

N°: 36 OCTOBER 2013

The ALBA Synchrotron newsletter

-

page Science
12 Highlights

**17** *Industry collaborations* 

Visitors during summer holidays

page **20** 

ALBA holds its page first User Meeting 06

# Summary

#### 4 Latest news in ALBA

#### **Main Issue**

6 First ALBA User Meeting

#### **Accelerator news**

10 ALBA works on the implementation of the Top-up mode injection

#### Science highlights

- 11 First atomic-resolution protein structures solved at ALBA
- 12 60 nm diameter magnetic bubbles imaged at Mistral
- 13 Understanding magnetic cobalt nanostructures
- 14 Pump-probe powder diffraction with a time resolution of 50µs at ALBA
- 15 Magnetic nanocrystals in GaN semiconductor measured at Circe
- 15 In situ magnesium hydration
- 16 First commissioning users at Near Ambient Pressure Photoemission (NAPP) endstation
- 16 New beamline scientists in ALBA

#### **Technical Developments**

17 Latest beamline technical progress

#### **Industry news**

- 18 Industrial developments at ALBA
- 18 Collaboration between ALBA Synchrotron and multinational company Henkel

#### 19 Agenda of events

#### **Outreach activities**

- 20 ALBA Synchrotron opens to visitors during the summer
- 20 Explaining ALBA to children in an animated cartoon series

I take the opportunity of this first issue of the renovated ALBA Newsletter to

ALBA has started its operation. The project which was started years ago by a small group of enthusiastic people has come to reality. The group has steadily grown, including now several late arrivals, among whom I have the honor to belong, and who are taking over the work of those who initiated the history of the Spanish synchrotron radiation facility.

Today we are a user facility. We have just opened the third user call. Scientists from Spain and from the rest of the world are routinely using our infrastructures, visiting the seven operating beamlines, served by a reliable

We have of course faced problems and difficulties, typical of young new infrastructures. We have still much work to realize to fully exploit what is already in our site, but in the meantime we are looking towards the future. A Strategic Plan for the next four years has been produced and will be the starting point to draw the actions for the expansion of ALBA. The already existing collaboration with the user community has been fruitfully sealed

during the AUSE and ALBA user meeting, as reported in the Newsletter. This Newsletter is the evolution of the previous format, and will be published every four months. We expect to collect in it scientific results, description of events, news on the laboratory life, information on the staff. Enjoy the reading.

Sincerely,

Caterina Biscari ALBA Director





Participants of the meeting came from CIEMAT, Centro de Microanálisis de Materiales – UAM-, Centro Nacional de Aceleradores -CNA, Sevilla-, ESS-Bilbao, IFIC Accelerator Group -Valencia-, Institut de Tècniques Energètiques – UPC, Barcelona-, Linac Research Facility – LRF, Huelva- and ALBA. Photograph: ALBA

## Spanish experts on accelerators meet at ALBA

Accelerator science and technology has considerably grown in Spain in the last years, resulting in the development of national facilities, like ALBA synchrotron, and in key contributions to the main international collaborations. like CERN, ILC, IFMIF, or XFEL. ALBA hosted the first meeting of Spanish experts on accelerators on July 2013.

Spanish experts on accelerator science and technology met for the first time at the ALBA Synchrotron. The aim of the meeting was to create a working group to increase and improve the coordination among all the Spanish researchers dealing with accelerators.

Experts on accelerator science and technology cover a wide variety of activities, dimensions and profiles, ranging from university research to the design and commissioning of different types of accelerators, such as colliders, synchrotron light sources or cyclotrons.

## ALBA hosted the PaNdata meeting

In conjunction with UmbrellaID.org and ICAT workshops and within the framework of the PaNdata collaboration project, ALBA Synchrotron held a project meeting on September 9th and 10th to discuss the open data infrastructure used in European photon and neutron laboratories. More than 30 attendees from seven different European countries participated in the meeting.

The purpose of this meeting was to review the status of the 8 different work packages belonging to the PaNdata ODI project, and a particular workshop Ithe Umbrella project, within the WP3 (AAA - Authentication/Authorization/ Accounting), took place. The status of the deployment of another work package, in which ALBA is significantly participating, the ICAT project (WP4 - Data Catalogue), was also reviewed.

PaNdata Open Data Infrastructure is a FP7 supported project created with the aim of implementing a common open data infrastructure, in particular on software and data catalogues, user identities and authentication systems or optimized data analysis methods. Formed in 2008, the PaNdata collaboration currently brings together eleven major world class European research infrastructures.

More information about the project: www.pandata.eu

"Increasing the development of the knowledge-based economy."

#### The mayor of Barcelona visits ALBA

The mayor of Barcelona, Xavier Trias, has visited ALBA's facilities. During the visit, he was accompanied by the Chairman of the Executive Commission of ALBA, Ramon Pascual, the director of ALBA, Caterina Biscari, and the Scientific Director, Miguel Angel García Aranda.

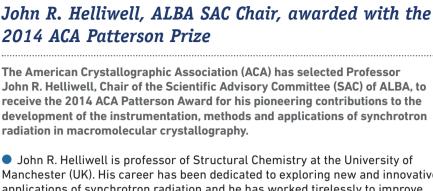
• Xavier Trias had the chance to see the experimental hall of ALBA as well as the seven different beamlines currently in operation. Jaume Ciurana, deputy mayor, and Josep Lluís Alay, Director of Museums and Heritage of Barcelona, also visited ALBA's facilities.

The mayor highlighted the relevance of a scientific infrastructure such as ALBA in increasing the development of the knowledge-based economy. Equally, ALBA managers have recognized the role of the administration in preserving quality research.

This visit is within the framework of the program for institutional relations of the ALBA synchrotron to increase awareness of the features and applications of the only synchrotron light source in Spain.



