

# ALBA NEWSLETTER

April 2013

## Letter of the Director



In these first months of 2013 ALBA is facing the reality that many representatives of our staff are changing affiliation, attracted by laboratories around the world at the front end of scientific and technological advances.

This is a natural process once a project has finished its constructing phase. Even if worried by the loss of experienced people, I want to express my congratulations to all of them for the opportunities that they have been offered thanks to their excellent capacities and experience, and I take the opportunity to welcome the newcomers, who will add fresh ideas and strength to our organization.

My special congratulations go to two of the founders of ALBA: Jörg Klora, nominated IT Head at ITER, and Lluís Miralles, nominated Head of General Infrastructure Department at CERN. They demonstrate the excellence of ALBA and of the Spanish science, and we all wish them a fruitful and successful experience as they have had in our laboratory.

### Caterina Biscari

Director of ALBA synchrotron



Jörg and Lluís are attending the first ALBA Management Board Meeting. From left to right: Jörg Klora, Lluís Miralles, Salvador Ferrer, Dieter Einfeld, and Joan Bordas.

## SUMMARY

### Experiments Division news

**MSPD news.** Innovative in situ time resolved experiments have been measured on the Powder Diffraction station of the beamline. [Page 2.](#)

**MISTRAL news.** X-ray cryo-tomography has been performed on HeLa cells. [Page 3.](#)

**NCD news.** First small angle scattering measurements of proteins in solution have taken place. [Page 4.](#)

**XALOC news.** The first paper has been published using diffraction data collected at the beamline. [Page 5.](#)

**CLÆSS news.** The first official experiments were carried out at the beamline. [Page 6.](#)

**CIRCE news.** The first spectrum has been measured at the Near Ambient Pressure Photoemission station electron energy analyzer. [Page 7.](#)

**BOREAS news.** An oxygen K-edge spectrum has been collected in the total fluorescence yield and total electron yield configuration on a ZnO semiconductor sample at room temperature. [Page 9.](#)

### Accelerators Division news

Information on accelerators operations, time dedicated to beamlines, machine experiments results and developments are given, [Page 10.](#)

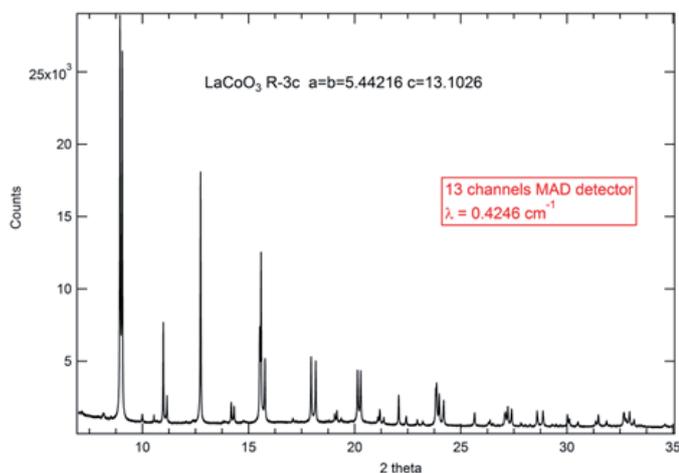
### PERSONNEL

New staff has been hired at the different divisions of ALBA. [Page 10.](#)



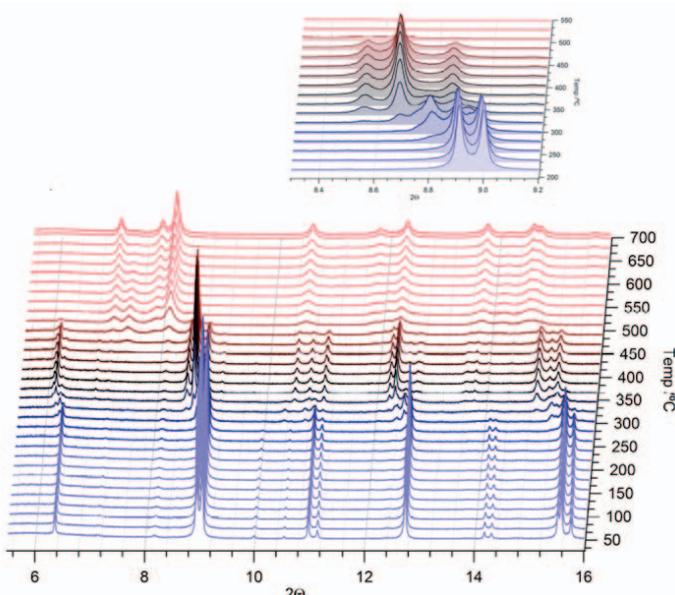
## BL04-MSPD Materials Science and Powder Diffraction

**User experiments in the powder diffraction end station with the Mad26 detector:** Laura Barrio and Noelia Mota Toledo from Madrid (CSIC - *Instituto de Catálisis y Petroleoquímica - Energía y Química Sostenibles*) have performed innovative in situ time resolved experiments in the Powder Diffraction (PD) Station of BL04-MSPD. Their aim was to follow the structural transformations, both during crystallization and reduction, of a series of  $\text{LaCoO}_3$  perovskites under in situ conditions, with particular focus on analyzing the effect of the partial substitution of La and Co positions by other elements. In situ time resolved X-ray diffraction data collected at  $\sim 30$  keV energy allowed mapping the structural changes under controlled gas flow conditions as function of temperature. The use of the high resolution provided by the MAD detection setup contributed to distinguish easily between the rhombohedral initial structure (Fig. 1) and the distorted orthorhombic/monoclinic phases occurring in this perovskite as a function of temperature and gas flow conditions (Fig. 2).

