ALBA Newsletter
March 2013

The ALBA's newsletter is a monthly publication containing the latest news, updates, and developments of the ALBA Synchrotron Light Source.

Summary

The first user group of the multicrystal analyzer detector (Mad26) has performed an experiment in BL04-MSPD. First soft X-ray cryo tomographies have been measured at BL09-MISTRAL. The company Henkel has signed an agreement for R&D co-operation with BL11-NCD. Regular user operation is progressing well at BL13-XALOC. Fast full EXAFS spectra have been obtained during the reduction process of a copper catalyst at BL22-CLÆSS. Magnetic (XMCD) imaging with a lateral resolution of 20 nm was achieved in the PEEM station at the CIRCE beamline. MCD spectra have been measured at different temperatures from a ferromagnetic bulk Co-oxide sample under an applied field of 5 Tesla in the HECTOR cryomagnet of BL29-BOREAS.

New staff

http://www.cells.es/Jobs

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

Michele Carlà
oPAC – Marie Curie – Beam Dynamics
Michele is a physicist and has joined the Beam Dynamics group of the Accelerator Division on March 4th. He is the recipient of an Early Stage Researcher fellowship of the EU – FP7/People - Marie Curie actions, under the program Optimization of Particle Accelerator, oPAC.

Alain Flores
Convenio España – México
Alain is a Doctor in Physics and will join the Beam Dynamics group from March 20th until the end of 2013, under the agreement between Spain and Mexico to form personnel in Synchrotron Radiation technologies.

Accelerators

http://www.cells.es/Divisions/Accelerators

RUN_01 of 2013 finished on 16/2/2013 at 7h00 having provided 443.4 h for beamlines (BLs), a 98.5% of the foreseen hours. The beam dumps were related to RF trips and to water flow oscillations in the Storage Ring circuit of cooling water.

The storage ring has provided 120 mA with 2 injections a day. Beam stability is within 1 micron.

On the dedicated machine days the Accelerator Division is working hard to improve the reliability of the RF system. Shortly, the Accelerator Division will also start tests that should lead to the implementation of top-up at the end of this year.

Experiments
http://www.cells.es/Divisions/Experiments
http://www.cells.es/Beamlines

*** BL04-MSPD: Materials Science and Powder Diffraction.**

The second beam run (February-March 2013) is being devoted to user experiments at the powder diffraction endstation.

The hot air blower has provided controlled temperature (around 60 degrees) to capillary samples during the first user experiment of the run.

The cryostream will be used as well as sample environment in one of the user experiments of the run.

The first user group of the multicrystal analyzer detector (Mad26) has already performed an experiment. The group is led by Miguel Ángel Camblor from the ICMM (Instituto de Ciencia de Materiales de Madrid, http://www.icmm.csic.es/); and their research is devoted to the structural characterization of as-made zeolites and related compounds. The aim is to get insights on "structure-direction" by locating guest species (organic cations and fluoride anions) and studying the host-guest interactions in the compounds. The diffraction pattern of an as-made zeolite containing organic cations and fluoride anions is seen in Figure 1 (high 2theta angle region shown in the inset). It was measured in 187 minutes with lambda 0.6198 Å up to 48 º (0.76 Å).