Inside the ALBA tunnel, Ramon Pascual, Miguel Ángel García Aranda, Caterina Biscari, Xavier Trias, Josep Lluís Alay and Jaume Ciurana (from left to right). Photograph: ALBA



John R. Helliwell is professor of Structural Chemistry at the University of Manchester (UK). His career has been dedicated to exploring new and innovative applications of synchrotron radiation and he has worked tirelessly to improve synchrotron and neutron facilities worldwide.

2014 ACA Patterson Prize

radiation in macromolecular crystallography.

Helliwell is currently the Chair of the Scientific Advisory Committee (SAC) of ALBA. The ALBA SAC is a board of international renowned experts in the field of synchrotron radiation, which gives advice on the strategic scientific direction of the ALBA synchrotron with the aim of ensuring the quality and relevance of the research developed in ALBA.

John R. Helliwell will receive the ACA Patterson Prize at the 2014 ACA May meeting in Albuquerque, New Mexico (USA). This Prize is presented by the American Crystallographic Association every three years in recognition of outstanding research in the area of structural matter by diffraction methods.

#### Joan Casas, new Head of the Engineering division

John R. Helliwell is professor of Structural Chemistry

at the University of Manchester (UK). Photograph: American Crystallographic Association.

From October 4th 2013, Joan Casas is the new Head of the Engineering Division at ALBA.

Joan Casas started his career in Mier Comunicaciones as RF & Microwaves design engineer twenty years ago. During the last eleven years, Joan has been managing engineering teams, developing very demanding space projects (on-board satellites microwave equipment) and Radio Frequency broadcast equipment for Digital Terrestrial Television, contributing to Mier positioning as the Spanish leading company in these two fields.

Joan will take over the excellent work done by his predecessor, Lluís Miralles, in charge of the Engineering division at ALBA, willing to have a renewed fruitful contribution. We wish him a successful period in his professional career.



Joan Casas, new head of the Engineering division. Photograph: Joan Casas

"ALBA has prepared a strategic plan for 2013-2016 which includes a progressive construction of new beamlines to attend the scientific community demands. More information here. "



Caterina Biscari, director of ALBA, at the opening ceremony of the 1st ALBA User Meeting. Photograph: Pepo Segura.

# First ALBA User Meeting

From September 3rd to 6th, ALBA Synchrotron held its first User Meeting and the VI National Meeting of the Spanish Synchrotron User Association (AUSE). During this event, researchers from different scientific areas showed the first results obtained in ALBA and discussed the applications and advantages that synchrotron light has on Spanish science.

Selenium concentrations on wheat, solar cell development or the characterization of materials are only some examples of the different areas where synchrotron light can be used. During almost one week, ALBA users exchanged different views and results about the experiments performed in the Spanish facility. Caterina Biscari, director of ALBA Synchrotron, highlighted during the opening ceremony that "the dream of creating a Spanish synchrotron is now a reality".

ALBA User Meeting was held in conjunction with the VI National Meeting of the Spanish Synchrotron User Association (AUSE). AUSE was founded in 2004 with the aim of getting in contact and informing all the scientists from Spanish institutions who use the synchrotron light in their research projects. Nowadays, AUSE has 630 researchers of different profiles: physicists, chemists, biologists, medical doctors, engineers, etc.

New this year, the 2013 AUSE Thesis Prize for the best doctoral thesis using synchrotron

radiation defended in a Spanish university between 2011 and 2012 was awarded to Dr Immaculada Martínez Rovira, who studied radiotherapy techniques using spatially fractionated dose with synchrotron radiation. The meeting also included talks of international experts, who informed about the latest news of other European facilities, and one talk dedicated to discussing the role of women in science.

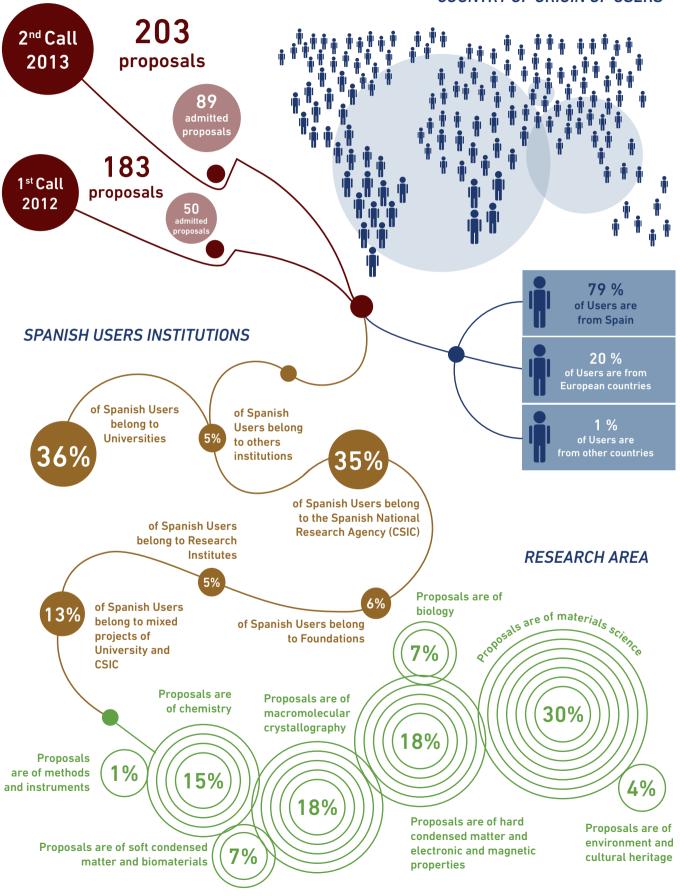
A full day session was dedicated to the applications of synchrotron light in the biomedicine sector, explaining how the synchrotron light can help in diagnose of Huntington, Alzheimer or leukemia and the structure-based drug design developed with synchrotron light.

Throughout the week, the Spanish scientific community has expressed the need of completing ALBA capacities with new beamlines to give service to the growing users' community.