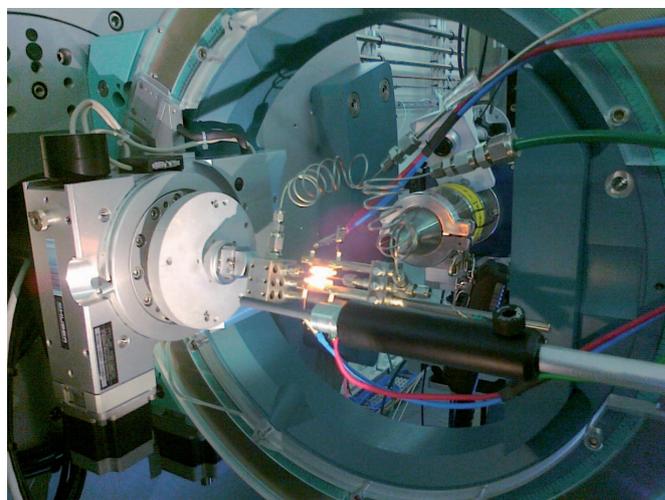


**Figure 1.** Long data collection at room temperature of the starting  $\text{LaCoO}_3$  compound in its well-known rhombohedral phase.

Users brought their own in situ reaction cell while ALBA supplied the gas flow/gas mixing infrastructure (the same as used on CLAES; Fig. 3). Since such a reaction cell can be of common interest for MSPD user groups, L. Barrio and co-workers agreed to share the technology and it is likely that this environment will be made routinely available in MSPD in the future.



**Figure 2.** Reduction of  $\text{LaCoO}_3$  to  $\text{La}_2\text{O}_3 + \text{Co}$  under 5%  $\text{H}_2/\text{He}$  gas flow. The transition is passing through a brownmillerite intermediate. Each temperature scan was collected in 8 minutes.



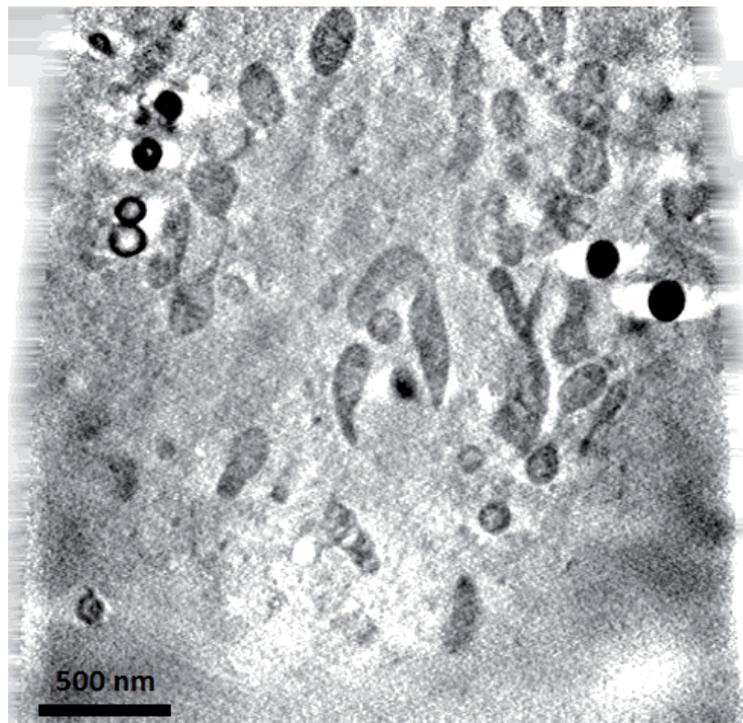
**Figure 3.** In situ reaction capillar flow cell mounted on the PD diffractometer. The shiny color is due to the radiation of the heating elements.



## BL09-MISTRAL Soft X-ray microscopy

Mistral started user operations in February 2013. Several proposals have been carried out during the months of February and March; many were devoted to cryo-tomography of cells and some to spectroscopy of iron oxide nanoparticles taken up by cells. Fig. 1 shows a slice of a reconstructed volume of a vitrified HeLa cell by the group of Dr. Lucy Collinson (Cancer Research Institute, London) showing mitochondria and other organelles.

We organized on March 22<sup>nd</sup> 2013 the first Biostruct-X workshop on Cryo X-ray Imaging for Biological Applications. The community of cryo soft X-ray tomography for biological applications is small, but it is growing fast. The objective of the workshop was to have experts and users of all levels exchanging expertise in sample preparation, correlative microscopy (confocal-electron microscopy-TXM), and data analysis. There were 47 registered participants and they came from Spain, Germany, UK, France, Israel, USA, and Taiwan.



**Figure 1.** Slice of a reconstructed volume of a vitrified whole HeLa cell by the group of Dr. Lucy Collinson (Cancer Research Institute, London) showing mitochondria and other organelles.