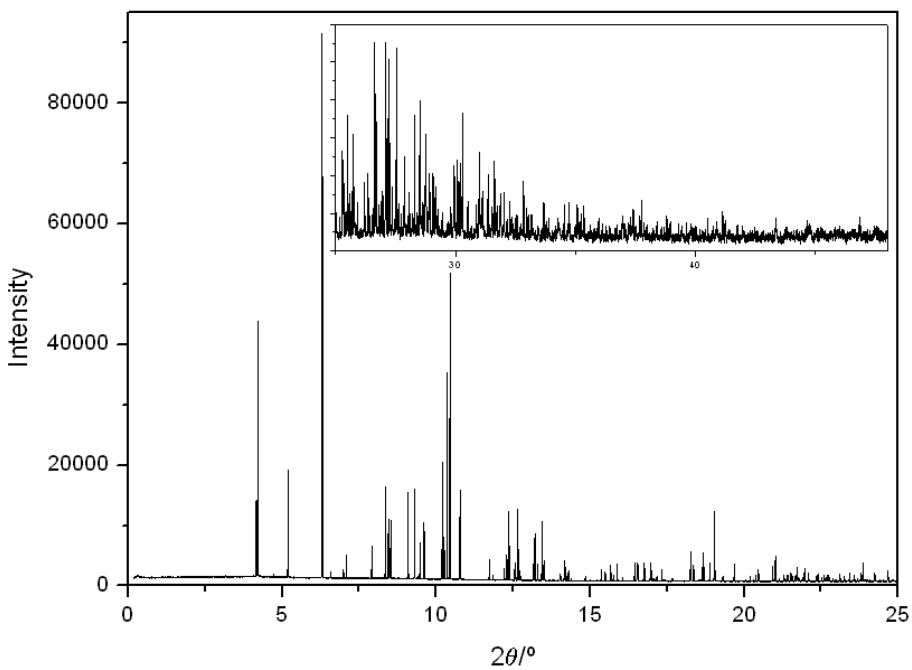

Figure 1. Diffraction pattern of an as-made zeolite containing organic cations and fluoride anions.

*** BL09-MISTRAL: X-Ray Microscopy.**

During the last weeks several groups of external users have obtained results at BL09-MISTRAL.

The group at CNB-CSIC leaded by Dr. F.J. Chichón and Prof. J.L. Carrascosa is interested in the *Vaccinia* virus infection. They have used soft X-ray cryo tomography to reconstruct in three dimensions whole infected Ptk2 cells at different post-infection times to study the cellular rearrangements caused by the virus infection as well as to quantify the number of morphogenetic intermediates in a cell. Figure 2 describes some of these results.

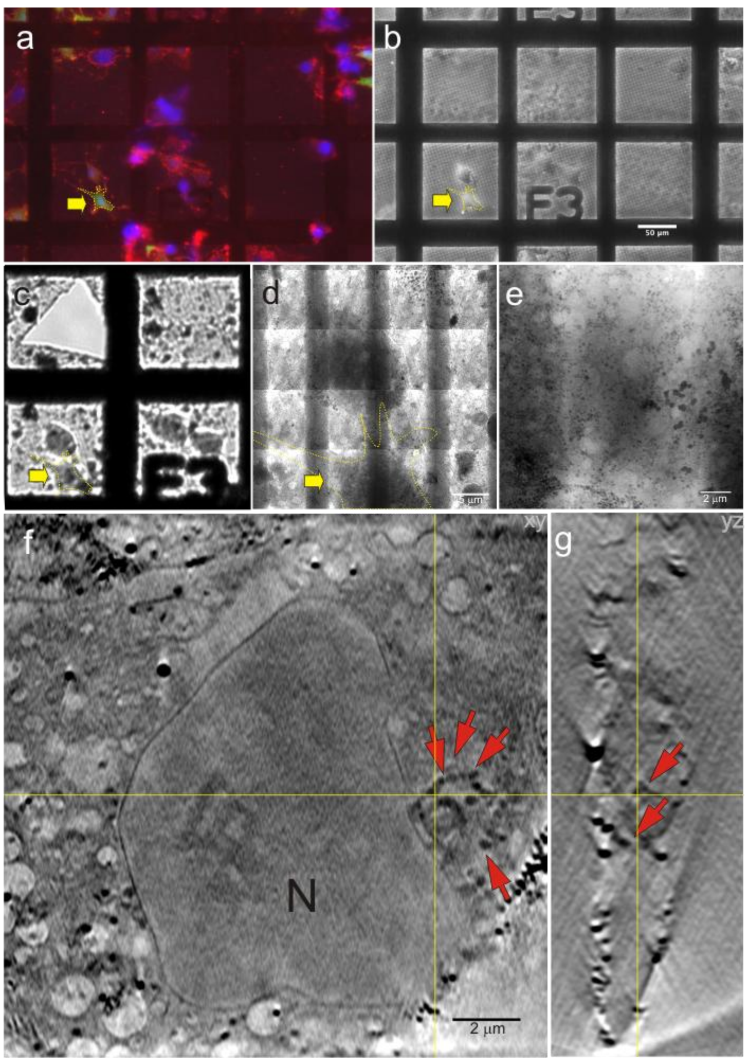

Figure 2. Correlative microscopy of the Ptk2 cells infected with MVA-C-DF1C (GFP). **a**) and **b**) *in vivo* light microscopy of the infected cells growing on holey carbon grid. **a**) Epifluorescent image. In blue, DAPI dye labels the DNA, in red WGA labels membranes of the cell and GFP in green expressed in the infected cells. **c**) Cryo-light microscopy inline with the Transmission X-ray Microscope of vitrified grid corresponding to the same area in a and b. **d**) Mosaic composition on cryo transmission soft X-ray microscopy images of the same area of the grid. Yellow arrows and

discontinuous lines show the position of the same cell from a to d. **e**) 0º soft X-ray projection image of the same cell at higher magnification. **f)** and **g**) Virtual slices of the reconstructed soft X-ray cryo-tomogram xy and yz planes respectively. An N marks the position of the cell nucleus and the red arrows point to the different viral forms of the VV inside the cell.

