001)).

## Unravelling the architecture of real catalysts

Jordi Llorca and Núria Jiménez Divins  
Institute of Energy Technologies  
Center for Research in NanoEngineering  
Universitat Politècnica de Catalunya (UPC)



● Catalysts used in industry and in pollution abatement applications usually consist of metal nanoparticles supported over high surface area supports, which are usually inorganic oxides. The identification of the active sites of these catalysts and the recognition of their exact reactivity are two of the most important issues addressed in catalysis because it is the way to improve our knowledge and to design new generation of better catalysts based on fundamentals rather than trial and error approaches.

We have been working at the Universitat Politècnica de Catalunya (UPC) for several years with bimetallic rhodium-palladium nanoparticles supported over cerium dioxide,  $\text{RhPd/CeO}_2$ , to produce hydrogen through the ethanol steam reforming reaction,  $\text{C}_2\text{H}_5\text{OH} + 3\text{H}_2\text{O} \rightarrow 6\text{H}_2 + 2\text{CO}_2$ , with the objective of designing an active, selective and robust catalyst for this reaction, which represents an attractive route for hydrogen generation from a renewable source (bioethanol) for energy applications. Rhodium was selected due to its ability to break the carbon-carbon bond of the alcohol molecule and palladium was chosen because of its ability to recombine hydrogen atoms into molecular hydrogen gas, whereas the cerium dioxide support played a fundamental role activating the water molecule and avoiding the deposition of carbon by providing oxygen atoms from its lattice due to its redox properties ( $\text{Ce}^{\text{IV}}\text{O}_2 \leftrightarrow \text{Ce}^{\text{III}}\text{O}_3$ ). With our in-house X-ray photoelectron spectroscopy (XPS) system, we could follow the rearrangement of Rh and Pd in the bimetallic nanoparticles after in situ exposure to  $\text{O}_2$ ,  $\text{H}_2$ , and ethanol steam reforming atmosphere in the presence and absence of the ceria support. We realized that the support had a tremendous effect on the segregation of the metals at the surface of the nanoparticles as well as on their oxidation states.

These promising preliminary results obtained under ultrahigh vacuum (UHV) conditions were the basis to apply for beamtime at the CIRCE beamline (NAPP endstation) at ALBA synchrotron. Well-defined bimetallic  $\text{Rh}_{0.5}\text{Pd}_{0.5}$  nanoparticles of ca. 4 nm in diameter were prepared by Inma Angurell (UB) and were deposited separately on a tungsten foil and on  $\text{CeO}_2$  particles, constituting a real catalyst, which was pressed to form a pellet. The samples were introduced into the NAPP and the same experiments previously performed were carried out under near-ambient pressure conditions (0.05 mbar). The experiments, conducted by ourselves and Carlos Escudero and Virginia Pérez-Dieste from ALBA, were carefully carried out with laser heating and leak valves to control the temperature and to introduce the different atmospheres in the analysis chamber, respectively. At each condition, spectra were recorded at three different photon energies (670, 875, and 1150 eV) in order to monitor the composition and oxidation state of the species present at different depths (the chosen photon energies account for inelastic mean free paths of  $\text{Rh}_{3d}$  and  $\text{Pd}_{3d}$  photoelectrons of ca. 0.7, 0.9 and 1.2 nm, respectively).

The results, published in *Science*, demonstrate that the structure of heterogeneous catalysts is dynamic and depends on the composition of the





surrounding environment. Both their surface structure and composition are modified when the gaseous conditions change in order to adapt their electronic properties and geometry to the new surrounding environment. In addition, some structures and active phases only exist under reaction conditions, and these differ from those identified under UHV conditions. Most importantly, the reducible ceria support plays a crucial role in the catalytic process by providing new active sites and strongly affects both the physical and chemical properties of the metal nanoparticles which, in turn, have a strong effect on the catalytic performance. In particular, Pd developed a core-shell structure of oxidation states, with a large oxidized Pd component in the outer shell of the  $Rh_{0.5}Pd_{0.5}/CeO_2$  catalyst. The interaction of the metal nanoparticles with the ceria support not only provided active oxygen atoms to the superficial metal atoms of the nanoparticles, but also limited the reorganization of the metals under reaction ("quenching effect"), with respect to the unsupported metal nanoparticles.

This work constitutes one of the few examples reported in the literature where the surface of a real catalyst has been characterized by XPS under controlled atmospheres closely reproducing working conditions. Most of the fundamental work is usually conducted in model systems such as single crystals, thin films, or isolated nanoparticles, but these systems lack some important features found in real catalysts such as a high surface area, different types of defects, exposed planes and anchoring sites, the presence of reactive hydroxyl groups, the ability to activate certain reactants, etc., all of which play an important role in the catalytic processes.

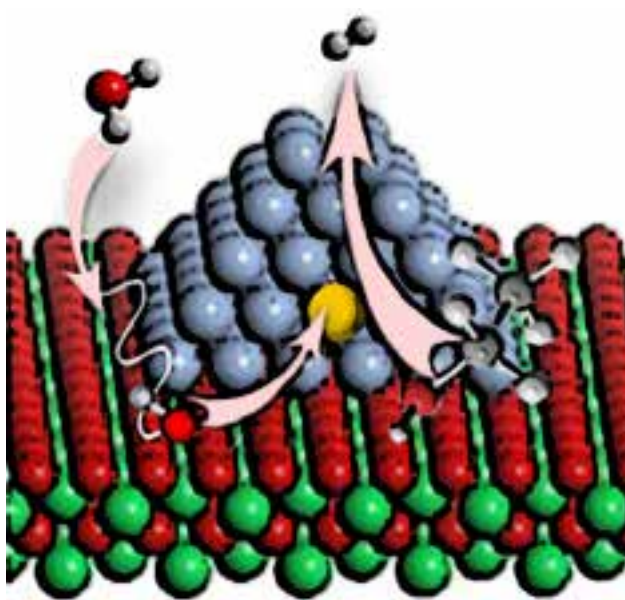


Figure 3: Drawing of a ceria-supported metal rhodium-palladium nanoparticle. The ethanol and water molecules are adsorbed at the interface between the nanoparticles and the support and they generate new reactive chemical species that make the rhodium and palladium atoms reorganise on the surface, change their oxidation state and thus facilitate the production of hydrogen.

"Influence of the support on surface rearrangements of bimetallic nanoparticles in real catalysts"  
Núria J. Divins, Inma Angurell, Carlos Escudero, Virginia Pérez-Dieste, Jordi Llorca. Science Vol. 346 no. 6203, 620-623



## Health & Safety participation in IRRMA-9 conference

● ALBA Health & Safety took part, on the 7th and 8th of July 2014, in the IRRMA-9 conference (9th International Topical Meeting on Industrial Radiation and Radioisotope Measurement Applications) in Valencia (Spain). Organized by the Department of Nuclear and Chemical Engineering of the Polytechnic University of Valencia (DIQN-UPV) and the Institute for Industrial, Radiophysical and Environmental Safety (ISIRYM), IRRMA is a triennial event with the objective to bring together scientists and engineers from all around the world to share their experience and

interests in radiation and radioisotope measurement applications.

The technical sessions included invited lectures by leading experts in their fields, lectures, papers and poster presentations. Attendees had the opportunity to exchange knowledge about industrial uses of radiation and radioisotopes, and also on a wide range of research areas and applications such as: Biomedical Applications of Radiation, Art and Cultural Heritage, Monte Carlo Methods and Models, Radiation in Environmental Sciences, Detection of Threat Material and Contraband,

Radiation Protection, Shielding and Dosimetry, Radiation Effects on Materials, Radiation Detection and Measurements.

During this conference, ALBA Health & Safety did an oral presentation about a paper entitled "A comparative shielding study at ALBA Synchrotron facility between Monte Carlo modelling and radiation monitors dosimetry measurements". The paper is a detailed study of the origin of the radiation produced at the BOREAS beamline, giving the characteristics of an additional shielding to ensure the classification of the outside of the optical hut as a public zone.

## Handling hazardous gases at ALBA

● Last September, ALBA H&S Group participated in the International Technical Safety Forum 2014 that took place at the Fermi National Accelerator Laboratory. One of the topics presented was "Handling hazardous gases at ALBA Synchrotron facility". The aim of the work was to explain how we face the presence of hazardous gases reaching the minimum risk level to the personnel and to the environment at ALBA, particularly at Beamline CLÆSS, dedicated to Core Level Absorption & Emission Spectroscopies.

This beamline uses hazardous gases from 20 bars to 50 bars to develop chemical and catalytical experiments. In order to reduce as much as possible the exposition to any toxic, flammable or corrosive gases, like CO, H<sub>2</sub> or H<sub>2</sub>S, a new setup is prepared. This setup is formed by safety cabinets, fixed gas detectors, electrovalves and an exhaust system. This project was managed by the Infrastructure section with the collaboration of beamline scientists, beamline technicians and the H&S Office staff.



## ALBA operates now in top-up mode

The ALBA storage ring has switched its operation mode from decay mode to top-up.



Figure 4a: Current in the SR as a function of time when operated in decay mode.

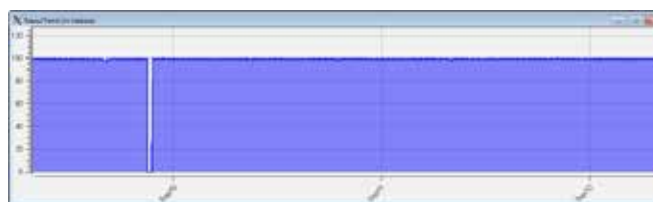


Figure 4b: Current in the SR as a function of time when operated in top-up mode.