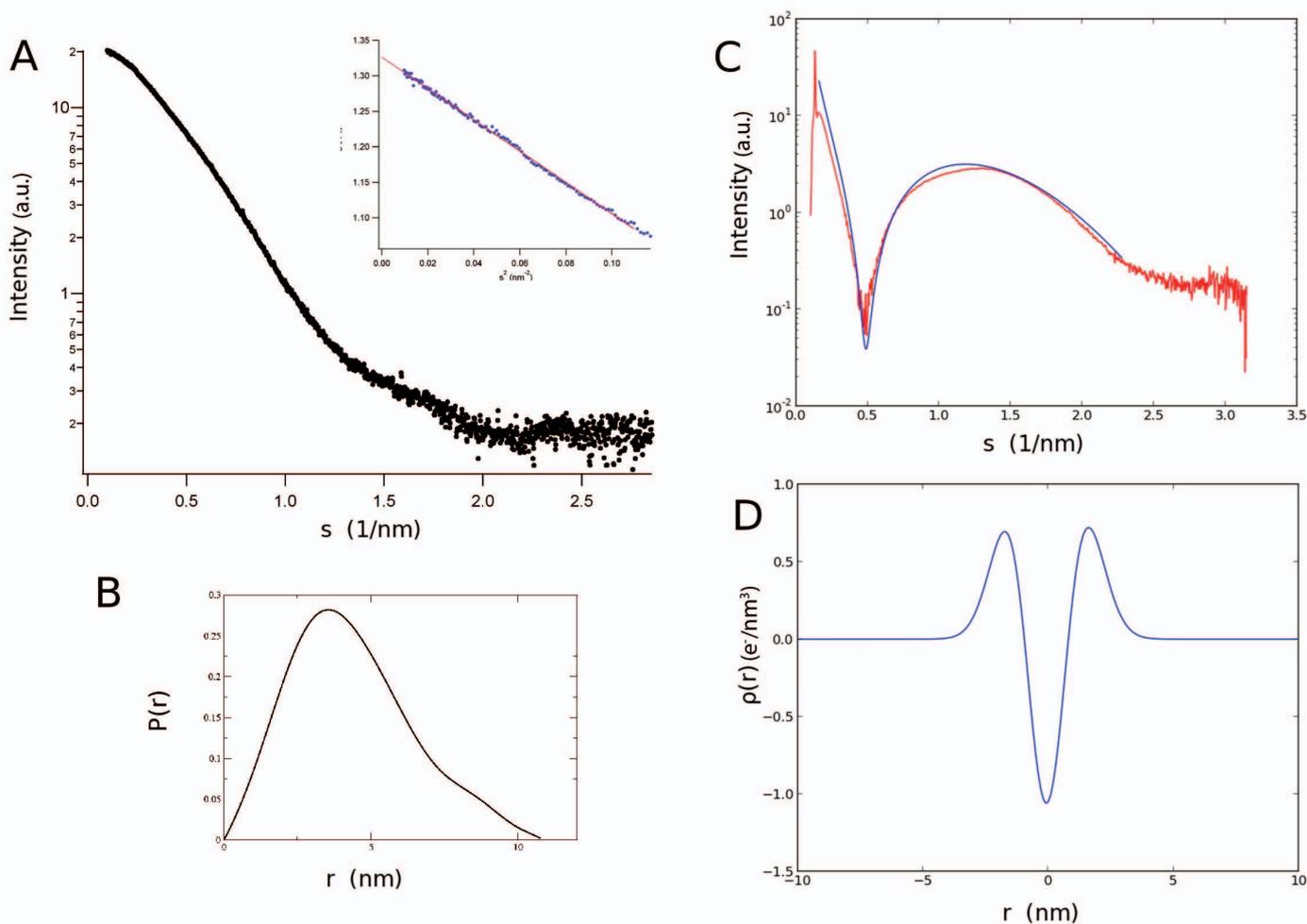


## BL11-NCD Non-Crystalline Diffraction

The first small angle scattering experiments (SAXS) of proteins in solution have taken place at the beamline. The scope of the scientific groups from the CNR - *Istituto di Biofisica* - Genova, Italy in collaboration with Faculty of Life Sciences, University of Manchester, UK, was to study purified proteins, enriched preparation of membranes, small unilamellar vesicles, and multilayer proteoliposomes.

The two post-docs taking part in the experiments made full use of the facilities in the biology su-

pport laboratory, Lab. P0-11, located next to the beamline and all necessary tools and facilities appear to have been available. Preliminary data and their analysis are shown in Fig. 1 below. Preparations have been made to be ready to install the new CCD detector that will be dedicated for wide-angle x-ray scattering applications. Finally, at the end of March we were happy to accommodate a Friendly User group from the Autonomous University of Barcelona who used the beamline for two days in order to study with success the internal structure of large biological systems.



**Figure 1.** In A is shown the SAXS data of a protein in solution. In the upper-right inset it is shown the Guinier plot used to determine the gyration radius, and in B it is shown the distance distribution function, that reveals the quasi spherical shape of the protein. In C is shown a graph of the SAXS data for a large unilamellar vesicle (LUV), and in D is shown the determined electron density of the vesicle wall.