Another project led by Dr. A. Rosell from Vall d'Hebron Research Institute (VHIR) and Dr. A. Roig from ICMAB (Figure 3) produced the first images of human endothelial stem cells. Preliminary results of the project aimed to image whole unsectioned, unstained endothelial progenitor cells (EPCs) containing nanoparticles by X-ray cryo-tomography. It is expected that the tomographic data sets of 50-60 nm resolution will allow gathering 3D views of the whole cells allowing a comparative study (size and morphology) of the iron-loaded cells with the pristine cells, as well as between the different EPC subtypes. In addition the researchers aim to determine the spatial bulk distribution of the endosomes/lysosomes containing nanoparticles inside the cells.

Figure 3. Group of nanocomposites and nanoparticles at ICMAB-CSIC, Anna Rosell from the Neurovascular Research group at VHIR and Eva Pereiro beamline responsible of BL09-MISTRAL.

Another group of visitors from UK led by Dr. L. Collinson (Cancer Research, UK) and Dr. E. Duke (Diamond Light Source) obtained good quality images of frozen mammalian cells and collected 20 tomographies that are currently being analyzed. Figure 4 shows a section that corresponds to one of the data sets. Note that the cell membrane exhibits a clearly visible two-layer structure (shown by the arrow). The bilayer spacing is approximately 20 nm which illustrates the resolution of the microscope.