ALBA Preced OCTOBER 2013- Nº 36

# Who are ALBA Users?

#### COUNTRY OF ORIGIN OF USERS



# OCTOBER 2013 - N° 36 ALBA Preces

7

Ma

Main Issue

# Spanish synchrotron user community grows up to 630

The interest in synchrotron light in Spain has been growing considerably over the last ten years. At the moment, AUSE has registered 630 Spanish users of synchrotron light while there were only 200 in 2004. On behalf of AUSE, her president María Eugenia Dávila, remarked on "the tremendous progress that synchrotron science has experimented in the last years in Spain".

The start of ALBA Synchrotron in 2012 has led to a growing usage of synchrotron light in Spanish science. ALBA's seven experimental beamlines, all part of

Phase I and in full operation at the moment, allowed 354 researchers to perform their experiments. 79% of all received proposals come from Spanish research institutions while the rest mainly come from other European countries.

Submitted research projects at the ALBA Synchrotron are mainly focused on materials sciences (30%), hard condensed matter(18%), maromolecular crystallography (18%), chemistry (15%), biology (7%), soft condensed matter (7%), environment and cultural heritage (4%) and methods and instrumentation (1%).

# New call for beamtime proposals 2014

The ALBA Synchrotron has opened its 3rd call for beamtime proposals. From October 7th to November 4th, researchers interested in performing an experiment at any of the beamlines of ALBA Synchrotron can submit a proposal. For this period, no Long Term Proposals will be accepted and all proposals for MX (BL13-XALOC beamline) with two or more principal investigators should be submitted as BAG's (Block Allocation Group). The proposal must include a summary of 300 words and a description of the experiment.

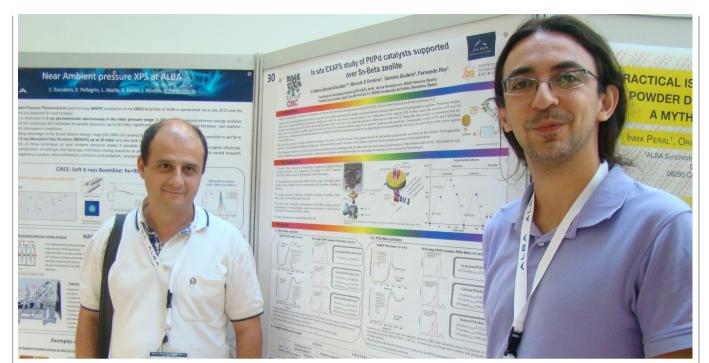
Once the proposal has fulfilled the technical requirements, it is evaluated by a panel, composed by international researchers from different research areas. The panel evaluation results on a ranking based on criteria of scientific excellence.

The resolution of this call is expected to be published in March 2014. The experiments will be performed from April to December 2014 in any of the seven beamlines of ALBA.

Researchers interested in performing an experiment in ALBA must submit a proposal through the ALBA User Office Portal: http://useroffice.cells.es/



Image of the auditorium during the 1st ALBA User Meeting. Photograph: Pepo Segura



Fernando Rey and José Luis Jordá, researchers at the Instituto de Tecnología Química de Valencia (ITQ). Photograph: ALBA.

# **Conversations with ALBA users**

Fernando Rey and José Luis Jordá work at the Instituto de Tecnología Química de Valencia (ITQ), a joint research center created by the Universitat Politècnica de València (UPV) and the Spanish National Research Council (CSIC). The ITQ is an international reference center in the area of catalysis and new materials (especially zeolites) and also in photochemistry.

Both of them are already users of the ALBA Synchrotron. On September 2013, they were participating in the 1st ALBA User meeting and we had the opportunity to talk to them.

#### What is your research area at the Instituto de Tecnología Química de Valencia (ITQ)?

One important area of our work is based on the development of solid catalysts. At a first stage, we work on the characterization of zeolites. Zeolites are microporous materials frequently used in many catalytic processes. We are able to include metals inside zeolites that act as a catalyst and finally we study the active centers of the catalyst, where the reaction happens.

#### Which is the added value of this research?

We are able to develop reaction cells to be studied in real operation conditions. In collaboration with ALBA (with beamlines 4 MSPD and 22 CLAESS), we have already developed two cells. These cells are used to simulate operation conditions over the catalysts.

# Which kind of synchrotron light techniques do you use in your research?

We mainly use diffraction beamlines for the structural studies of zeolites and absorption beamlines for metal

characterization and the study of active centers. The use of synchrotron light is essential in our research because zeolites are very complex materials and we require a excellent resolution and brilliance to be able to characterize them. Besides, we need to use synchrotron light to study the structures of the active centers of catalysts.

# Regarding the use of synchrotron light, which are your research interests?

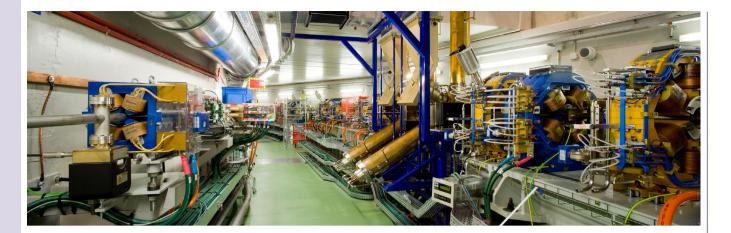
We are very interested in knowing more about what's happening in surfaces with catalysts. We also want to go deep in discovering what happens in the active center and check how the reactant interacts.

# In your center, do you develop industrial research projects?

We do basic research but, at the same time, this research is very interesting for the industry. We have had several collaborations with companies and now we know what they need and we try to help them designing or even fabricating prototypes of in situ reactant cells with automatic controls.

# Which results have you shown at the 1st ALBA User Meeting?

We are now working on a catalyst composed by nanoparticles of platinum and tin. Together with the PhD student Cristina Alonso – who is being guided by Gemma Guilera and Fernando Rey-, we have found an alloy of platinum and tin that works very well as a catalyst. We are now trying to obtain this alloy without having the support of a zeolite. This may have an industrial application, specially, in the oil sector.