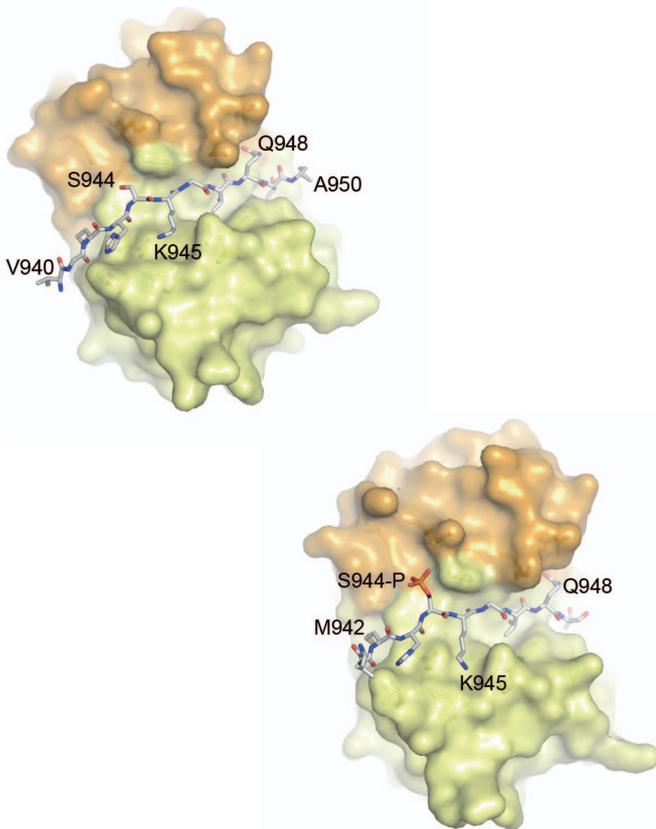


## BL13-XALOC Macromolecular Crystallography

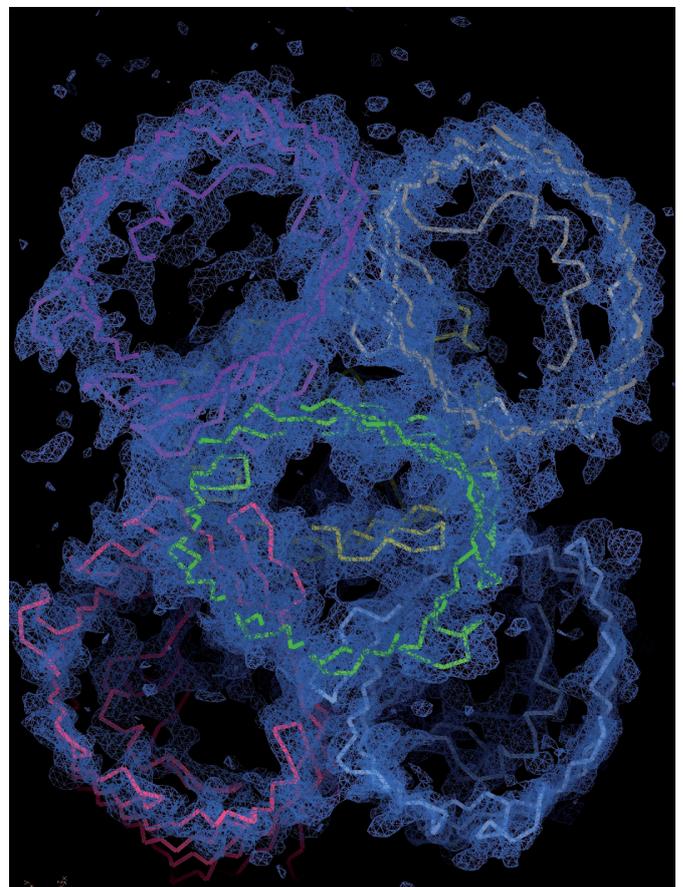
**First published paper with data collected at BL13-XALOC:** The first publication of BL13-XALOC is based in two crystal structures (Fig. 1) obtained with diffraction data collected at the beamline by the group of Prof. David Reverter (IBB, Autonomous University of Barcelona). These structures correspond to the complexes of human LC8 homodimers (yellow and orange) with the phosphorylated (right, PDB ID 3ZKF) and non-phosphorylated (left, PDB ID 3ZKE) versions of a Nek9 peptide. Nek9/Nercc1, Nek6, and Nek7 constitute a signaling module activated in early mitosis and involved in the control of spindle organization. Previous experiments suggested that LC8 binding to Nek9 was regulated by Nek9 autophosphorylation on Ser944, a residue

immediately located N-terminal to the LC8 conserved (K/R)xTQT binding motif, and that this was crucial for the control of signal transduction through the Nek/Nek6/7 module. The crystal structures obtained at BL13-XALOC of LC8 with both Nek9 peptides, together with a variety of complementary biophysical experiments, are able to explain the observed diminished binding affinity of Nek9 to LC8 upon phosphorylation on Ser944 in the Nek9 sequence, thus shedding light into a novel phosphorylation regulatory mechanism that interferes with LC8 protein-protein complex formation. The accepted article can be found [here](#).

The first integral membrane protein was solved at BL13-XALOC (Fig. 2) by the group of Prof. Chaptal (*Centre National de la Recherche Scientifique* – University of Lyon, France).



**Figure 1.** Complexes of human LC8 homodimers (yellow and orange) with the phosphorylated (lower right, PDB ID 3ZKF) and non-phosphorylated (upper left, PDB ID 3ZKE) versions of a Nek9 peptide.



**Figure 2.**  $2F_o - F_c$  map at 3.2 Å resolution of the first integral membrane protein solved at BL13-XALOC.

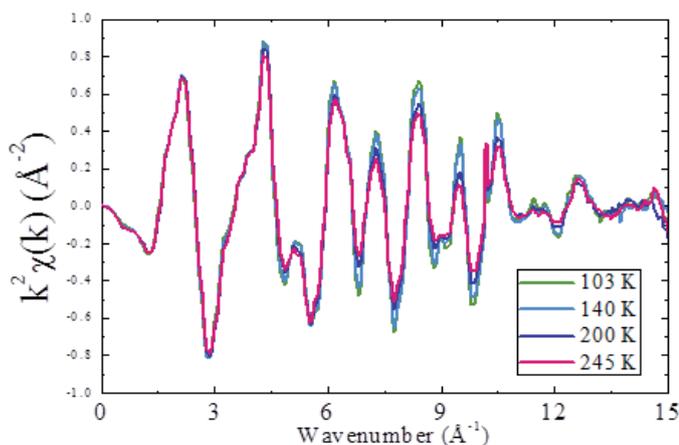


## BL22-CLÆSS

### Core Level Absorption & Emission Spectroscopies

The beamline responsible of CLÆSS (Konstantin Klementiev) left ALBA this month. He is now working as a beamline scientist at MAX IV Laboratory in Lund, Sweden.