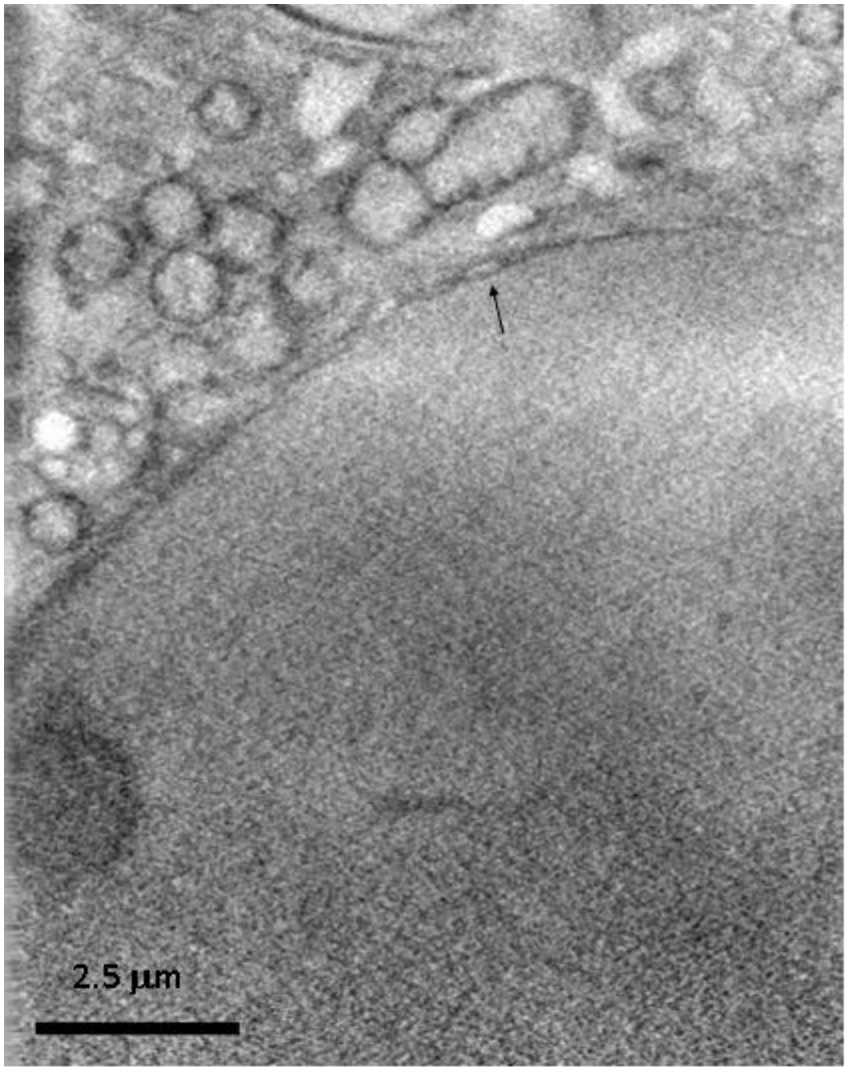

Figure 4. Image of a frozen mammalian cell collected at the MISTRAL beamline using cryo-soft X-ray tomography. The image shows the nucleus of the cell, enclosed by the nuclear envelope. Structures of around 20 nm can be detected in whole unstained cells. The aim of the project is to find and analyze infoldings of the nuclear envelope that have specific lipid compositions, which may be involved in diseases such as cancer. The work was performed in collaboration with the Cell Biophysics and Electron Microscopy Units at Cancer Research UK led by Dr. L. Collinson and Dr. E. Duke at the Diamond Light Source in the UK.

In addition to these users' experiments, the control software to be able to collect automatically energy stacks (keeping the sample in focus and constant magnification) has been tested and appears to work satisfactorily opening the possibility to perform spectroscopic imaging. Further commissioning on tests samples is planned.

*** BL11-NCD: Non-Crystalline Diffraction.**

Recently the beamline has moved from being a very new beamline with various minor problems to being a beamline that operates under steadier conditions. The data collection protocol is continuously being upgraded and hopefully improving following suggestions from the users. The current CCD detector is performing more reliably. From this point of view February has been an important time period for BL11-NCD.

a- Early February saw a user group from the CSIC - Institute of Agrochemistry and Food Technology Institute from Valencia - that recorded data from samples in solutions for the first time on the beamline. It was generally agreed that the data recorded were of good quality.

b- In the middle of February the beamline received a group of scientists from CSIC Institute of Polymer Science and Technology (Madrid) who dedicated five days to recording meso-phases in polymeric systems, again making use of the WAXS system.

c- Beamline staff recorded data for the industrial company Henkel that had provided a number of samples that required WAXS recordings as function of temperature and specific cooling velocities. Later in the month CELLS and Henkel signed an agreement for R&D co-operation.

d- Finally, for the first time anomalous small angle scattering, ASAXS, data have been recorded with the end station configured for a sample to detector distance of approximately 2.3 m. The scope of the scientific group from ICN - Magnetic Nanostructures Group in Cerdanyola del Vallès, was to measure the structure of nano-composites made of Fe_3O_4 3-dimensional structures embedded in ordered meso-porous Co_3O_4 templates.

During the ALBA shutdown in February, parts that were supplied by Bruker Advanced Supercon earlier in the year replaced the former internal water thermal stabilization circuit inside the double crystal monochromator, DCM, during an intervention that lasted half a day. After that the DCM underwent a bake-out for 10 days. Colleagues from the engineering division carried out the repair work.

We are happy to welcome and work with our new Master student, Dra. E. Moreno Calvo, into the NCD group. She will be carrying out structural analysis of vesicles in solutions from x-ray solution scattering data.

*** BL13-XALOC: Macromolecular Crystallography.**

As mentioned in the previous newsletter, we are currently allocating beamtime on a 24-hours basis (3 shifts) due to the increased reliability of the beamline.

The programs SHARP and autoPROC are now accessible at the beamline. More information about all the software available at the beamline can be found here.

We are advancing in the design and implementation of the crystal diffraction characterization and diffraction strategy programs (EDNA) and a new and user-friendlier version of the control software of the sample.

The mini-kappa can be mounted on the diffractometer on request, currently the maximum angle of the kappa angle is ~ 45 º, we are working on several design modifications of the cryostream translation table that should allow larger values of the kappa angle (Figure 5).