● In decay mode there used to be two injections per day, at 7:30 am and 7:30 pm. At those times, the front ends were closed and the storage ring was replenished. This is illustrated in figure 4a which shows the stored current as a function of time for a period of 60 hours. After the summer shutdown this has changed and now the storage ring operates in top-up mode. In the accelerator world, top-up means that the current in the storage ring is kept constant within a few per mil by performing frequent injections while maintaining the photon shutters open, so that there is no interruption of the photon beam reaching the beamlines.

This injection scheme increases beam stability by keeping the heat load and signal strengths constant over a long period of time in both the storage ring and the beamlines. Figure 4b shows the current evolution in the storage ring in top-up mode.

The first synchrotron light source to be designed for top-up operation was the Swiss Light Source (SLS) which started operation in top-up back in 2001. Nowadays most of the third generation light sources operate in top-up mode. Also light sources which were not designed for top-up have moved or are planning to move to top-

up operation because of the benefits for the beamlines.

ALBA was already designed to operate in top-up mode, nevertheless there were several developments necessary before top-up could run reliably for users. First of all, it had to be shown that it is safe to inject electrons into the storage ring with the photon shutters open. Other developments included an acoustic shielding for the injector power supplies, increased local radiation shielding, an overall check of the injector system in order to increase its reliability and control automation of the injection.

## A pinger magnet for ALBA

During the summer shutdown a new diagnostics tool was installed in the ALBA storage ring. The tool is a fast pulsed magnet, with a pulse length shorter than 1.5  $\mu$ s, and a magnetic amplitude of approx. 50 mT, that will be used to perform non-linear optics studies of the storage ring.

● Non-linear optics plays an important role in the context of storage ring optimization. In a perfect and ideal circular accelerator, where the magnetic fields along the reference orbit are constant (dipolar field, as the ones used to bend the beam), or vary proportionally to the distance from the orbit (quadrupolar field, as the one used to focus the beam), every particle trajectory is stable. Unstable trajectories, instead, originate from non-linearities of the magnetic fields. Non-linear fields are for example sextupolar magnetic fields and high order terms which are needed to correct chromatic aberrations or can be originated by magnetic errors. These fields have negligible effects

close to the reference orbit, but at large amplitudes their consequences can be dramatic, limiting the amplitude of stable trajectories to a small region of the space (dynamic aperture).

The characterization of non-linear optics is a crucial step for optimizing the dynamic aperture. An increase in dynamic aperture translates into an increased injection efficiency as well as an increased lifetime.

The measurements are performed by transversely kicking a single electron train by means of the fast pulsed magnet (pinger), resulting in the excitation of a betatron transverse oscillation around the reference

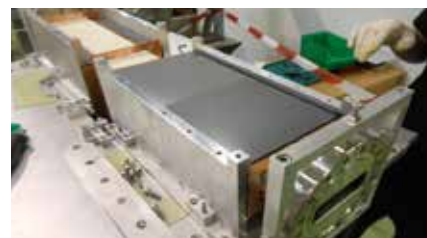


Figure 5: Assembly of the pinger magnet

orbit. If the kick is strong enough the oscillation can reach the boundary of the non-linear region where the magnetic fields of the optics exhibits strong non-linearities. The evolution of the dynamic of the electron train is then sampled, turn after turn, by beam position monitors (BPM). There are two so-called pinger magnets installed, one acting on each transversal plane. The maximum kick that can be produced is 1.5 mrad in both planes. The magnets have been designed and built in-house and the installation was done during the summer shutdown. Test with beam has started this autumn.

**BIOSCIENCES**

# Three-dimensional architecture of Hepatitis C virus replication factory studied by soft X-ray cryo-tomography

*BL-09 MISTRAL*

MISTRAL in-house research in collaboration with the National Center of Biotechnology (CNB-CSIC) groups of José Lopez Carrascosa and Pablo Gastaminza Landart has produced the first 3D map of the Hepatitis C virus infected cell.

● Hepatitis C virus (HCV) is a major cause of chronic liver disease, with an estimated 170 million people infected worldwide. The HCV chronic infection can lead to scarring of the liver and ultimately to cirrhosis, liver failure or liver cancer in the patients, nevertheless, the three-dimensional modification at cellular level promoted by the HCV infection was unknown.

The goal of this study is to obtain the first layout of a whole cell infected by the Hepatitis C virus in conditions close to the living physiological state. The cryo soft X-ray tomography performed in MISTRAL beamline is the only technique that allows obtaining

the total cartography of a whole cell without any addition of chemical compounds for fixation or staining.

The study has reported the kinetics of induction of the alterations that appear in the cells during HCV infection. For safety reasons the biological specimens used in the experiments are cells infected with HCV pseudoparticles with a subgenomic RNA replicon at different times postinfection and transfected cells Huh-7.5.1 with a HCV subgenomic stable replicon. The first significant modification that has been founded in the tomograms (3D cellular volume) is the cytoplasmic accumulation of large and small

heterogeneous vesicles and finally the appearance of multivesicular bodies and atrophied mitochondria. The viral infection dramatically changes the internal morphology of the cell. It promotes the formation of a real HCV factory web by reorganizing all the membranous compartments in the cells.

The understanding of the membranous replication factory of HCV provides a powerful tool for the analysis of host-virus interactions and offers a perfect platform at the cellular level for pharmaceutical trials of new antiviral drugs or vaccines.

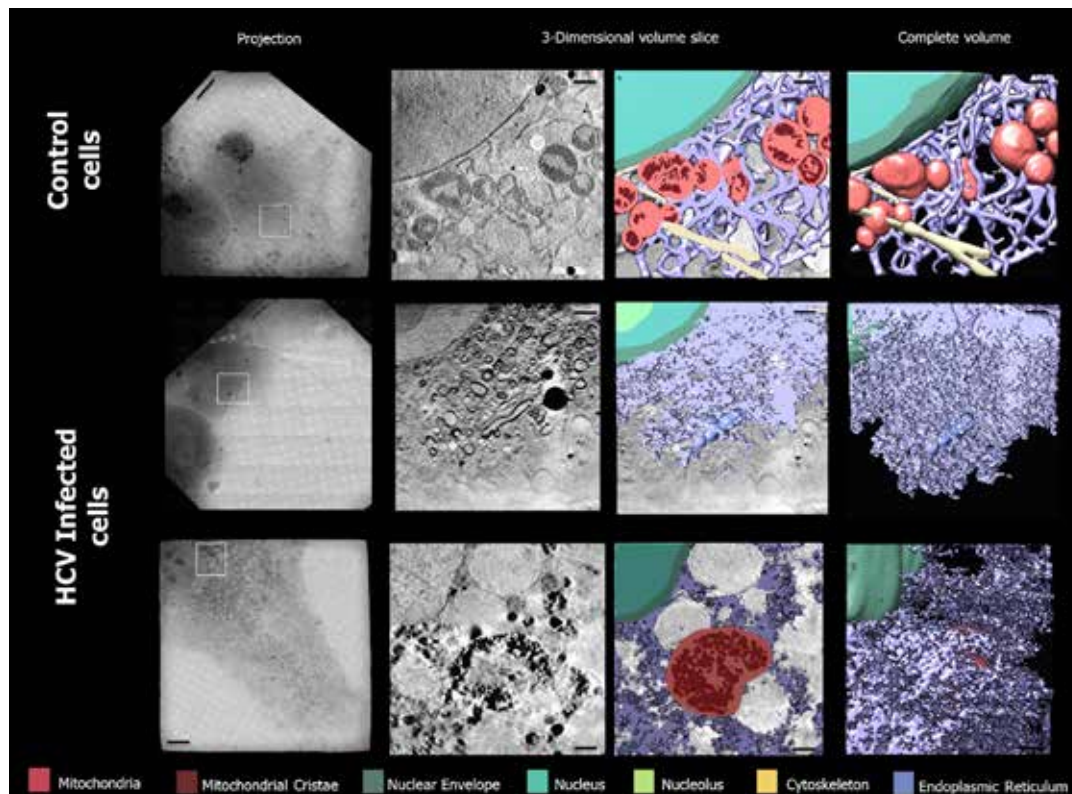


Figure 6: X-ray images of a human hepatic cell (a), 24hours infected cells with Hepatitis C virus (b) and chronic Hepatitis C replicating cell (c). Left panels: Projection Images: Cryo X-ray microscopy 0° projection image of the cell (zone plate objective with drn = 40 nm, effective pixel size 12 nm). The area enclosed for the tomography is marked in white square. Middle panels: 3-Dimensional Volume slice: Represent the central planes of the X-ray tomographic reconstructions. The central plane of the volumes is segmented to visualize cellular structure. Right panels: The 3D data corresponding to the whole cell with all the organelles segmented in different colors.

## Researchers prove the effectiveness of a new drug against malaria

### *BL-13 XALOC*

Researchers from the Universitat Politècnica de Catalunya - BarcelonaTech (UPC), the Institute of Medical Chemistry (IQM-CSIC) and the University of Glasgow have proved that the CD27 drug is a true alternative against malaria. They have analysed the crystalline structure of the DNA with the drug by performing X-ray diffraction experiments at the ALBA Synchrotron.

● An international group of researchers, led by Lourdes Campos, from the Department of Chemical Engineering at the Universitat Politècnica de Catalunya - BarcelonaTech (UPC), has proved that the CD27 drug can be a reliable option against malaria. Researchers came to this conclusion after studying the 3D crystalline structure of the complex of DNA with the drug.