# ALBA works on the implementation of top-up injection

ACCELERATORS

Present accelerators at ALBA operate with typically two injections per day with a lifetime of 20 hours approximately, which means that the decay in current between two consecutive injections is about 40%. Now ALBA is implementing a top-up injection mode, which will maintain an almost constant current and will offer a greater stability of the photon beam. This upgrade should be running for users from May 2014. • The ALBA accelerators consists of a 100 MeV Linac, followed by a full energy Booster and a 3.0 GeV Storage Ring. The Storage Ring, with 268.8 meters of circumference runs nowadays with 120 mA, with two injections per day. It has an emittance of 4.5 nmrad, which means that beam sizes are as low as few micrometers. Orbit and beam stability, required to be 1/10 of the beam size, are of major importance to maintain the quality of the photon beam for the experiments.

But the photon beam stability is also affected by the thermal drift of the mechanical components of the accelerator and the beamlines. In the actual operation mode, decay mode, the thermal load in these components is decaying together with the beam current, and the photon stability is affected.

In the top-up operation mode, the current in the storage ring is kept constant. This is achieved by injecting almost continuously a very small fraction of current, typically a few per mil, to cope continuously with the beam losses due to the finite lifetime. It also requires that the front ends, which connect the Storage Ring with the beamlines, remain open during injection.

In this situation, there is a constant thermal load on the accelerators and on the optical component of the beamlines, which increases greatly the position stability of the photon beam at the sample. This, together with the fact that a constant photon flux at the sample means a constant signal level at the detectors, will sensibly improve the data quality of the experiments performed at ALBA.

At present, safety radiation tests are being performed at the different beamlines with successful results. Additional safety, hardware and software tests are planned for the next months, with the target of having top-up operation mode implemented for users from May 2014.

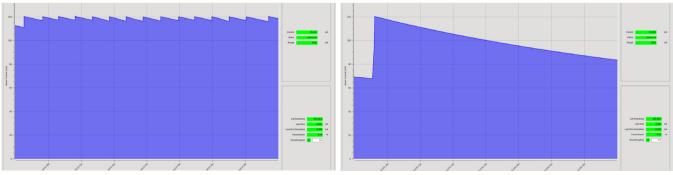


Figure 1. Comparison of top-up injection mode (firsts tests) and decay injection mode. The images show the evolution of the stored current intensity with time for top-up and decay operation mode.

10

# BIOSCIENCES

# First atomic-resolution protein structures solved at ALBA

#### BL-13 XALOC

.....

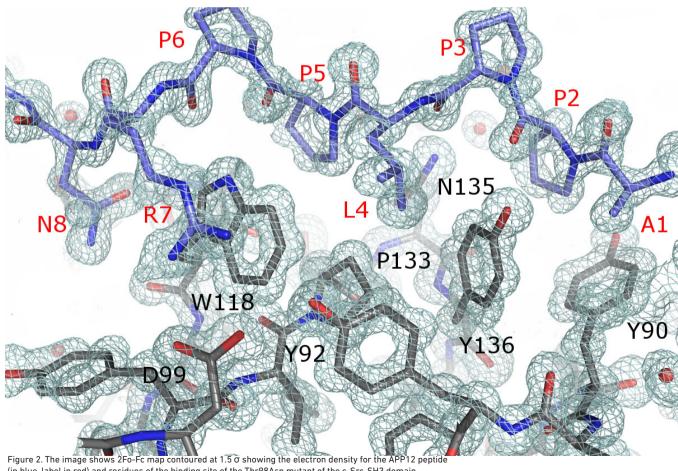
Professors Julio Bacarizo and Ana Cámara-Artigas, from the University of Almería, have determined at atomic resolution the crystal structures of the c-Src SH3 domains mutant in complex with the high affinity peptide APP12. These protein structures are the first ones solved at atomic resolution in ALBA. Information obtained at XALOC beamline could have implications in pathologies such as cancer, AIDS or osteoporosis.

• The SH3 domains, found in many different proteins, in diverse numbers and combinations, are related to deregulated signaling pathways during cancer development and are also associated to other pathologies such as AIDS, osteoporosis or inflammatory processes. Solving these structures at atomic resolution allows a detailed analysis of their function and features.

Beamline 13-XALOC, devoted to macromolecular crystallography, helped professors Bacarizo and Cámara-Artigas to measure crystals of these proteins at atomic resolution (0.98 Å). X-ray diffraction data were collected using the PILATUS 6M detector.

These structures have been deposited in the Protein Data Bank (PDB IDs 4HVU and 4HVW) and the research has been published in Acta Crystallographica Section D.

*Reference:* Atomic resolution structures of the c-Src SH3 domain in complex with two high-affinity peptides from classes I and II. Julio Bacarizo and Ana Camara-Artigas, Acta Cryst. (2013). D69, 756–766; doi:10.1107/S0907444913001522.



(in blue, label in red) and residues of the binding site of the Thr98Asp mutant of the c-Src-SH3 domain (in grey, label in black).

11

# MATERIAL SCIENCES

# 60 nm diameter magnetic bubbles imaged at Mistral

**BL-09 MISTRAL** 

A collaboration research among the **ALBA Synchrotron, the University** of Oviedo and the Centro Nacional de Microelectrónica (CNM-CSIC) has demonstrated that the effect of magnetic pulses can alter the microscopic structure of the domains at remanence. Depending on the magnitude of the pulsed field, either maze. bubbles or intermediate situations have been observed.

The study of the topology and structure of magnetic domains is one of the active topics in modern research in nanomagnetism. Beamline 9 Mistral offers the possibility of high resolution imaging of domains using the microscope and the circular magnetic dichroic absorption contrast. Thanks to the stability of the microscope and beamline optics, it is possible to achieve detailed information on the changes of a specific magnetic domain when a pulsed magnetic field is applied.