The beamline is still in partial commissioning



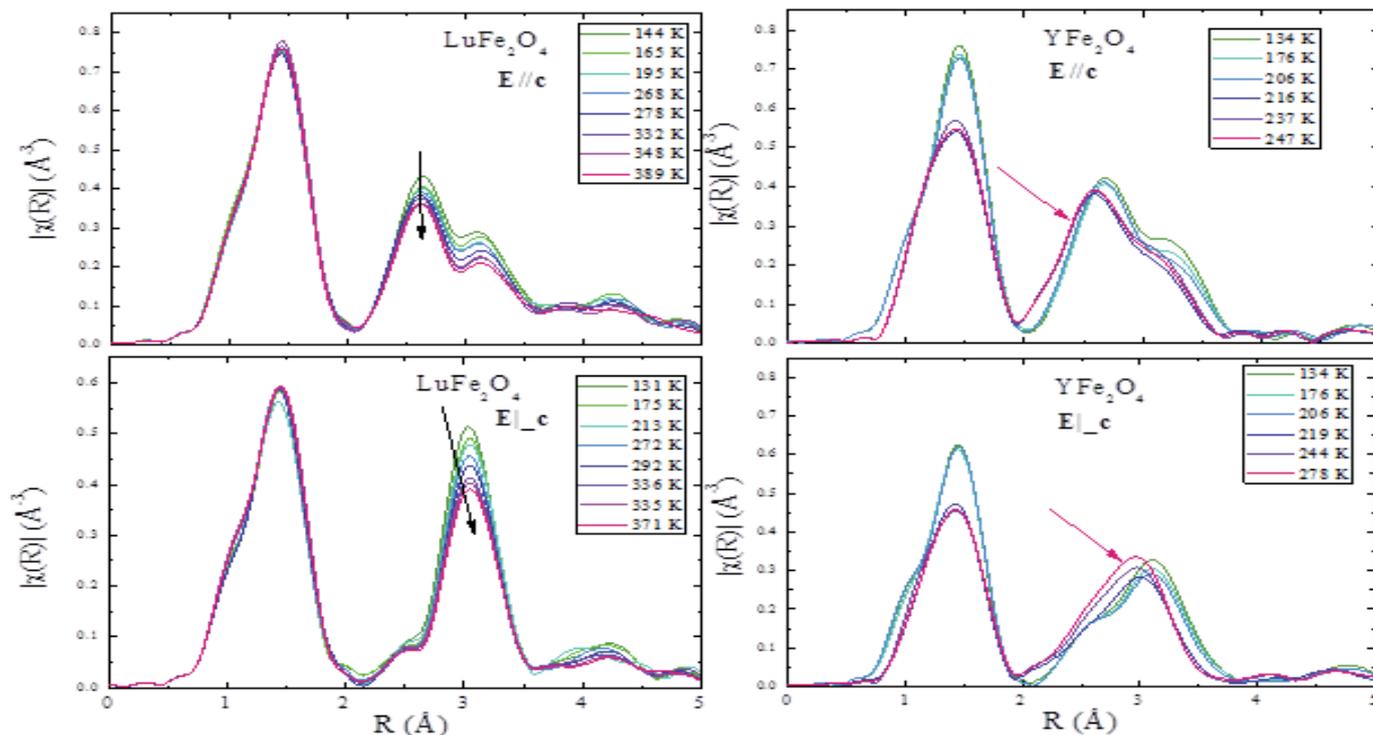
**Figure 1.** EXAFS signal, weighted in  $k^2$ , of  $YFe_2O_4$  at different temperatures. Spectra were taken in 10 min using the continuous scan mode.

mode. This month the fluorescence Silicon Drift Detector was commissioned at different energies with different real samples.

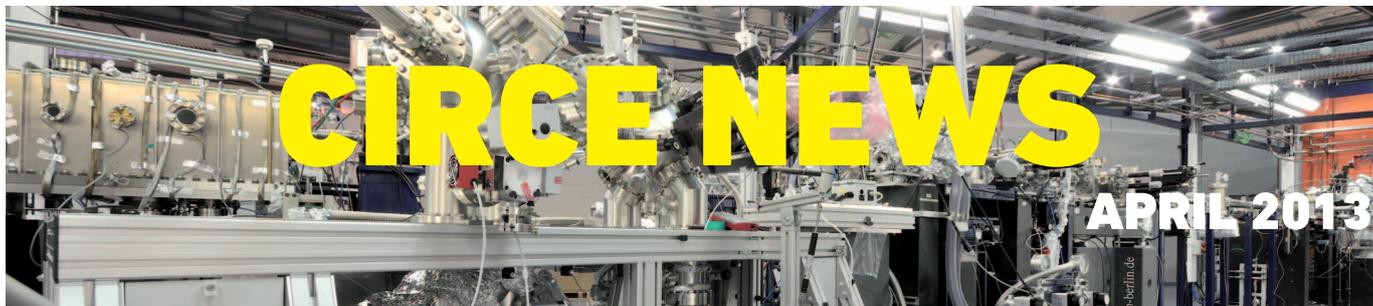
In addition, the two first official experiments were carried out at the beamline. One of them was a collaboration between the ICMAB (Dr. Carlos Frontera) and the University of Oslo and the second one was performed by the group of Prof. Joaquín García from the ICMA (*Instituto de Ciencia de Materiales de Aragón*, University of Zaragoza and CSIC). Both of them consisted on EXAFS measurements in transmission where spectra were collected using the continuous scan of the monochromator.

As an example, in Fig. 1 we can see several EXAFS signals obtained at different temperatures.

In Fig. 2 we show some results of the modulus of the Fourier Transform of the EXAFS signals of several samples measured by ICMA group, at different temperatures and different polarization configurations. In the case of Lu-sample, changes on Lu-Fe distances (second peak) are observable when heating the sample. On the other hand, there is a structural phase transition on the Y-sample below 210 K which is in accordance with the transition temperatures observed by resistivity and DSC measurements.



**Figure 2.** Modulus of Fourier Transform at different temperatures and different polarization configurations of  $LuFe_2O_4$  and  $YFe_2O_4$ . Again, spectra were taken in 10 min (1.5 keV energy range).



## BL24-CIRCE

### Photoemission Spectroscopy and Microscopy

**The first spectrum has been measured at the Near Ambient Pressure Photoemission (NAPP) station electron energy analyzer:** On Friday, March 22nd, a first X-ray Photoemission Spectrum was acquired at the CIRCE NAPP experimental station. The spectrum in Fig. 1 is a survey scan of a test Ag sample in ultrahigh vacuum. The in-situ capabilities of the NAPP, i.e. operation at pressures up to 20 mbar, will be tested during the next run.