The CD27 drug is a complex synthesized by researchers led by Christophe Dardonville at the Institute of Medical Chemistry of the Spanish National Research Council (IQM-CSIC), in Madrid. The UPC research group in Crystallography, Structure and Function of Biological Macromolecules (MACROM), led by Lourdes Campos, had been working for over a year to obtain the crystalline structure of DNA. In the next phase, researchers analyzed the crystals of the complex of DNA with the drug using X-rays at the XALOC macromolecular diffraction beamline of the ALBA Synchrotron. When MACROM researchers solved the 3D structure, they identified the details of CD27's structure enabling the drug to recognize the regions of DNA covering the minor groove and preventing the development of the parasite. At the same time, these studies contribute to a rational design of new drugs bearing in mind the molecular interactions caused by CD27.

Results of this research have been validated and deposited at the Protein Data Bank, and published in the journal *Acta Crystallographica D*.

The drug is patent-free and can be produced by any pharmaceutical company interested in its development.

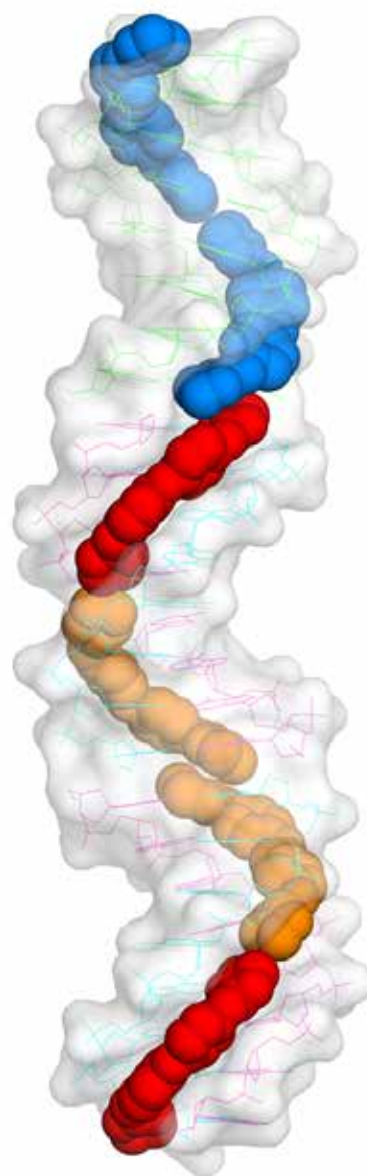


Figure 7: The drug CD27 completely covers the minor groove of the DNA complex.

**Reference:** "In and out of the minor groove: interaction of an AT-rich DNA with the drug" Francisco J. Acosta-Reyes<sup>1</sup>, Christophe Dardonville<sup>2</sup>, Harry P. de Koning<sup>3</sup>, Manal Natto<sup>3</sup>, Juan A. Subirana<sup>1</sup> and Lourdes Campos<sup>1</sup>  
*Acta Crystallographica Section D Biological Crystallography* 70 1614-1621 (2014).  
 DOI: 10.1107/S139900471400697X

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<sup>2</sup> Instituto de Química Médica, IQM-CSIC,

<sup>3</sup> Institute of Infection, Immunity and Inflammation, College of Medical, Veterinary and Life Sciences, University of Glasgow

## MATERIALS SCIENCE

# Scientists analyse the behaviour of rare-earth orthovanadates under high pressure

BL-04 MSPD

X-ray diffraction experiments were performed at the ALBA and Elettra synchrotrons. This material may have applications as a photocatalyst for hydrogen production, improving renewable energy sources. This work has been highlighted in the *Journal of Physics Condensed Matter*. It has been also chosen for IOPselect.

● Rare-earth orthovanadates are technologically important materials with applications in lithium ion batteries and also as scintillators or photocatalyst materials. In particular they have attracted considerable interest due to potential applications in hydrogen production, offering cleaner energy solutions.

A group of international researchers from India, Spain and Mexico have studied the structure and properties of the rare-earth orthovanadate  $\text{HoVO}_4$  under high pressure conditions. In this study, researchers combined experimental and theoretical work to determine the structure of the high

pressure phases of  $\text{HoVO}_4$ , one of the less studied orthovanadates under high pressure. X-ray diffraction experiments were performed at the MSPD beamline (Materials Science Powder Diffraction) of the ALBA Synchrotron and at the XRD1 beamline in Elettra. Results revealed how, under high pressure conditions, this material has the following structure sequence: zircon  $\rightarrow$  scheelite  $\rightarrow$  fergusonite. Calculations fully agreed with the experiments, showing a high consistency, and suggesting the presence of another phase transition at higher pressure ( $P > 32$  GPa). By squeezing orthovanadates at pressures hundreds of thousand times

greater than atmospheric pressure their structural and mechanical properties were studied, improving the knowledge of the physical properties of other materials having the orthovanadate structural arrangement.

**Reference:** "High-pressure structural behaviour of  $\text{HoVO}_4$ : combined XRD experiments and ab initio calculations" Alka B. Garg<sup>1</sup>, D. Errandonea<sup>2</sup>, P. Rodríguez-Hernández<sup>3</sup>, S. López-Moreno<sup>4</sup>, A. Muñoz<sup>3</sup> and Catalin Popescu<sup>5</sup> *Journal of Physics: Condensed Matter* 26 265402 (2014). DOI: 10.1088/0953-8984/26/26/265402

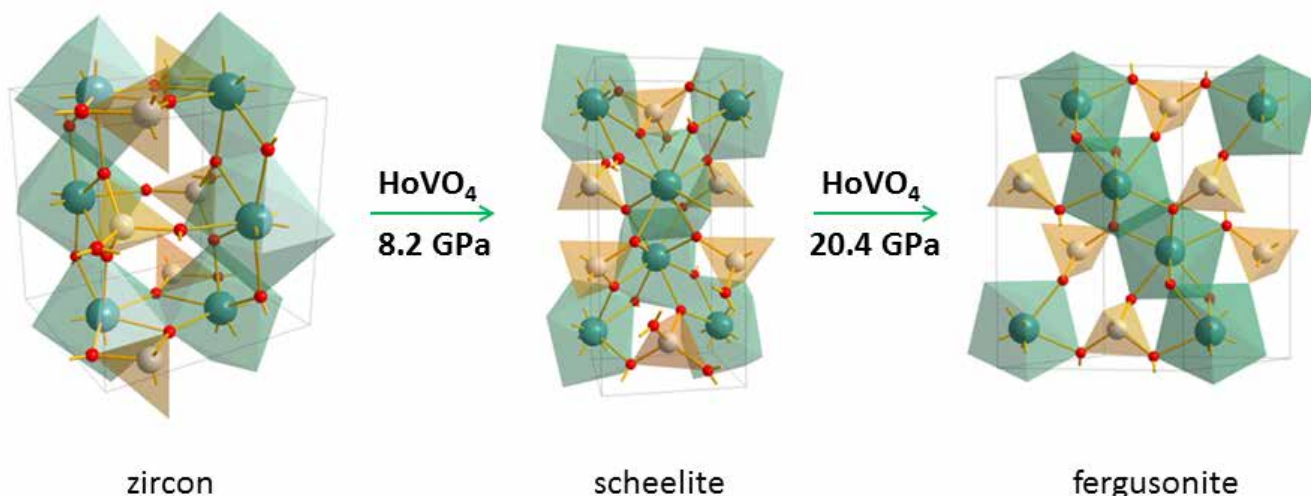


Figure 8: Structure sequence of the  $\text{HoVO}_4$  under high pressure conditions.

<sup>1</sup> High Pressure and Synchrotron Radiation Physics Division, Bhabha Atomic Research Centre

<sup>2</sup> Departamento de Física Aplicada-ICMUV, MALTA Consolider Team, Universidad de Valencia

<sup>3</sup> Departamento de Física, Instituto de Materiales y Nanotecnología, and MALTA Consolider Team, Universidad de La Laguna

<sup>4</sup> Escuela Superior Cd. Sahagun, Universidad Autónoma del Estado de Hidalgo

<sup>5</sup> ALBA Synchrotron



# Multiscale structural characterisation of yarn-like CNT fibres by synchrotron X-ray diffraction

*BL-11 NCD*

One of the most promising routes to exploit the axial properties of carbon nanotubes or graphene is to assemble them into a macroscopic fibre, with the graphitic layers aligned parallel to each other and to the fibre axis.

● At IMDEA Materials Institute, this assembly is carried out by directly spinning continuous macroscopic fibers made up of carbon nanotubes (CNT) of a controlled number of layers, ranging from singlewalled to multiwalled.

CNT fibres at different length scales have a unique porous yarn-like (hierarchical) structure, with the subfilaments being the nanotube bundles (Figure 9). The high internal surface area ( $270 \text{ m}^2/\text{g}$ ) of this comparatively open structure is readily accessible to liquid/polymer molecules; liquid/polymer infiltration occurs without applying pressure and is simply driven by capillary forces, thanks to its yarn-like structure.

Composite properties of interest (such as load transfer) are clearly expected to be influenced by the degree of infiltration of molecules into these fibre yarns across different length-scales. Synchrotron X-ray diffraction recording simultaneously SAXS and WAXS patterns in NCD beamline, as shown in Figure 10, is essential to provide multi-scale structural information about CNT fibres, ranging from a few microns to a few angstroms.

By monitoring the structural changes during the ingress of liquid into dry CNT fibre, it is expected to better understand some of the key issues when the yarn-like fibre gets in contact with polar liquid. A case study of N-Methyl-2-pyrrolidone (NMP) infiltration into the CNT fibres as an example is illustrated in Figure 11. Interestingly, there is no evidence that the scattering peak from 002 CNT plane of inter-graphene layers is affected in scattering vector  $q$  value upon NMP infiltration, indicating that neither intercalation nor debundling of CNTs from bundles takes place upon interaction with liquids. From a larger scale point of view, a decreased value of invariant is found in the NMP infiltrated fibre, compared with dry CNT fibre, which suggests that NMP molecules are able to penetrate into the voids of the CNT fibre as they have a higher electron density than air. The infiltration of liquid molecules into the fibre changes its bulk electrical conductivity due to the interplay between a higher electron tunneling probability and a slight fibre swelling, overall making an interesting sensor of liquids and polymers.