The samples analyzed were thin films (80 nm thickness) of Co5Nd alloys grown by sputtering techniques at the University of Oviedo and were deposited on silicon nitride membranes fabricated at the Centro Nacional de Microelectrónica (CNM-CSIC). located in Bellaterra, that fit exactly the geometry of the microscope sample holder. The pulses of magnetic fields were created with a microcoil developed at ALBA. They could achieve +/- 1 T as a maximum amplitude and had a duration of about 15 µs.

The figure at the top displays the characteristic labyrinth domains which are the most common magnetic structure at remanence after applying saturation pulses either positive or negative. Black and white domains correspond to inward and outward perpendicular magnetizations. The inset is the Fourier transform which shows a ring pattern indicating azimuthal disorder. The inverse of the thickness of the ring gives the lateral correlation length of the distribution which is about 120 nm.

The new and somewhat surprising result observed is displayed in the lower figure. Applying pulses of selected amplitudes below the saturation of the perpendicular magnetization, resulted in a bubble domain structure with a very narrow distribution of the dimensions of the bubbles which had a diameter of about 60 nm. The bubbles have mostly 6 fold and also 5 fold coordination and do not display long range order as shown by the annular shape of the Fourier transform in the inset.

These are the initial results of a systematic exploration of the different domain topologies that will be carried on the near future.

Reference: Universidad de Oviedo: Carlos Quiros, Cristina Blanco, María Velez and José M. Alameda Centro Nacional de Microelectrónica (CNM-CSIC) : Jaume Esteve, Marta Duch and Nuria Torras ALBA: Ricardo Valcárcel, Jose Ávila and Óscar Matilla for the development of the microcoil, Eva Pereiro, Andrea Sorrentino and Salvador Ferrer for performing the measurements.

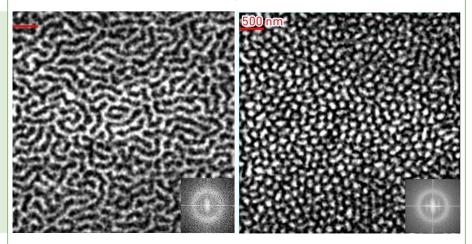


Figure 3. Images of the experiment showing, in the first image, the characteristic labyrinth domains and, in the second image, the bubble domain structure. The insets are the corresponding Fourier transforms.

13

OCTOBER 2013 - N° 36 ALBA Preced

# **MATERIAL SCIENCES**

# Understanding magnetic cobalt nanostructures

#### **BL-29 BOREAS**

An international collaboration among researchers from Nanjing University, the Max Planck Institute for Chemical Physics of Solids and the ALBA Synchrotron has discovered new aspects of the microscopic magnetic mechanisms in cobalt nanostructures. This research will contribute to a better understanding of the magnetic exchange bias systems used for magnetic information storage devices. These systems enable the production of smaller hard disks with a higher information density. • Computer hard disks use electromagnetic recording systems. To do so, they use "heads" to write or read onto or from the different disks, respectively. These heads are composed of layers made of ferromagnetic and antiferromagnetic materials. In the core of each layer, the electron spins carry the magnetic moment of the atoms.

A group of researchers from the ALBA Synchrotron, Nanjing University, and the Max Planck Institute for Chemical Physics Solids have discovered that cobalt nanostructures in a non-magnetic host material show a surprisingly high "exchange bias" effect. The exchange bias occurs when a ferromagnetic material (FM) and an antiferromagnetic material (AFM) get in touch and, as a result, there is a strong magnetic interaction between them. This effect has a great impact on magnetic recording systems. Nowadays, the exchange bias is used to pin the magnetic moments within the magnetic reference layer in spin valve readback heads.

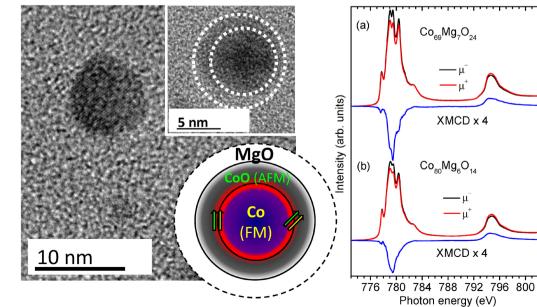
The researchers designed and produced samples consisting of Co/ CoO core-shell nanoparticles (~4 nm diameter Co metal core and CoO shell with ~1 nm thickness) embedded in a non-magnetic MgO matrix. Cobalt is one of the most used materials in modern magneto-electronics, because of its outstanding magnetic properties. This Co/CoO system exhibits a surprisingly large exchange bias field HE $\approx$  2460 Oe and a large coercive field HC  $\approx$  6200 Oe at T= 2K, which is in sharp contrast to the small exchange bias and coercive field in the case of other non-magnetic Al<sub>2</sub>O<sub>3</sub> or C matrix materials reported in previous studies.

Using the brilliant circular polarized soft x-ray beam of the BOREAS beamline, they measured the soft x-ray magnetic circular dichroism (SXMCD) of the samples and a ferromagnetic signal originating from the nominally antiferromagnetic CoO shell could be observed. This finding gives direct evidence for the existence of rotatable interfacial uncompensated Co spins in the CoO shell, thus supporting the uncompensated spin model as a microscopic description of the exchange bias mechanism.

This research, which has been developed within the framework of a BOREAS in-house research project, has been accepted for publication in the "Nanoscale" journal.