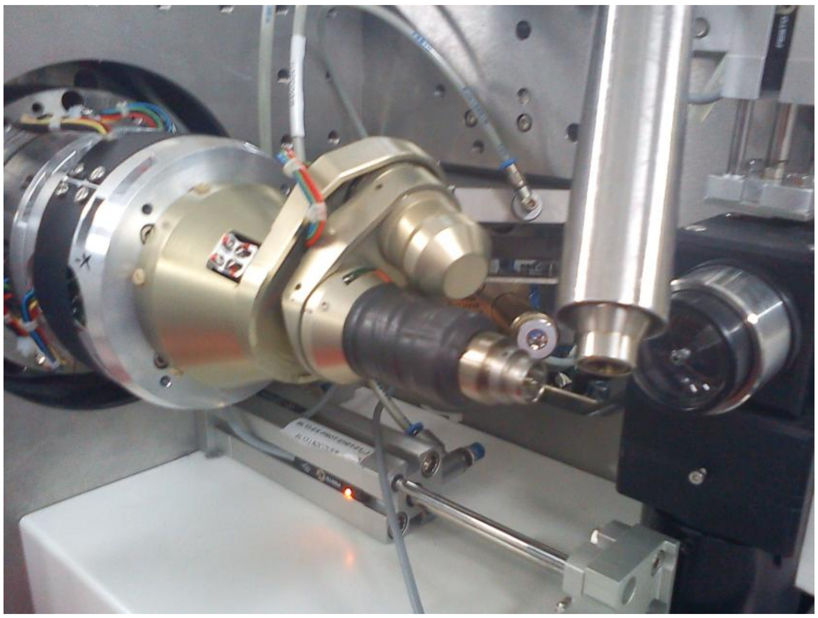

Figure 5. The mini-kappa mount in the diffractometer of BL13-XALOC.

*** BL22-CLÆSS: Core Level Absorption & Emission Spectroscopies.**

The most remarkable achievement of the last months at BL22-CLÆSS, from an instrumental point of view, has been the implementation of the "continuous scan" by the Controls group of ALBA. This continuous scan, also known as 'quick-scan', allows us to move the double crystal monochromator very quickly so that it enormously decreases the acquisition time of the absorption spectra. At present the maximum number of points per scan that can be taken is 1000 and it will be doubled shortly.

The next figure (Figure 6) depicts the temporal evolution of the XANES region of a set of full EXAFS spectra obtained during the reduction process of a copper catalyst. The energy step of the scan is 1.2 eV.

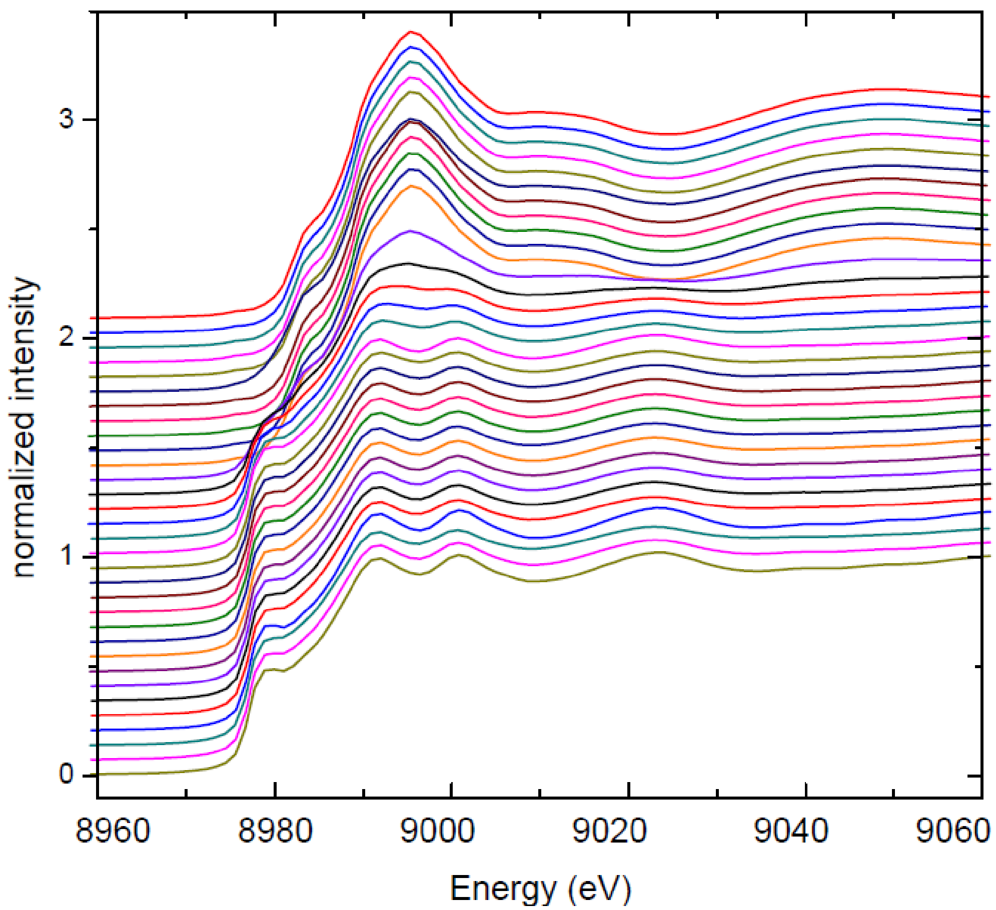

Figure 6. Temporal evolution of the Cu K XANES absorption spectra during the reduction of a Cu catalyst at 250 ˚C in a gas mixture (5 % H_2/He) at 1 bar total pressure. The catalyst was a pellet of 20 % Cu on an Al_2O_3 matrix used for methanol synthesis. Complete EXAFS scans of 1200 eV were acquired in 5 minutes in the continuous scan operation mode. Note the evolution from oxidized Cu^{2+} species to metallic copper. This experiment is part of a Long Term Project from the group of Dr. A. Urakawa (Institut Català d'Investigació Química, Tarragona) in collaboration with the ALBA team.