This research based on WAXS and SAXS analysis forms an integral part of various scientific and industrial projects of the research group, as well as four PhD dissertations currently being prepared at IMDEA Materials. It is expected that the results of this work will also contribute to the understanding of composites with graphene and other nano-scale reinforcements, which are also characterized by imperfect packing and therefore substantial porosity.

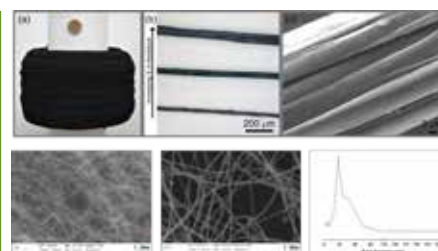


Figure 9: Images of macroscopic fibres of at different length scales. The porosity of the fibre is demonstrated by high resolution micrographs and BJH desorption measurement.

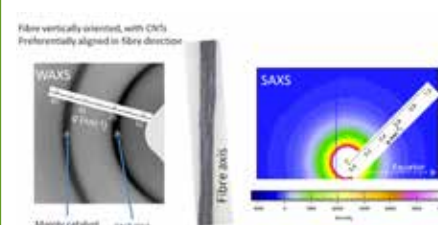


Figure 10: 2D WAXS&SAXS patterns of CNT fibres as-made showing multi scale structural information.

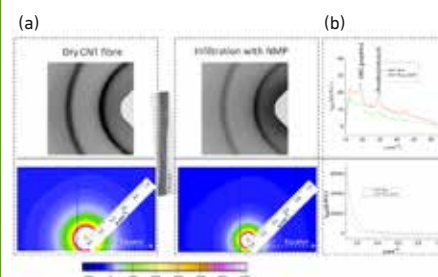


Figure 11: 2D WAXS patterns (a) of CNT fibres infiltrated with liquid N-Methyl-2-pyrrolidone (NMP), together with integrated scattering patterns (b) after azimuthal integration.

## MATERIALS SCIENCE

## Remote plasma-enhanced chemical vapor deposition (rPECVD) of graphene on various substrates

BL-24 CIRCE

Marc González Cuxart from the Universitat Autònoma de Barcelona (UAB) synthesized multiple layers of graphene films and vertical graphene nano-sheets on several substrates by means of low-pressure RF plasma in order to enhance the typical chemical vapor depositions (CVD).

● In the context of the “Nanotechnology and Material Science” master and under the supervision of Eric Pellegrin and Igors Šics (ALBA Synchrotron) and Alejandro Goñi (ICMAB-CSIC), Marc González Cuxart synthesized multiple layers of graphene films (Figure 12) and vertical graphene nano-sheets on several substrates, such as HOPG, Ni(111), Ni foils and 6H-SiC(0001) by means of low-pressure RF plasma in order to enhance the typical chemical vapor depositions (CVD).

As a first approach to this technique carried out in ALBA, remote plasma was used to decouple the dissociation process of the gas from the growth process of the graphene on the substrate, to reduce the growth temperature compared to conventional CVD, and also to minimize the effect of the plasma electrical field on the orientation of the grown graphene films.

In order to assess the quality of the graphene layers, a systematic characterization process based on a sequence of several characterization tools (Raman spectroscopy, XPS, AFM and SEM) and a subsequent detailed cross-check study was carried out, including a comparison with other carbon allotropes (Figure 13). Furthermore, LEEM, PEEM and LEED measurements were carried out at the CIRCE beamline for a more complete analysis.

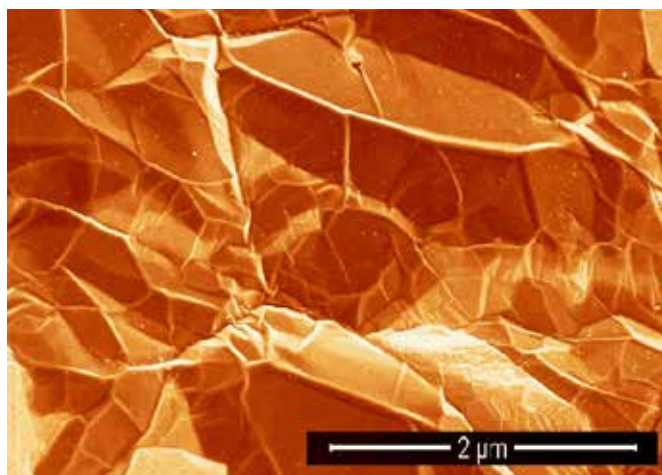


Figure 12: SEM image of a graphene sample grown on Ni foil revealing a semi-transparent “shrink wrap” appearance (SEM images taken at ICN2).

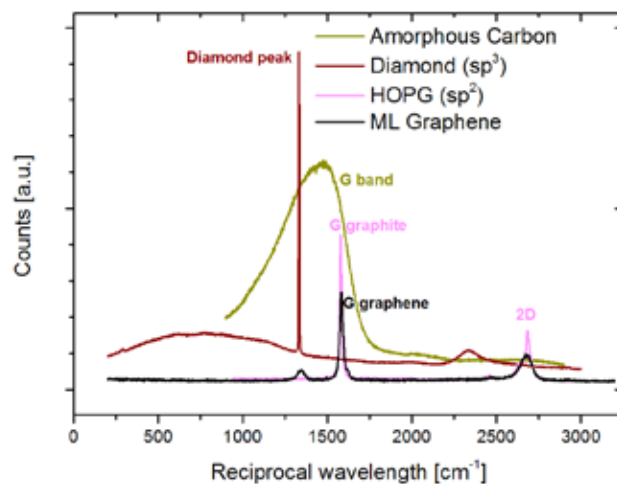


Figure 13: Comparison between Raman spectra corresponding to different allotropes and one of our graphene samples synthesized on Ni foil (Raman data acquired at ICMAB-CSIC).

## MAGNETISM

## Shedding light onto the mechanisms underlying the in-plane current induced magnetization reversal of magnetic tunnel junctions

BL-29 BOREAS

In this work published in *Physical Review B*, a group of international researchers from Switzerland, Spain, Italy and France have studied the so-called “spin-orbit field” mechanisms for in-plane current induced magnetization reversal of magnetic tunnel junctions (MTJs), and their optimization.

● MTJs are planar magnetoresistance devices through which the flow of an electron current experiences a large or low resistance depending on the magnetic configuration of the device, and with the novelty of exploiting the Quantum Tunnelling of electrons through an insulating layer with a thickness at the scale of a nanometer (i.e. of only a few atoms). These “spin-orbit fields” (or torques) originate from the exchange of angular momentum between the crystal lattice and the magnetization, and provide a route to control the magnetization of a single ferromagnetic layer without transferring spin momentum from a second ferromagnet. The detailed mechanisms giving rise to such spin-orbit torques are still debated, and this

investigation aimed to shed some light on such phenomena.

The main investigation via transport measurements was complemented using the BOREAS beamline for Soft X-Ray Absorption Spectroscopy and Magnetic Dichroism (XAS, XMCD) at ALBA Synchrotron, to trace at the atomic level the evolution of the sample properties upon thermal annealing leading to the enhanced response. The study results evidence that transverse and longitudinal spin-orbit fields occurring on Ta/CoFeB/MgO trilayers have a complex behaviour depending on the annealing history of the sample and the magnetization direction. X-ray measurements were important to establish that this behaviour is related

to the chemical and structural changes taking place in the samples, inducing significant variations of the magnetic moment and anisotropy of the layers.

**Reference:** “Fieldlike and antidamping spin-orbit torques in as-grown and annealed Ta/CoFeB/MgO layers” Can Onur Avci<sup>1,2</sup>, Kevin Garello<sup>1,2</sup>, Corneliu Nistor<sup>1,2</sup>, Sylvie Godey<sup>2</sup>, Belén Ballesteros<sup>2</sup>, Aitor Mugarza<sup>2</sup>, Alessandro Barla<sup>3</sup>, S. Manuel Valvidares<sup>4</sup>, Eric Pellegrin<sup>4</sup>, Abhijit Ghosh<sup>1</sup>, Ioan Mihai Miron<sup>5</sup>, Olivier Boulle<sup>5</sup>, Stephane Auffret<sup>5</sup>, Gilles Gaudin<sup>5</sup>, and Pietro Gambardella<sup>1,2</sup> *Physical Review B* 89 214419 (2014). DOI: <http://dx.doi.org/10.1103/PhysRevB.89.214419>

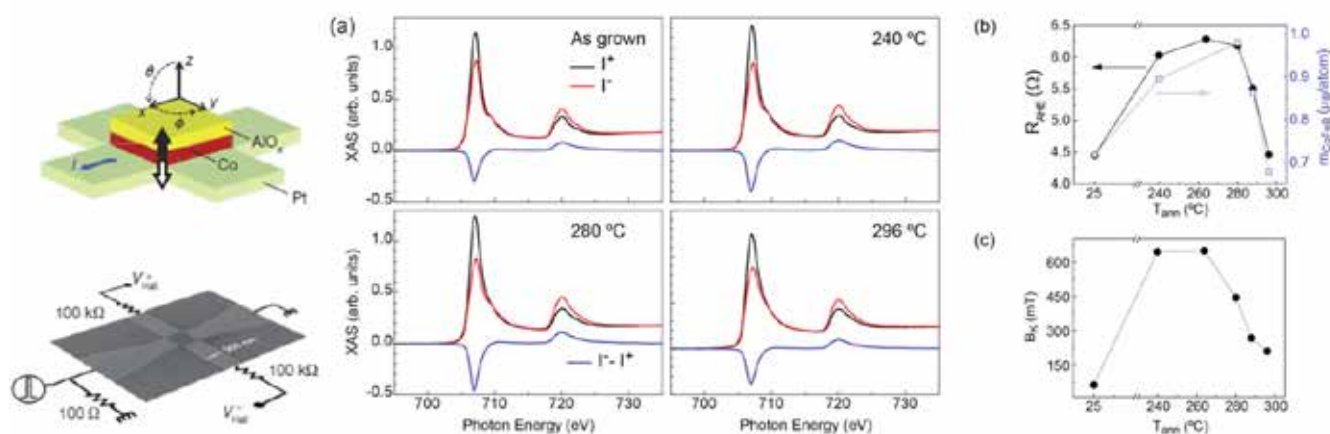


Figure 14: Left (top) Device schematic with current leads for in-plane current-induced switching studies; (bottom) scanning electron micrograph of the sample showing lithographic electric terminals and circuitry required for Hall measurements. Middle and Right: (a) XAS and XMCD of as-grown and annealed Ta/CoFeB/MgO trilayers measured at the Fe L<sub>2,3</sub> edges (b) Correlation between anomalous Hall resistance and magnetic moments measured by XMCD (c) Anisotropy field versus annealing temperature (taken from Phys. Rev. B 89 214419 (2014)).