*Reference:* "Direct observation of rotatable uncompensated spins in the exchange bias system Co/CoO–MgO" Chuannan Ge, Xiangang Wan, Eric Pellegrin, Zhiwei Hu, S. Manuel Valvidares, Alessandro Barla, Wen-I. Liang, Ying-Hao Chu, Wenqin Zou and Youwei Du Nanoscale, 2013, 5, 10236

Figure 4. Samples analyzed at BOREAS beamline Samples consisted of a ~4 nm diameter Co metal core and a CoO shell with ~1 nm thickness. The red zone shows the magnetic interaction area between the FM Co core and the AFM CoO shell, where the "exchange bias" effect takes place. The transmission electron micrographs (TEM) show the size and morphology of the Co<sub>40</sub>Mg<sub>2</sub>O<sub>24</sub> sample, revealing the Co/CoO core-shell particles embedded in a MgO matrix. Co-L<sub>2,3</sub> XMCD spectra of (a) Co<sub>49</sub>Mg<sub>7</sub>O<sub>24</sub> and (b) Co80 Mg6014 measured at 80 K under a 5T magnetic field.



14

# MATERIAL SCIENCES

# Pump-probe powder diffraction with a time resolution of 50µs at ALBA

BL04-MSPD

An international group of researchers from Germany and France have investigated structural changes of industrially relevant ferroelectric materials on a time scale down to 50 microseconds. Ferroelectrics find a multitude of applications as actuators. sensors, or data storage devices.

\_\_\_\_\_

Recently, research focused on lead free substitutes for the widely used PZT (PbZr\_Ti, 03). Knowledge of the rather complex phase diagram as well as the microstructural changes during duty cycles and especially after fatigue is of fundamental importance to improve these technologically relevant materials.

At Beamline 04 MSPD the individual structural contributions from phase transitions, texturing and domain switching could be identified during electric cycling of new and fatigued samples. The fast MYTHEN detector together with the excellent beam conditions allowed for full pattern recording of a Q-range up to almost 10  $Å^{-1}$  and 50µs time resolution.

The ferroelectric materials were measured with a MYTHEN II detector system in a pump-probe setup at beamline 04 MSPD. It was the first time such a time-resolved experiment was performed at ALBA. The research group (composed by Manuel Hinterstein, Hans Kungl, Jérôme Rouquette and Michael Knapp) was

able to measure complete diffraction patterns in a Q-range up to about 10 Å-1 at 28keV and with a time resolution down to 50µs as a function of an applied electric square wave field and cycled with 50 Hz. The kinetics of the poling mechanism like domain switching, piezoelectric effect and the tetragonal-rhombohedral phase transition discovered earlier in similar systems (Phys. Rev. Lett. 107, 077602) can be studied this way. The very high time resolution was achieved with a pump-probe setup where the detector readout was gated and synchronized with the electric field cycling. By recording full diffraction patterns the individual contributions to the microstructure including evolution of texture can clearly be separated.

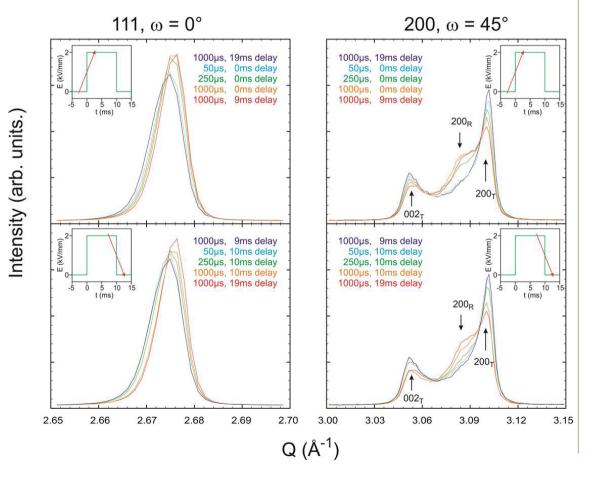


Figure 5. The experiment shows a phase transition on the rising edge of the electric field in the range of 250us (see the peak labeled 200R), while on the trailing edge several intermediate steps can be observed over a period of more than 1000us. At the same time the tetragonal reflections (002T. 200T) show the opposite behaviour. At the 111 reflection similar differences can be observed between rising and trailing edge.

# **MATERIAL SCIENCES**

# Magnetic nanocrystals in GaN semiconductor measured at Circe

# BL24-CIRCE

Within the framework of a collaboration among Dimitri Arvanitis from the University of Uppsala, I. Kowalik from the Institute of Physics, Polish Academy of Sciences and Miguel Ángel Niño from IMDEA Nanociencia, diluted magnetic semiconductors were measured last July 2013 at Circe beamline.

• The magnetic configuration of Fe-based nanocrystals embedded in a GaN semiconductor was imaged by XMCD-PEEM at room temperature.

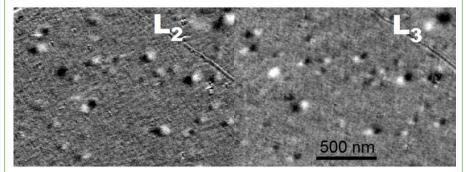


Figure 6. XMCD-PEEM images at the FeL3 and FeL2 absorption edges (the contrast inversion confirms the ferromagnetic character of the domains). Despite their small size, typically around 50 nm, most crystals contain more than one magnetic domain.

# In situ magnesium hydration

#### **BL24-CIRCE**

During the spring shutdown, Juan de la Figuera from the Instituto de Química Física Rocasolano (CSIC) used the end-station in Low Energy Electron Microscopy mode to analyze magnesium hydration process.

 Ultrathin Mg films were grown in situ on a Ru(0001) surface and then exposed to atomic hydrogen in order to study the hydration process and assess the feasibility of Mg as a hydrogen storage material.