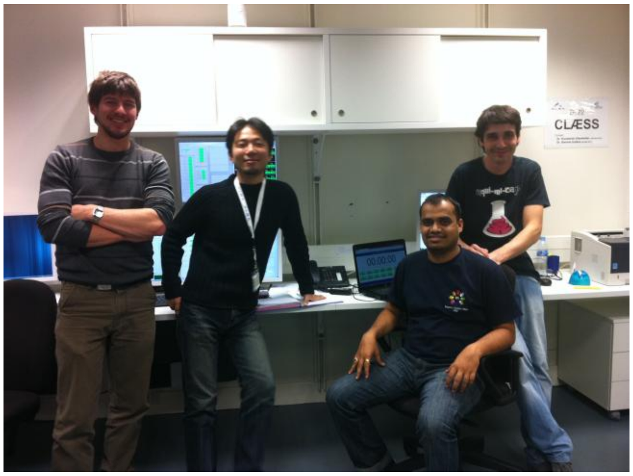

Figure 7. Several members of the group of Dr. A. Urakawa (Institut Català d'Investigació Química, Tarragona).

The same group also performed an experiment which involved a high pressure (200 bar) flow reactor specially designed for catalysis experiments (Figure 8). The mixture of the reactants at high pressure is introduced into the capillary from the left. The capillary is filled with a catalyst and it is irradiated with the X-ray beam to collect transmission EXAFS data using the continuous scan mode.

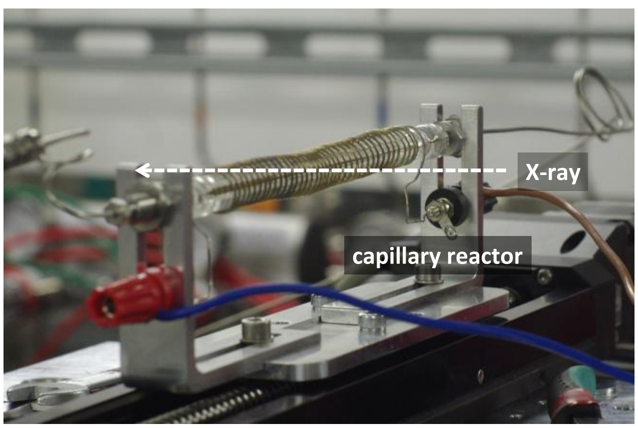

Figure 8. The high-pressure flow reactor as installed at the beamline.

*** BL24-CIRCE: Photoemission Spectroscopy and Microscopy.**

Magnetic (XMCD) imaging with a lateral resolution of 20 nm was achieved in the PEEM station at the CIRCE beamline (Figure 9).