1 Department of Materials, ETH Zurich  
 2 Catalan Institute of Nanoscience and Nanotechnology (ICN2)  
 3 Istituto di Struttura della Materia (ISM)  
 4 ALBA Synchrotron  
 5 SPINTEC

## MAGNETISM

# Expanding magnetic imaging techniques at the CIRCE XPEEM

### BL-24 CIRCE

#### X-Ray Magnetic Linear Dichroism

● Using a ferromagnetic  $\text{Fe}_3\text{O}_4$  natural single crystal, first images with X-ray magnetic linear dichroism contrast (XMLD) have been obtained in Photo Emission Electron Microscopy (PEEM) at CIRCE (see figure 15). Although the XMLD effect is typically much weaker in ferromagnetic materials than the more commonly used circular dichroic effect (XMCD), it is a unique tool when it comes to the investigation of antiferromagnetic systems without net magnetic moment. L. Martin, M. Monti, J. Marco, and J. de la Figuera from Instituto de Química Física Rocasolano (CSIC) used complementary imaging techniques to understand the effect of the surface on the measured spin and orbital magnetic moments.

#### Out-of-plane magnetization and in-situ magnetic fields

● Matteo Cantoni, Lorenzo Baldrati, Christian Rinaldi and Riccardo Bertacco from the Politecnico de Milano have used the CIRCE-PEEM endstation to investigate magneto-electric devices based on a single, 1.1 nm thin, CoFeB layer with perpendicular magnetic anisotropy (PMA) on ferroelectric  $\text{BaTiO}_3$  (see figure 16). A new sample holder with a built-in electromagnet was used for in-situ magnetization reversal of the samples, allowing us to take images in different remanent states and with a magnetic field applied. Due to the near grazing X-ray beam incidence in the PEEM, the realization of out-of-plane magnetic contrast is more challenging than in-plane contrast. Future studies will address the influence of the ferroelectric  $\text{BaTiO}_3$  on the magnetic switching of the top CoFeB structure.

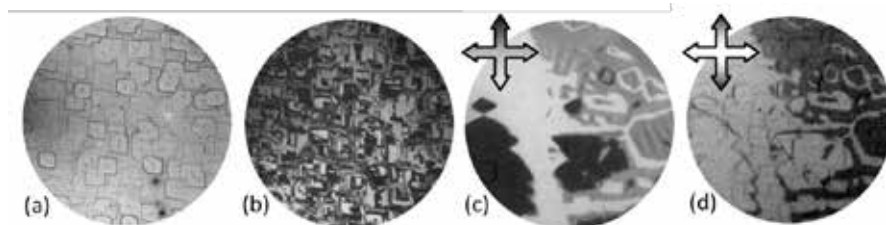


Figure 15: Images of the same area based on different contrast mechanisms (field of view 20  $\mu\text{m}$ ): (a) Surface topography by LEEM, (b) Surface reconstruction domains by dark-field LEEM, (c) Magnetic domains by XMCD at the Fe-L3 absorption edge, and (d) Magnetic domains by XMLD at the Fe-L2 absorption edge. The dominating four-fold anisotropy of the  $\text{Fe}_3\text{O}_4$  crystal can be appreciated. The intensity grey scale in (c) and (d) corresponds to the magnetization orientation relative to the incoming X-ray beam as indicated by the inset arrows. For the same magnetization pattern, the different angle dependence of the XMLD effect results in the same intensity for antiparallel domains.

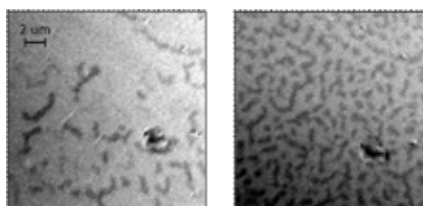


Figure 16: XMCD-PEEM images at the Fe-L3 absorption edge of a CoFeB layer in two different remanent states, exhibiting the typical worm-like domains of PMA materials. The grey scale contrast corresponds to the direction of the perpendicular magnetization (up or down), and arises from the different projections on the photon beam illuminating the sample under  $16^\circ$  grazing incidence.

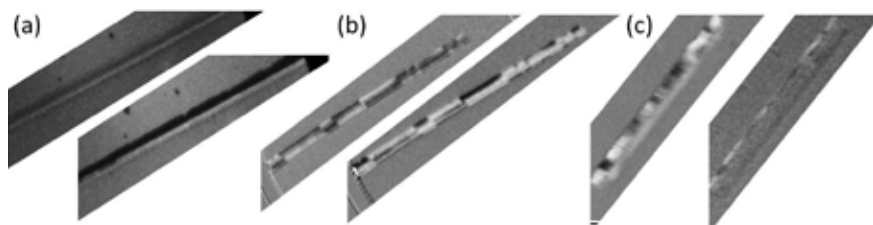


Figure 17: (a) Direct images from a crystalline Co nanowire before (left) and at the Co-L3 absorption edge showing the strong shadowing effect at the resonant energy (b) XMCD images at the Co-L3 (left) and L2 (right) absorption edges. In the shadow region, emission from the Si substrate reflects the magnetic dependent attenuation through the wire depth and thus its internal domains (see J. Kimling et al, Phys. Rev. B 84, 174406 (2011)). (c) XMCD images at slightly different photon energies separately highlight the bulk and surface magnetic structure, due to partial oxidation of the latter.

#### Transmission XMCD-PEEM

● M. Vázquez, R. Pérez del Real, C. Bran, E. Palmero, A. Fraile and A. Asenjo from Instituto de Ciencia de Materiales de Madrid (CSIC) and Universitat de Barcelona used XMCD-PEEM to study the magnetic structure of nanowires with modulated composition and width (see figure 17). Thanks to the photon beam incidence angle, electrons emitted from the nanowires' shadow provide information on its magnetic depth profile far beyond the electron escape depth due to magnetization-dependent beam attenuation.

# The many electronic facets of praseodymium-based cobaltites

BL-29 BOREAS / BL-22 CLÆSS

Cobalt oxides display a plethora of interesting properties like other strongly correlated transition metal oxides but are still poorly known. In addition, they present exceptional electronic and magnetic features related with the ability of Co ions to change their spin state as a function of temperature, applied pressure or laser excitation. This ingredient has a direct influence on unconventional conductivity and magnetic properties and makes them promising for spintronic applications. The unexpected spin-state-induced valence transitions of Pr and Co recently found by our team on  $(\text{Pr,Ln,Ca-Sr})\text{CoO}_3$  cobaltites containing Pr are restimulating the field.

● In this paper, which is the result of a collaboration between local staff at ALBA and ESRF synchrotron sources, and scientists from the Institute of Materials Science of Barcelona (ICMAB-CSIC), the authors extend their previous studies on an abrupt Pr-O hybridization in  $\text{Pr}_{0.5}\text{Ca}_{0.5}\text{MnO}_3$  (PCCO) to investigate  $\text{Pr}_{0.5}\text{Sr}_{0.5}\text{CoO}_3$  (PSCO). PSCO presents an anomalous magnetostructural transition at 120K, and the possible hybridization of Pr 4f and O 2 p states is discussed making use of different experimental techniques.

The complementary X-ray absorption spectroscopy (XAS) data recorded at the BOREAS and CLÆSS beamlines served to complete the information provided by neutron diffraction, which had revealed unexpected structural/magnetic changes at  $T_s=120$  K, and a strong contraction of some Pr–O bonds.

Surprisingly, while the Pr–O bonds contraction in the relative  $\text{Pr}_{0.5}\text{Ca}_{0.5}\text{CoO}_3$  oxide is accompanied by the striking appearance of  $\text{Pr}^{4+}$  at low temperatures as also shown by the same authors using XAS at Pr  $M_{4,5}$  edges in a recent work, soft and hard X-ray absorption spectra rule out any relevant thermally driven Pr ions electronic change in PSCO. Structural data do however point to an active participation of Pr ions to catalyze the transition (which vanishes replacing Pr by another lanthanide). Co  $L_{2,3}$  edge and O K edge XAS spectra confirm the stability of the Co spin-state below  $T_s$ , in contrast to the behavior of PCCO at low temperature. In view of the large density of empty  $t_{2g}$  - symmetry states observed, the overall metallic behavior even below  $T_s$  is attributed, in contrast to PCCO, to a (nearly temperature-independent) mixture of  $\text{Co}^{3+}$  ions in the intermediate- or high-spin configuration together with  $\text{Co}^{4+}$  ions in a low- or intermediate-spin state.