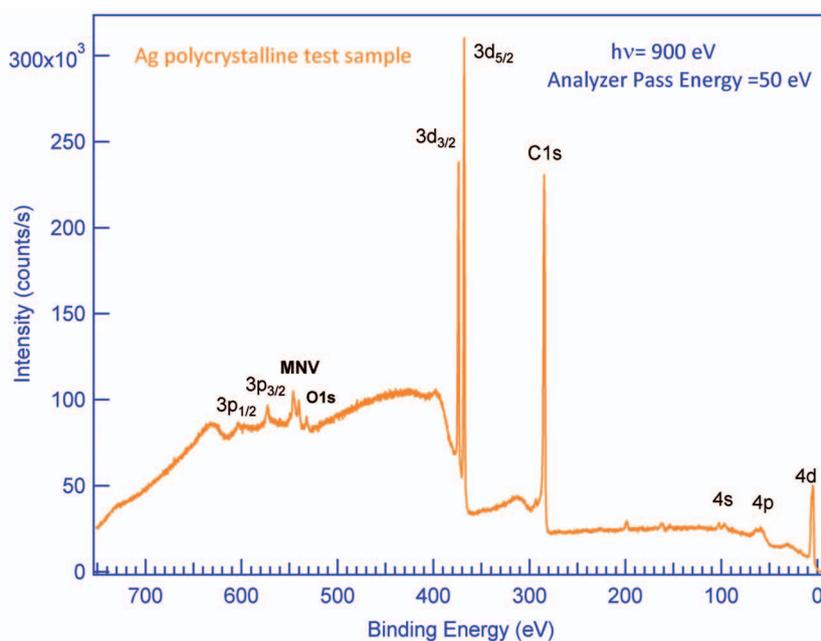
### Identifying Graphene by counting atomic layers:

Researchers from the *Centro de AstroBiología* and *Instituto de Ciencia de Materiales de Madrid* studied the reactivity of different graphene types by X-ray spectromicroscopy (XPEEM) at the CIRCE beamline (Fig. 2). Samples were prepared in

situ on silicon carbide substrates, monitoring the evolution of the Low Energy Electron Diffraction



**Figure 2.** Scientists from the *Centro de AstroBiología* and *Instituto de Ciencia de Materiales de Madrid* together with Lucía Aballe and Michael Foerster from CIRCE.



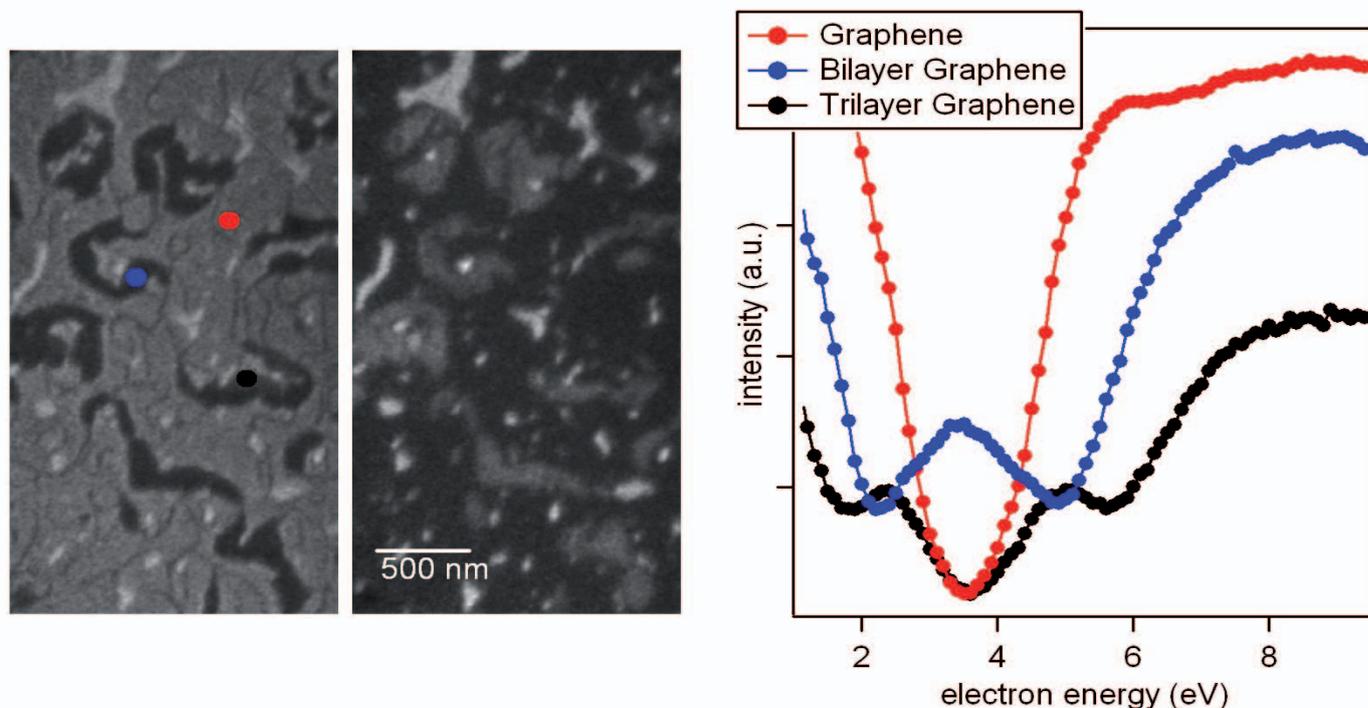
**Figure 1.** First X-ray Photoemission Spectrum acquired at the CIRCE NAPP experimental station.