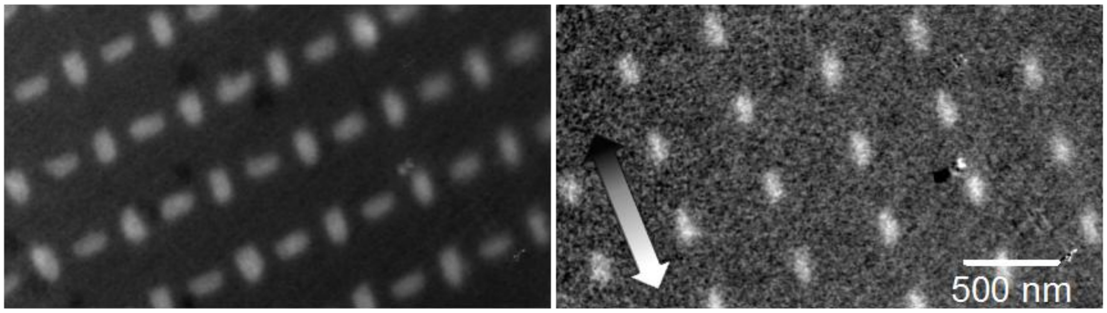

Figure 9. Left: Permalloy (NiFe) nanostructures imaged by photoelectron microscopy (PEEM) using X-rays tuned to the FeL3 absorption edge. Nanostructures of dimension 80 nm x 200 nm x 30 nm (W x L x H) appear bright, while the Si background is dark (chemical contrast). **Right**: XMCD image of the same region (difference between images with left/right handed circular polarized X-rays), showing purely magnetic contrast. The direction of contrast (parallel to incident beam) is indicated by the arrow. Due to their shape anisotropy, all nanostructures are magnetized along their respective long axis. The XMCD image highlights magnetization along the beam direction (white), while the structures with magnetization perpendicular to the beam disappear into the background (gray). The lateral resolution (step width) in the XMCD image is 20 nm, demonstrating the high resolution of the PEEM endstation at BL24-CIRCE. (Sample courtesy of P. López-Barberá & J. Nogués from ICN Barcelona and J. Perron & L. Heydermann from PSI Switzerland).

The photon beam at the NAPP branch was successfully aligned through the newly installed differentially pumped beam entrance. The setup, comprised of three apertures and a capillary on each of the independent alignment tables, allows maintaining nine orders of magnitude of pressure difference between the beamline and the sample environment.

The water chiller and vacuum forepumps have been moved away from the beamline, outside the critical slab, in order to reduce vibrations and ambient noise.

Users from Centro de Astrobiología de Madrid and Instituto de Ciencia de Materiales de Madrid have been performing experiments at the BL24-CIRCE PEEM from February 27th until March 6th.

*** BL29-BOREAS: Resonant Absorption and Scattering.**

The fluorescence yield diode was installed and its commissioning has started. This diode is an AXUV100 silicon photodiode with an almost constant responsivity in the photon energy range 100-6000 eV and very high quantum efficiency. The diode is mounted on a linear arm (
Figure 10) to allow a close positioning (~5 mm) with respect to sample surface. All the design and construction of the linear arm has been performed at ALBA.

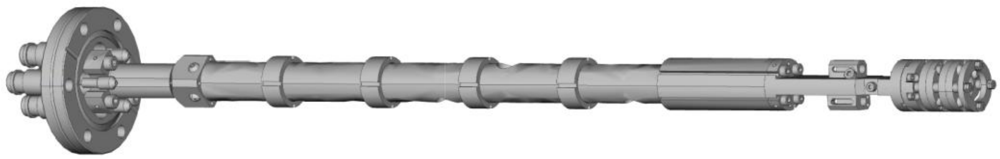

Figure 10. **Fluorescence diode assembly designed and built at ALBA.**

Although the diode will be available for user experiments from April 1st, the first spectrum was acquired during the last user beamtime (Figure 11):