**Reference:** "Stability of the cationic oxidation states in  $\text{Pr}_{0.5}\text{Sr}_{0.5}\text{CoO}_3$  across the magnetostructural transition by X-ray absorption spectroscopy" Jessica Padilla-Pantoja<sup>1</sup>, Javier Herrero-Martín<sup>2</sup>, Pierluigi Gargiani<sup>2</sup>, S. Manuel Valvidares<sup>2</sup>, Vera Cuartero<sup>2,3</sup>, Kurt Kummer<sup>3</sup>, Oliver Watson<sup>3</sup>, Nicholas B. Brookes<sup>3</sup>, and José Luis García-Muñoz<sup>1</sup>  
Inorganic Chemistry 53 (17) 8854 - 8858 (2014)  
DOI: 10.1021/ic403117j

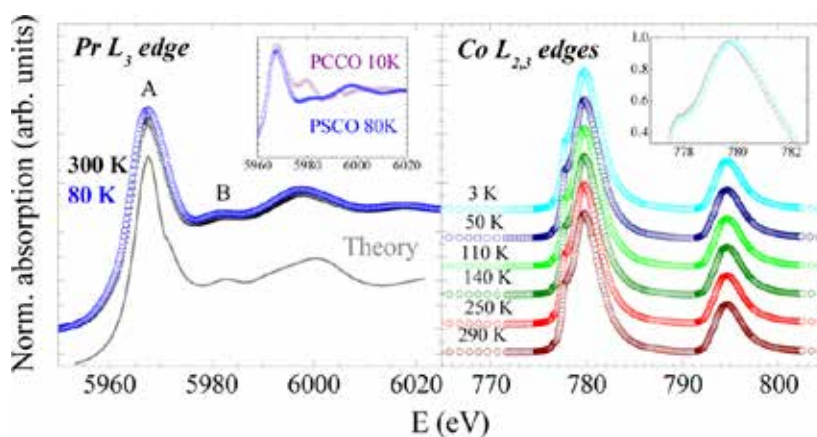


Figure 18: Left: Pr  $L_3$  edge XAS measured spectra of PSCO at  $T > T_s$  and  $T < T_s$  as compared to the calculated curve following multiple scattering theory. The inset shows a comparison between PCCO and PSCO in low-temperature phases. Right: Pr  $L_3$  XAS of PSCO above and below  $T_s$  as compared to a multiple-scattering theory calculation. In the inset the low T phase spectra of PSCO and PCCO are shown, where the large shoulder (B) in the latter around 5980 eV indicates the presence of  $\text{Pr}^{4+}$ .

1 Institut de Ciència de Materials de Barcelona (ICMAB-CSIC)

2 ALBA Synchrotron

3 European Synchrotron Radiation Facility

# Installation of the new sample transfer system

## BL-29 BOREAS

The interstation sample transfer system (SPTR) - see Fig 19 - is an UHV interconnection system built between HECTOR and MARES endstations (not shown). It connects the endstations with each other and with a third chamber/system either brought by users or to a (currently under design) satellite surface-preparation chamber for BL29, allowing to transfer a sample mounted on a standard Omicron/Specs type plate between each system in UHV conditions.

- Users willing to attach their specific preparation/analysis system to the beamline will be able to easily and quickly transfer the samples in UHV to each endstation. Moreover the SPTR offers a docking port for the ALBA-vacuum suitcase allowing transferring samples within a wider range of systems.

The first official user group using the SPTR was in October 2014 when the system was used to couple HECTOR to an STM chamber (including sample preparation facilities) brought by the ICN-Atomic Manipulation Spectroscopy group (A. Mugarza, G. Ceballos), in order to characterize with the STM microscope the samples prepared before transferring them to the HECTOR measurement chamber.

A big acknowledgement goes to everyone who worked hard on the installation: ALBA engineering division (A.C., C.C.) for design of the SPTR system together with scientific staff (P.G., M.V., E.P., J.H.), alignment group (M.L., J.L.), vacuum group (J.N., L.G.), ALBA workshop and crane team (J.F., O.B., K.M.), electronics (X.S., X.F.) and BL technicians (F.F., R.V.) who put a lot of effort to mount the whole system. Further acknowledgements go to VG-Scienta that manufactured the rack-and-pinion system at the base of the SPTR, Trinos Vacuum that built the three vacuum chambers, Ferrovac, Specs, VAT, MDC vacuum and Avactec for all the other manipulators, bellows, valves and UHV parts, Recam Laser and Blumeprot that manufactured the small parts of the system and Ambar that contributed to the electrical cabling of the endstation.

Figure 19: Boreas beamline after the SPTR installation: A) HECTOR vector magnet with preparation chamber, B) SPTR system. Right side panels: on top, a picture taken during installation and a close-up of the transfer carriage with the STM plate placed inside; beneath, the rack with the traveler entering the HECTOR preparation chamber.



# ENANTIA uses ALBA's X-rays to detect crystalline impurities in drug products

ENANTIA is a private company dedicated to research in organic synthesis and solid forms for the pharmaceutical and fine chemical industries.

● The majority of drugs are administered as solids and solid-state properties influence significantly their performance. The optimal solid form should have chemical and crystalline stability, the right pharmacokinetic profile and be easy to process. When the solid active ingredient exhibits crystalline structure transformations, they need to be taken into account for performance and intellectual property considerations. In the case of generic drugs, the presence or absence of a specific polymorph, and/or the formation of specific hydrates or solvates could determine the feasibility of launching a certain product.

Enantia collaborates with pharmaceutical companies in the identification and detection of different crystalline phases in drug substances and products. Regarding the equipment of choice to undertake such projects, ALBA Synchrotron beamline BL04-MSPD (Materials Science and Powder Diffraction) is the perfect complement to Enantia's state of the art X-ray diffractometer.

In comparison with conventional radiation, synchrotron light is better as it is much more intense and monochromatic, and its wavelength can be finely tuned. This implies an enhanced signal-to-noise ratio (so smaller peaks can be detected), a shortening of the measurement time, the possibility of acquiring data from very small amounts of sample, and the possibility of discerning close peaks alongside with a more precise determination of peak intensity ratios.

Hence, Enantia is currently using the synchrotron light to detect small amounts of chemical impurities and undesired polymorphs/hydrates/solvates (depending on the structure, detection of a 0.05% content of a "contaminant" phase is possible). Kinetic studies when dealing with unstable samples, determination of the crystal structure from powder diffraction (when single crystals are not available) and analysis of small sized single crystals can also be performed.

[www.enantia.com](http://www.enantia.com)  
[info@enantia.com](mailto:info@enantia.com)

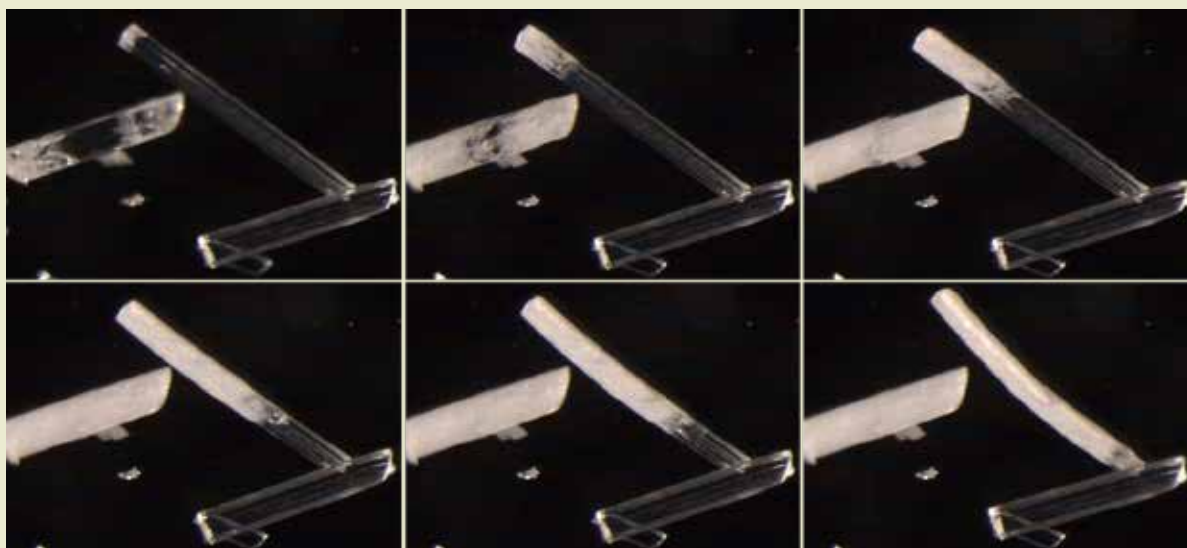


Figure 20: Crystalline phases can undergo structural transformations under changing conditions.

## More than 30 companies attended the ALBA industry workshop

Renowned companies working in the area of Chemistry and Materials Science, like CEPESA, ERCROS or AC MARCA group, were among the attendees.

● The aim of the workshop organised by ALBA last April was to illustrate the industrial applications of the ALBA Synchrotron in the field of Chemistry and Materials Sciences to the almost 50 registered participants.

During the event different scientists from ALBA explained how the current ALBA beamlines can be used to solve complex structures of cement, pigment or food components or how synchrotron light can help to promote a cleaner energy (through catalytic processes to control vehicle emissions or developing new fuel cells). It is also worth noting the role that synchrotron light can play to improve cosmetics and other healthcare products such as shampoo, soap, etc. "Synchrotron light offers analytical solutions to companies for saving time and money due to the fact that some experiments can be very short – in some cases, just one day – and with non-destructive methods", says Miguel Ángel García Aranda, scientific director of ALBA.