(LEED) pattern in the microscope during heating up to 1250 °C.

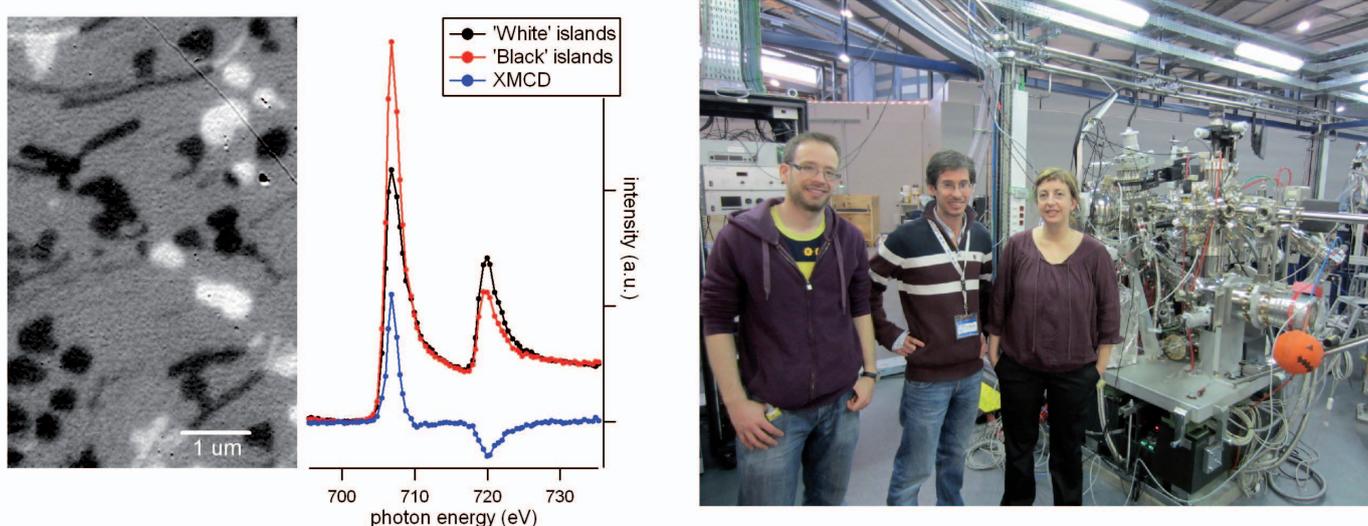
Depending on the number of atomic carbon layers, the electron reflectivity in Low Energy Electron Microscopy (LEEM) has distinct voltage dependence, as shown in the spectra below (Fig. 3). Identifying graphene and different graphene-like materials permits studying selectively their

reactivity by XPEEM spectro-microscopy.

Magnetic spectromicroscopy on submicrometer islands: Together with researchers from IMDEA Nanoscience and *Universidad Autónoma de Madrid*, the CIRCE scientists obtained full XMCD-PEEM spectra from submicroscopic objects: Fe islands grown in situ on a W(110) substrate (Fig. 4).



**Figure 3.** Left: Electron reflectivity image at 2.4 eV energy. Center: electron reflectivity image of the same area at 3.4 eV energy. Right: reflectivity versus electron energy curves of the different areas (color code in the inset), showing quantum size oscillations. The number of minima is directly correlated with the number of atomic graphene layers.



**Figure 4.** Left: XMCD-PEEM image at the  $FeL_3$  edge energy showing white/dark contrast where the magnetization is parallel/anti-parallel to the beam direction, obtained by subtracting images with opposite circular polarizations. Center: (red and black) Fe L-edge spectra from both island species obtained by XMCD spectromicroscopy (acquisition of an image stack while scanning the photon energy) and their difference or magnetic dichroism (blue). Right: Scientists from IMDEA Nanociencia and CIRCE.

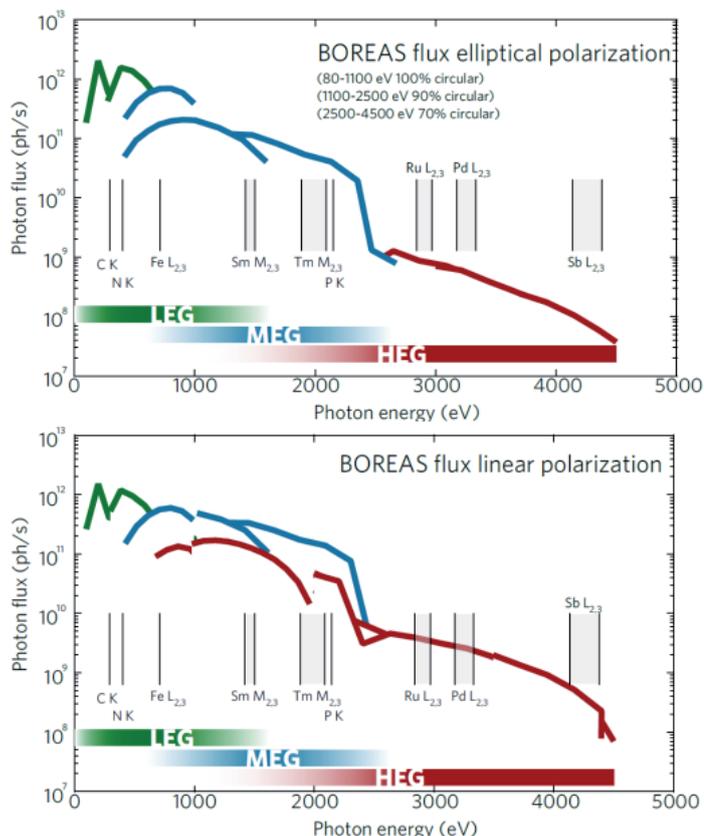


## BL29-CIRCE

### Resonant Absorption and Scattering

**Instrumentation and commissioning:** The flux measurements of the last commissioning shifts are finished. The photon flux at BOREAS has been characterized for the different combination of gratings (Low, Medium, and High Energy, LEG, MEG, and HEG, respectively) and mirrors (see Fig. 1 for the flux results in circular and linear horizontal polarizations). The energy range 100-4500 eV can be exploited for experiment with both elliptically and linearly polarized light. First XMCD results at the Ru L<sub>2,3</sub> have been collected during the experimental run of March (see previous March newsletter).