Juan de la Figuera with Michael Foerster and Lucía Aballe (both from ALBA) during the experiment. In the background, a growing Mg-hydride island. Photograph: ALBA 16

ALBA neuco OCTOBER 2013- Nº 30

## **CHEMISTRY**

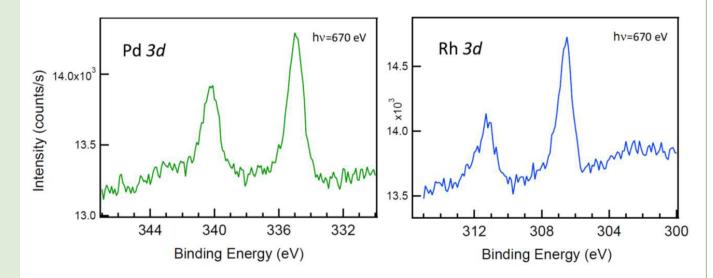
# **First**

commissioning users at Near Ambient Pressure Photoemission (NAPP) endstation BL24-CIRCE In September 2013, beamline 24 **CIRCE** received the first friendly users at the Near Ambient Pressure Photoemission branch. The aim of the experiment, proposed by Jordi Llorca and Nuria Jiménez from the Institute of Energy Technologies at UPC Barcelona, was to investigate the electronic structure of 3nmsized RhPd nanoparticles supported over CeO<sub>2</sub>, during the ethanol steam reforming reaction to produce hydrogen. The optimization of hydrogen production processes is a crucial step for its implementation as a fuel for local electricity generation, in electric vehicles.

After the catalyst activation by means of oxidation-reduction cycles, the ethanol-water mixture was introduced in the NAPP analysis chamber using a bubbler connected to the chamber through a leak valve.

The evolution of Rh and Pd 3d core levels during the activation and reaction steps at sample pressures of 5x10<sup>-2</sup> mbar and sample temperature of 550 °C was monitored by in-situ XPS.

Figure 7. Rh and Pd 3d levels from RhPd nanoparticles during ethanol steam reforming reaction. Pressure= 5x10<sup>-2</sup> mbar of 1 Et0H/ 6 H<sub>2</sub>O/Ar, sample temperature= 550 °C



# **EXPERIMENTS DIVISION**

# New beamline scientists in ALBA

On September 2013, three new beamline scientists have started working at ALBA Synchrotron. They are Laura Simonelli, in charge of beamline 22 CLAESS (Core Level Absorption & Emission Spectroscopies), Marc Malfois, responsible of beamline 11 NCD (Non-Crystalline Diffraction) and Daniel Fullà, scientist of XALOC (Macromolecular Crystallography).

• Laura Simonelli, with a doctoral degree on Material Science, has been second scientist at the European Synchrotron Radiation Facility (ERSF) and responsible of the stage of Juri Nyrow on "Mise en service de ID20". Her field of research interest is condensed matter physics, with a particular interest to the study of the interplay between lattice and electronic properties in highly correlated systems and other technologically relevant materials, such high Tc superconductors or battery materials.

Marc Malfois, PhD in Biochemistry, has been Senior scientist at the Small Angle X-ray Scattering Beamline (I22) at Diamond Light Source for more than 10 years.

He has also worked as a Scientist at the Dutch – Flemish Beamline (BM26) at ESRF (Grenoble, France) and Post Doc at the X33 Beamline at the European Molecular Biology Laboratory (Hamburg, Germany). His scientific interests include macromolecules in solution and the change of their state under the influence of different stimuli.

Daniel Fullà obtained his PhD in Physics at the European Molecular Biology Laboratory (EMBL) in DESY (Hamburg) developing X-ray optics for protein crystallography. Afterwards, he moved to Chile and worked in the radiotelescope ALMA as Commissioning Scientist for the European Southern Observatory (ESO). At ALBA, Daniel will be focused in the automation and control of XALOC with scientific interests in protein crystallography methods.

# Latest beamline technical progress

## BL11-NCD

• During the last months beamline 11 NCD (Non-Cristalline Diffraction) has been working on the improvement and automation of the sample environment in order to do experiments more efficiently. In that sense, a set of linear stages have been set up to complement the sample table movements. Now the sample can be moved with a resolution up to about 100 nm. In addition it is possible to align the sample with customized diagnostics. For temperature controlled experiments new software features have been implemented to create and customize different profiles and perform consecutive ramps. On the other hand, the beam size has also been improved achieving a spot size of 160 µm horizontal x 47 µm vertical (FWHM) in the SAXS detector plane (focus plane).

#### **BL-MIRAS**

• Possibility of developing previously confirmed project of Infrared Microspectroscopy project (MIRAS) at ALBA has been reviewed. Project is currently at a stage of design where optical calculation part has been completed and technical design of beamline layout and individual optical hardware components is under way.

A Bruker LUMOS Infrared microscope has been made available by Bruker España S.L. to MIRAS project at ALBA. It enables scientists from areas of biology, chemistry and material science perform IR measurements at ALBA even before completing Beamline construction process.

The LUMOS system is currently available to potential users.

#### **BL24-CIRCE**

• In September 2013, a **UHV sample parking for up to 5 samples** was added to the experimental station. This new development will greatly reduce preparation times for samples with sensitive surfaces and thus enhance sample throughput.



18

ALBA Jueans OCTOBER 2013- Nº 30

# New industrial developments at ALBA

One of the main activities of ALBA is designing and developing their own equipment and devices, which may have an industrial interest. Other European facilities have already acquired some electronic units designed and produced at ALBA.

•Within an in-house research project, ALBA has developed an electrometer to measure very low currents (pA). This 4 independent channel electrometer is based on transimpedance amplifiers and integrates high resolution ADC converters and an Ethernet communication port. Each channel has independently configurable range, offset and low pass filter cut-off frequency settings and the main unit has external I/O to synchronize the data acquisition with the rest of the control system.

The excellent results raised interest among other European Facilities to acquire and test this device. Nowadays, there are more than 40 units implemented at ALBA beamlines and two European synchrotron light facilities have already expressed their interest on it.



Image of the electrometer developed at ALBA. Photograph: ALBA

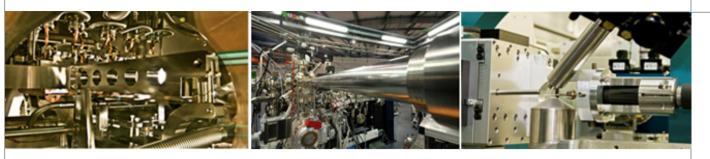
# Collaboration between ALBA Synchrotron and multinational company Henkel

The collaboration agreement between ALBA and Henkel signed last February 2013 is now a reality. Since last September, the postdoctoral researcher Kang Wei Chou, who is funded by Henkel, started working at ALBA. The scope of the collaboration is to research and develop projects of new products using synchrotron light techniques.