Avelino Corma, founder and researcher of the Instituto de Tecnología Química de Valencia (ITQ), gave an enlightening

talk on how synchrotron light is helping in the development of new chemical processes of industrial interest. He showed several cases of success directly related to his investigation.

The last speaker was Kan Wei Chou, researcher funded by Henkel, who explained how the German multinational company uses synchrotron-based research to develop new adhesive materials.

All the attendees had the opportunity to visit the experimental hall of ALBA and the beamlines' equipment and capabilities. Companies and scientists could also discuss about future industrial experiments to be performed in ALBA.

The ALBA Synchrotron management considers that this activity has been very positive. "This event shows the mutual interest between the industry and the synchrotron. ALBA is a large scientific infrastructure available to the Spanish industry", says Alejandro Sánchez, responsible of the Industrial Office of ALBA.



Attendees at the Maxwell auditorium during the beamline talks.



Networking coffee with scientists and companies.



Visit to the experimental hall and beamlines.



Inside the CLAESS beamline.



## What's on? Agenda of events

2015  
FEBRUARY  
16<sup>th</sup>

Opening ceremony of the International Year of Light 2015 in Spain

2015  
APRIL  
21<sup>th</sup> - 24<sup>th</sup>

EuCARD-2 2nd Annual Meeting

2015  
MAY  
9<sup>th</sup>

ALBA Open Day

2015  
JUNE  
15<sup>th</sup> - 16<sup>th</sup>

Diagnostics Experts of European Lightsources

2015  
JUNE  
16<sup>th</sup> - 19<sup>th</sup>

ALBA Users Meeting and VII Meeting of the Spanish Synchrotron User Association (AUSE)

## ALBA hosted the Biostruct-X meeting

● During 29th and 30th September, members of the Biostruct-X project met at ALBA's facilities. More than 50 attendees participated in the different talks and reports, focused on the progress of the Biostruct-X project.

BioStruct-X is a project funded by the FP7 Programme of the European Commission that establishes a state-of-the-art coordinated multi-site infrastructure to support access to established and emerging key methods in structural biology.



Attendants to the annual Biostruct-X meeting held in ALBA.

## Successful course on EXAFS and XANES

● From 6th to 9th October the ALBA Synchrotron and the Universitat Autònoma de Barcelona (UAB) organised a specialization course of XANES & EXAFS for chemical speciation on environmental systems. The aim of the course was to explain basic concepts about synchrotron techniques when studying environmental aspects and biomaterials.

The course, organized by the Research Group of Separation Techniques in Chemistry, GTS, from the UAB and the ALBA Synchrotron, was a success according to professors and students. It belongs to a European project called ORQUE-SUDOE from Interreg-SUDOE program with eight different partners from France, Portugal and Spain. The aim of this project is to create an environmental observatory of the SUDOE region based on the study of pollution in the coasts of France, Portugal and Spain using biomarkers such as molluscs (oysters and mussels) which have been collected in eight different areas. Synchrotron techniques have been the key to determine metallic pollutants on these biomarkers.



54 people attended the course, coming from five different synchrotron facilities from all over the world: ESRF (France), Maxlab (Sweden), Soleil (France), PSI (Switzerland), Brookhaven (USA) and ALBA (Spain).

## Fulbrighters at ALBA

● On 2014, a group of ex-alumni from the Fulbright program visited ALBA to learn about the operation and scientific capabilities of the facility. This activity was organized by Carlos Escudero from the Experiments Division and ex-alumni from the same program.

Sponsored by the Bureau of Educational and Cultural Affairs of the United States Department of State, the Fulbright Program is an international educational exchange platform designed to increase mutual understanding between the people of the United States and the people of other countries. This program provides funding for students, scholars, teachers, and professionals to undertake graduate studies, advanced research, university teaching, and teaching in elementary and secondary schools.



Photograph taken during the visit to the ALBA Experimental Hall.

## Great success for the ALBA Open Day with more than 1.600 visitors

For the third year in a row, the ALBA Open Day has raised the interest of the general public. On Saturday 11th October, 1.630 visitors came to ALBA to discover what a synchrotron is and how it works.

● Following an itinerary of about one hour and a half, visitors had the chance to see ALBA's facilities, participate in demonstrations and experiments and talk to ALBA scientists and technicians. They also had the opportunity to observe the interior of the accelerator's tunnel which was opened on that occasion. This year, one of the novelties was a new section devoted to the transversal services of engineering, computing & controls and health & safety, where the attendants could see different devices such as gas masks, communication racks and a laser tracker, used to precisely level all the devices installed in the accelerator. The kids' area was one of the most visited and, in particular, the group mural with ALBA's logotype was one of the most successful activities. This mural will remain over the external accelerator wall.

The event organisation was possible thanks to the collaboration of nearly 100 volunteers from ALBA staff and Civil Protection volunteers, offering their support and expertise during the event. As in previous editions, tickets for the ALBA Open Day ran out before the event. Access to the event was free but required booking through the internet.

The ALBA Synchrotron has developed an outreach program, which includes the organization of the ALBA Open Day as well as guided tours inside the facility. Both activities welcome more than 5,000 visitors every year.

The ALBA Open Day was organized with the support of the Spanish Foundation for Science and Technology (FECYT), the Spanish Ministry of Economy and Competitiveness, the Catalan Government and the sponsorship of La Caixa Foundation.



Photographs@Pepo Segura



## ALBA celebrated the European Researchers' Night

On September 26th the ALBA Synchrotron organized free outreach activities for all ages. The event was held at the Museum of Art of Cerdanyola del Vallès from 18:00 till 23:00 h.



Exhibition about crystallography and synchrotrons on the occasion of the International Year of Crystallography 2014.



Researchers explained how they use synchrotron light to discover new drugs or analyze historical objects.



The Synchrotron game: very useful to explain to children how a synchrotron works.



Final party with science monologues to celebrate European Researchers' Night.

Photographs@SandraGross

● The 10th edition of the European Researchers Night took place on Friday 26th September. Simultaneous popular activities were organized in 300 cities of 26 European countries to increase the visibility of science.

ALBA's activities wanted to bring science and research to the public. Attendees participated in "Meetings with researchers", where scientists explained how the synchrotron light is generated, how it helps discover new drugs or analyze historic and artistic objects. Participants were able to know the researchers' daily work, their research and its impact on our life and they also had the chance to talk to them in person and in twitter chat sessions.

On the occasion of the International Year of Crystallography 2014, the museum included an exhibition on crystallography and synchrotrons to show the relationship between these two disciplines which contributed to numerous scientific advances in the last century.

In addition, attendees were able to participate in a demonstration about diffraction to understand how it helps us analyze the structure of matter. Finally, a drawing contest for children was organized under the title "Draw a scientist".

### A project with the support of the European Commission

The European Researchers' Night is an initiative of the European Commission as a part of the Marie Curie Actions, which is a program of the European Union to enhance European researchers' careers.

The European Researchers' Night has been celebrated on the last Friday of September every year since 2003 to promote research, encourage youth interest in science careers and offer the general public a closer vision of science and researchers.

The event organized by the ALBA Synchrotron during the European Researchers' Night is part of the activities organised by a consortium of 14 different partners from all around Spain: universities, research centers, science museums and associations.

The ALBA activities for the European Researchers' Night are cofunded by the European Commission with the support of the Town Council of Cerdanyola del Vallès.

## New ALBA website

● The ALBA Synchrotron has launched a new version of its website, with a new image and new contents. The new website is a multilingual platform (in English, Spanish and Catalan) which includes more visual resources as well as new sections such as Industry, Media and Outreach.

Visit our new website:  
[www.albasynchrotron.es](http://www.albasynchrotron.es)



## 2014 Prince of Asturias Research Award to Avelino Corma

● ALBA user Avelino Corma has been bestowed with the 2014 Prince of Asturias Award for Technical and Scientific Research together with scientists Mark E. Davis and Galen D. Stucky (USA). Avelino Corma, founder of the Instituto de Tecnología Química (ITQ-UPV, CSIC), is ranked among the 25 most-cited chemists in the world, with more than 900 articles and 120 patents. Corma and the whole ITQ-UPV team have been committed users during the development and the commissioning of the ALBA Synchrotron, with several joint collaborations.



Teatro Campoamor, Oviedo. Ceremony of Prince of Asturias Awards © FPA / Iván Martínez

## Award to the thesis of Valentí Massana

● Our colleague Valentí Massana was selected to receive the "Eduard Fontseré" award (Institut d'Estudis Catalans, IEC) for the best thesis of 2013 in the Physics area. The thesis was directed by Jordi Marcos and Pep Campmany (both from ALBA Accelerators division). The study "Optimization of the construction process for a permanent magnets undulator" of Valentí Massana is a research work focused on the inhomogeneities of permanent magnets that are used to construct insertion devices in synchrotron light sources, especially undulators. The thesis had two main objectives: to model the effect of these inhomogeneities on the magnetic fields generated by magnets and to find new ways of minimizing the effects of inhomogeneities in the insertion devices.

## Supporting young talents

● Caterina Biscari has been the graduation supporter of the 3<sup>rd</sup> promotion of Inno+Talent25, a programme launched by HP, La Salle Campus Barcelona University and Leitat Technological Centre. This program is based on a dual training system to promote young talents from scientific and engineering careers.

In the graduation ceremony, Caterina Biscari commented that a larger collaboration is needed between public and business research centres to enhance the promotion of young talents.



Members of the 3<sup>rd</sup> promotion of Inno+Talent25 © HP

## Ian Robinson awarded Gregory Aminoff Prize 2015

● Ian Robinson, member of the ALBA Scientific Advisory Committee, has been awarded by the Royal Swedish Academy of Sciences in recognition of his development of diffraction methods for studying surfaces and nanomaterials.