A monochromator intervention was successfully accomplished by the TOYAMA team. After the substitution of the grating exchange linear-drive bellow, the motorized grating exchange will



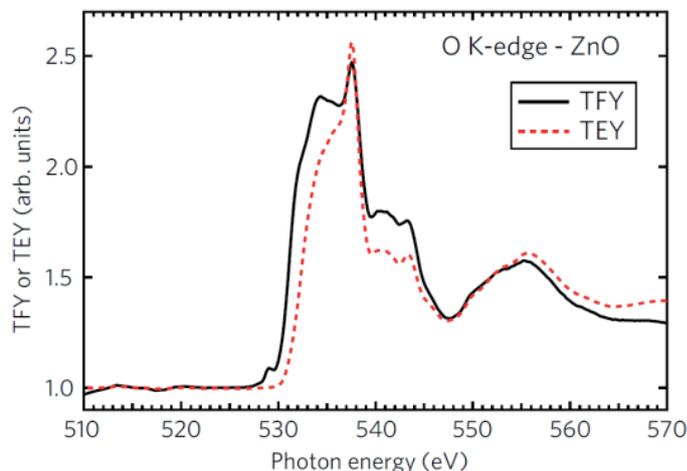
**Figure 1.** Photon flux measured on a silicon diode after the last optical element of the BOREAS beamline.

be tested and should be available again to users during the 2013 experiment run.

**Remanence magnetic field:** An off-line commissioning time has been employed to investigate the remanence magnetic field in HECTOR endstation. We implemented a procedure to reduce the parasitic field allowing users to perform “zero field cooling” experiment with a base magnetic field lower than 2 Gauss in perpendicular (y) and transverse (x) directions.

**New macros:** The temperature control of the HECTOR cryostat has been now implemented in SPOCK macros. The automatic temperature control has been first tested during an off-line commissioning time with good results.

**FYD:** The commissioning of the Fluorescence Yield Diode detector continues. During the last official user experiment, the low energy performance of the FYD at the Oxygen K-edge (530 eV) has been explored with very good results. The spectrum in Fig. 2 has been collected in the same measurement conditions that are used for normal TEY experiment (0.2 sec per point, 0.1 eV energy step, total acquisition time ~ 8 min) with

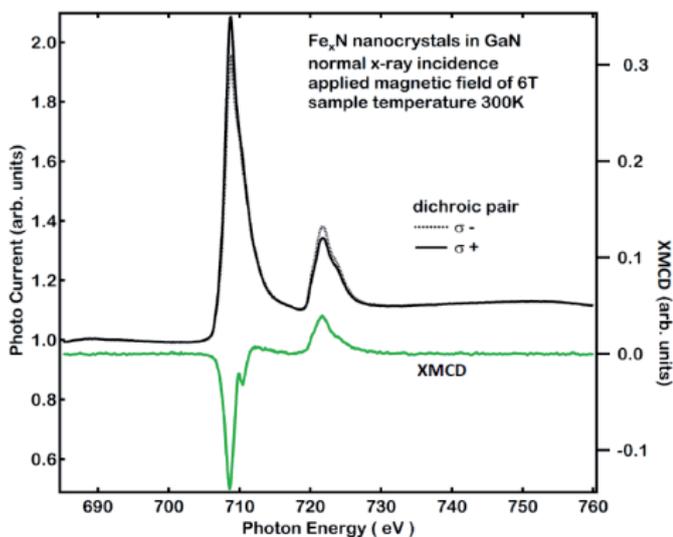


**Figure 2.** Oxygen K-edge collected in the total fluorescence yield (TFY) and total electron yield (TEY) configuration on a ZnO semiconductor sample at room temperature.

more than satisfactory s/n ratio. The photo-electron repulsion by the electrically polarized gold mesh seems to give promising results and will be further tested during the next on-line commissioning turn.

The MARES chamber bakeout finally started on March 27th. Another step to have MARES ready!

**User experiments:** The last experimental run has successfully concluded at the end of March with measurements on a sample of a GaN semiconductor with embedded Fe-based nanocrystals. The aim of the experiment is to investigate the magnetic properties of the embedded nanocrystals. The Fe L<sub>2,3</sub> XMCD measurements, performed at room temperature in a 6T magnetic field, evidenced features that were unobserved in previous experiments with lower magnetic field (See Fig. 3).



**Figure 3.** Fe L<sub>2,3</sub> XAS and XMCD at 300K, 6T on a GaN with embedded Fe-based nanocrystals..

## Accelerators Division news

RUN\_02 of 2013 finished on March 23rd 2013 at 7 h having provided 461.9 h for beamlines (BLs) or 97.7 % of the foreseen hours. Around 60 % of the downtime was due to RF trips and to water flow oscillations in the Storage Ring circuit of cooling water, while the rest was caused by several minor failures.

On the 2nd week of the run, the current in the storage ring was increased to 135 mA. Unfortunately we had to revert to 120 mA due to a vertical blow up of the beam which appeared for a given combination of gaps of the Insertion Devices. Specific machine shifts will be dedicated to study the beam stability under any ID gap combination.

## New staff

We are pleased to announce the following new employee who has recently joined the ALBA Synchrotron Light Source:

**Nahikari González.** Projects engineer. Transversal Section. Nahikari has a master on industrial engineering being specialized on mechanics. She has previous experience in mechanical systems and mechanisms design.