Henkel, world leader in the production of lines of laundry and home care, beauty care and adhesive technologies, is very interested in applying to its developments the advanced characterisation and analysis techniques by synchrotron light. That aspect, together with the know-how exchanging amongst scientists and technologists from both organisations, will enable to reinforce and expedite the innovation process of its products.

For ALBA Synchrotron this agreement means the definitive confirmation of the very relevant contribution that this kind of large scientific and technological facilities can offer to industry and society. It is not only a key factor in competitiveness of leading companies in research, development and innovation, like Henkel, but it also turns into a major attraction for future advanced technological companies.

Adhesive Industrial technology of Henkel.Image courtesy of Henkel



## Synchrotron light applications in nanoscience and molecular materials characterization

Next November 14th and 15th, ALBA Synchrotron organizes a workshop with the aim of describing the available tools and the analytic potential of the current ALBA beamlines. This workshop will take place in ALBA's facilities and has no registration fees. There is a limited attendance of 50 participants.

Nowadays, ALBA Synchrotron has seven different operational beamlines. In order to promote the use of synchrotron light among researchers, ALBA is organizing a workshop with the support of the "Grupo Especializado en Nanociencia y Materiales Moleculares (MAM) de la Real Sociedad Española de Química (RSEQ) y de Física (RSEF)".

During two days, those researchers still being unfamiliar with the possibilities of synchrotron radiation will have the opportunity to hear firsthand information on ALBA beamlines features and to show their main research topics in order to get a feedback from ALBA scientists on how ALBA beamlines could contribute to promote their research. Participants will also be able to visit beamlines and check in-situ the experimental tools available in of each of them.

More information and registration: *http://albasync2013.cells.es/* 



# ALBA Synchrotron opens to visitors on summer holidays

750 people have visited ALBA during the summer holidays. Groups of families, friends and students could walk through the experimental hall and visit the different beamlines of ALBA.

For the first time. ALBA opened its doors to visitors during the summer shutdown. 750 visitors had the opportunity to know ALBA's facilities and discover the possible applications of the synchrotron light source. Organized in groups of 25 people, the visitors were introduced to ALBA and synchrotron light radiation techniques in guided tours of 1 hour and 30 minutes.

Visitors showed a great interest on the experiments performed in ALBA and other aspects regarding the engineering and organization of ALBA.

For ALBA, it was a good opportunity to bring people closer to science and encourage scientific interest among youngest generations.



Visitors at the ALBA Synchrotron during the summer period. Photograph: Anna Bosch

Explaining ALBA to children in an animated cartoon series

The ALBA Synchrotron is the central topic of the new animated cartoon series "Descubre con Tadeo", addressed to children. Tadeo Jones, an animated intrepid explorer, explains what ALBA is and which applications it has in science.

• "Descubre con Tadeo" ("Discover with Tadeo" in English) is a children cartoon series where the explorer Tadeo Jones disovers the most highlighted scientific facilities of Spain. The objective of this series is to outreach about the quality and variety of the Spanish research and also to encourage curiosity among the youngest.



Frame of the ALBA episode of "Descubre con Tadeo". Image courtesy of telecinco.es



This television series has been created by Mediaset España. Lightbox Entertainment in collaboration with El Señor Studio and with the advice of the Spanish Science and Technology Foundation (FECYT). It is broadcasted in the Spanish television channels of Mediaset group during the weekends. Here, you can watch the complete episode devoted to ALBA.

20



#### **ALBA Main parameters**

Electron beam energy	3 GeV
Storage ring circumference	269 m
Natural horizontal emittance	4.4 nm rad
Nominal beam current	250 mA
Number of insertion straight sections	17
Number of Phase-I BeamLines	7

ALBA is the Spanish 3rdgeneration synchrotron light source, located in Cerdanyola del Vallès, near Barcelona, recently come into operation with seven Phase-I Beamlines. The e– beam energy is 3 GeV, and the photon energy available in these first beamlines ranges from UV up to hard X-ray of tens of keV.

Different techniques of synchrotron radiation utilization are available including diffraction, spectroscopies and imaging.

#### ALBA Beamlines

ALBA has seven operative beamlines organized in three groups for their scientific applications: Biosciences, Chemistry and Condensed Matter – magnetic structures, electronic properties and nanoscience-.

Beamline Name	Techniques	Scientific applications
MSPD	High-resolution powder diffraction High-pressure diffraction	Structure of materials, Time-resolved diffraction
MISTRAL	Soft X-ray full-field transmission X-ray microscope. Optimized on the "water window"	Cryogenic tomography of biological objects. Spatially resolved spectroscopy
NCD	High-resolution small- and high-angle X-ray scattering/diffraction	Structure and phase transformations of biological fibres, polymers, macromolecules in solution. Time-resolved X-ray studies
XALOC	X-ray diffraction from crystals of biological macromolecules	Macromolecular crystallography, with particular emphasis on large-unit-cell crystals
CLAESS	EXAFS, XANES, Quick-EXAFS, XES	Material science, chemistry, time-resolved studies
CIRCE	Photoemission microscopy (PEEM) Near atmospheric pressure photo-emission (NAPP)	Nano-science and magnetic domain imaging (PEEM). Surface chemistry (NAPP)
BOREAS	Circular Magnetic Dichroism Resonant Magnetic Diffraction	Magnetism, surface magnetism and magnetic structures



ALBA Synchrotron - Carretera BP 1413, de Cerdanyola del Vallès a Sant Cugat del Vallès, Km. 3,3 08290 Cerdanyola del Vallès, Barcelona, Spain - Tel: +34 93 592 43 00 - www.cells.es