Ian Robinson is professor at the London Centre for Nanotechnology, University College London and BBSRC Professorial Diamond Fellow at RCaH and member of the Scientific Advisory Committee of ALBA, which he will chair from June 2015.



## Best final grade project

● On 23<sup>rd</sup> October, the Escola Universitària Salesiana de Sarrià (EUSS) celebrated the graduation of the 18<sup>th</sup> year of engineers. Ricard Núñez, former member of the Accelerators division, received an award during the ceremony.

His final grade project inside the Industrial and Automatized Electronics degree, which was entitled "Design of pulsed magnets for the characterization and diagnosis of the ALBA synchrotron storage ring" and directed by Montse Pont, was chosen the best final grade project of the EUSS university.

## New faces in ALBA

● During 2014, many new people have joined the ALBA synchrotron.



**JORDI ANDREU**  
Controls, Computing



**NÚRIA AYALA**  
Accelerators



**ROELAND BOER**  
XALOC Beamline,  
Experiments



**MANUEL BROSETA**  
Controls, Computing



**ALFONSO BURGOS**  
MIS section,  
Computing



**ANTONIO  
CARBALLEDO**  
Engineering



**ROGER FOS**  
Accelerators



**DAVIDE LANAIA**  
Accelerators



**CARLO MARINI**  
CLÆSS Beamline,  
Experiments



**MANUEL MURCIA**  
Engineering



**WOJCIECH OLSZEWSKI**  
CLÆSS Beamline,  
Experiments



**GABRIEL PEÑA**  
Engineering



**JORDI PRAT**  
Experiments



**VICENTE REY**  
Controls, Computing



**MARC RODRÍGUEZ**  
Systems, Computing



**ORIOL SERRES**  
Accelerators



**Mª JOSÉ SIMANCAS**  
Administration



**MARC SOS**  
Accelerators



**HARI BABU VASILI**  
BOREAS Beamline,  
Experiments



**JORGE VILLANUEVA**  
Controls, Computing



## Carmen Vela - Secretary of State for Research, Development and Innovation

Carmen Vela, member of the Governing Council of the ALBA Synchrotron, is since 2012 the Secretary of State for Research, Development and Innovation (RDI) in the Ministry of Economy and Competitiveness of the Spanish Government. We have discussed with her the future of Spanish science and the role of scientific facilities like ALBA.

**• You took office as Secretary of State for RDI in 2012, in a situation with serious economic constrains. Two years later, what is your evaluation?**

It has been a period with many difficulties and intensive work, but also with positive results.

We have approved the Spanish Strategy of Science, Technology and Innovation: the scenario where we are developing the necessary policies to build a strong RDI ecosystem that will be able to transform knowledge into wealth and welfare.

We have also worked on the corresponding annual plans, which include the calls for projects and human resources in every exercise whose periods are being normalized gradually. We have solved very difficult situations happening long time ago at the CSIC or the CNIO. We have strengthened excellence with more Severo Ochoa calls, that this year is open to research units. We have rethought our HR calls, transforming grants into contracts, creating new calls like the industrial PhD or adopting measures to make Ramon y Cajal calls more stable inside the RDI system once the 5 years grant is finished. We have widened our presence in very important international organisms and we have participated in infrastructures such as the EELT - the biggest telescope worldwide - and SESAME synchrotron facility - where we participate as observers-. We have created new collaboration programmes between the public and private sector like the CIEN and we have approved measures to promote companies in the innovation path, like the fiscal cheque or the 40% bonus contribution to the social security for researcher

### Access to scientific and technical facilities (ICTS) is one of the most important assets to keep the leadership in research

staff. We have had our system analysed by European experts. We are designing a new non-functional career for researchers with the approval of 50 contracts in public institutions of research. We have improved our participation in Europe through the programme Horizon 2020, where we are the 3rd country after Germany and the United Kingdom winning highly competitive projects. We have been involved in the design of the smart specialization strategies of the regional governments (comunidades autónomas) which might be helpful to avoid duplicities and gain efficiency. We have approved a new map of Scientific and Technical Infrastructures (ICTS). Summarizing, we have not been short of work.

**• How do you imagine the Spanish science in ten years? What measures are needed?**

In ten years, I imagine Spain with a science and innovation system similar to those of the most advanced countries. To reach this goal, we have to work on different variables such as human resources, funding, fiscal system, administrative flexibility or recognition, among other issues. These are measures that we are tackling at the State Secretary. Regarding human resources, we have a double challenge: to train in excellence and to count on an RDI system capable of absorbing the talent coming out of the classrooms. We need to implement measures that encourage profiles of innovative entrepreneurs, with creative and novel ideas. And, of course, we need to promote international mobility from the academia to the entrepreneurial world and vice versa. It is necessary to establish measures to make the collaboration between academia and companies recognized as a merit in the development of the professional career of researchers.

Another priority is to have appropriate funding, avoiding the variability occurred in the last years. We need an RDI system with a sustainable, foreseeable and stable growth that let us take the convenient decisions in a far horizon.

To achieve a beneficial fiscal system for RDI activities is another of our challenges. We, the public administration, must offer a proper environment for innovation, with a stable regulation and tax benefits.

It is also necessary to introduce measures for administrative flexibility, balancing flexibility and control (accountability).

Another important issue on which we have to make a great effort is recognition, we need the society to recognize the important role of RDI.

**• How do you think European science will be in the next years with the Horizon 2020 programme?**

Horizon 2020 programme has the biggest budget of the European Union history aimed at RDI: almost 80,000 million euros. And, for the first time, research and innovation are both

included in the same programme, which was very necessary and will be beneficial for everybody. This fact requires that research institutions, centres and universities collaborate with the business world and vice versa more closely. We did the same exercise in 2013 when we approved the Spanish Strategy of Science, Technology and Innovation that, for the first time, gathers research and innovation in the same document, without any distinction.

• **Recently, the Spanish map of large scientific infrastructures (ICTS) has been updated. What is the importance of these facilities for the Spanish research?**

The scientific and technical research of excellence needs to rely on an advanced network of infrastructures and scientific and technological equipment, and have access to first level facilities, like the ICTS. Access to those facilities is one of the most important assets to maintain the leadership in research, to increase the specialized training capability in RDI activities and to attract new talent. Additionally, the development of high competitive R&D entrepreneurial activities depends critically on the access to scientific and technological facilities.

ICTS are one of a kind. They have a very high cost of investment and maintenance and their importance and strategic character justifies their availability to all the RDI community. This is why ICTS are essential to perform excellent and cutting-edge scientific and technical research. The scientific and technological leadership, the research at the frontier of knowledge and the development of high competitive R&D entrepreneurial activities depends critically on the access to scientific and technological facilities. The National Plan of Scientific and Technical Research will support general access to the ICTS for all the agents of the Spanish National System of Science, both public and private, and will promote their scientific specialization and enable the consolidation of ICTS as one of the execution agents of the activities of the RDI system. At the same time, it will also try to reduce the territorial spread and to assure the coordination among ICTS from the same area, promoting their industrial use and the support to the Science Industry.

• **And in the case of the ALBA Synchrotron, how would you value its role within the Spanish scientific community? Are there good funding prospects for building new beamlines in the future?**

Since the beginning of its official operation in 2012, the ALBA Synchrotron has a great scientific demand that grows progressively. With its current capacity, it's giving service to more than 1,000 researchers per year and 75% of them come from Spanish institutions. These data give us an idea about the important role that this scientific facility plays in our scientific community.

The ALBA Synchrotron has seven operative beamlines and has the capacity to install 21 more. There are currently two new beamlines – MIRAS and LOREA – under construction

and design, respectively, which are expected to be available to users in 2016.

Other possible beamlines are now being defined, considering scientific and technical aspects but also the necessary funding planning. In any case, the ALBA Synchrotron is an important proof of collaboration between the central Government and the Generalitat de Catalunya, and both administrations work closely to make this ICTS realize its maximum potential.

• **Different studies point out that the quality of research in Spain is considerably high. However, knowledge transfer and innovation are still pending issues. Do you agree? Are there new actions that might be taken?**

I completely agree with that. High quality science is being performed in Spain. We are the 10th country in scientific production. And if we consider publications in journals with high impact factor, our situation is even better, according to "Nature" last index report which put Spain in 8th position. Even though our situation is improvable, we must feel proud of having researchers with worldwide impact, excellent researchers and centres which are international models. We need to do the same exercise with innovation.

First of all, we need to change our mind and take the RDI process as a path to walk through from the beginning till the end. We need to put together research and innovation, bringing research institutions, universities and companies closer and having them work together since the very first moment that the idea is generated until this idea becomes a product or a service. Besides, we need to take into account six variables already commented: appropriate funding, talent generation and ability to use it, mobility – at the public and private, national and international level –, a fiscal system favouring innovation, administrative flexibility and the recognition of the role of RDI by the society.

• **You have been actively involved enhancing the role of women in science. How would you assess the situation of Spanish women scientists? In your opinion, what further measures should be adopted?**

The situation of Spanish women scientists is quite similar to the rest of European women scientists, according to the last reports. In general terms, we are in the European average, with some differences: in Spain there are more women scientists but, taking into account the vertical integration of high positions, different indicators conclude that we are slightly under the average.

The conclusion of the European report She Figures is also applicable to the Spanish case: the higher you move along the hierarchy, the wider the gender gap gets.

Regarding the measures to be adopted, the legislative progresses have enabled the progressive approval of equality plans at universities and OPIS, as well as the introduction of gender priority in RDI strategies at the national and European levels. It is necessary to follow up and ensure the effective implementation of those plans and priorities.

**We need to put together research and innovation since the very first moment that the idea is generated until this idea becomes a product or a service**

# Merry Christmas and happy New Year! 2015