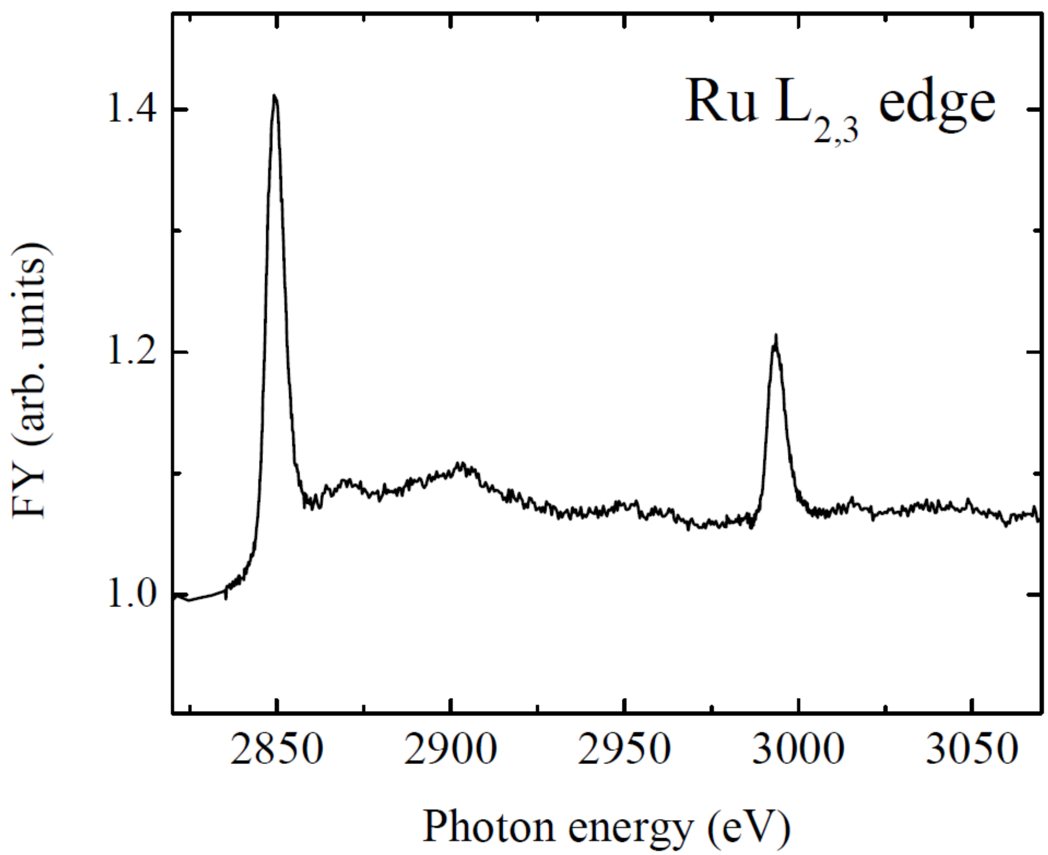

Figure 11: Ru L2,3 edge collected with the fluorescence yield detection diode.

The metrology tests of the cryomanipulator sample positioning for the MARES scattering station are being performed. Some representative results are: main eigenfrequency at ~6 Hz with rms vibration amplitude of 0.25 µm, repeatability of z sample coordinate: 2 µm.

In the following figure (Figure 12), we can see several illustrative results from users' experiments:

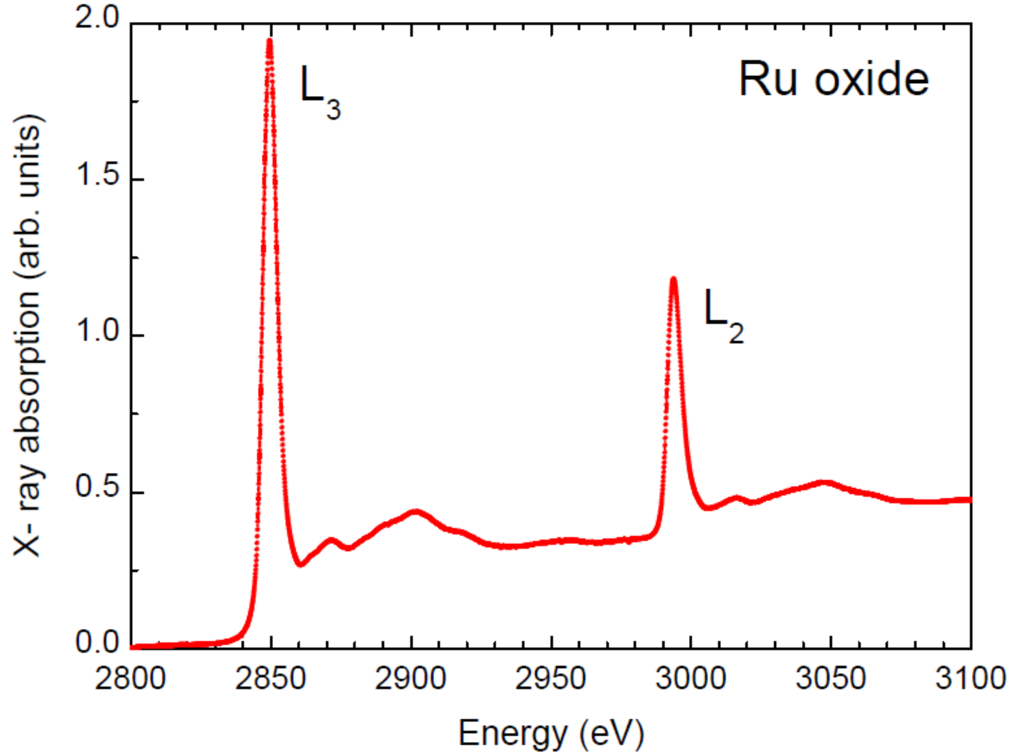

Figure 12: XAS signal of a film of Ru-based oxide with linear horizontal polarized light in grazing incidence (10°). This shows the good performance of BL29-BOREAS beamline in the soft to hard x-rays transition region.

The Ru L2,3 signal has been collected with TEY with a sample current of around 100 pA to be compared with the usual signal measured on transition metal oxides which is in the range 4-10 nA. The quality of the data in Figure 12 (0.5 sec per energy step) illustrates that this energy range is well suited for experiments at BL29-BOREAS.

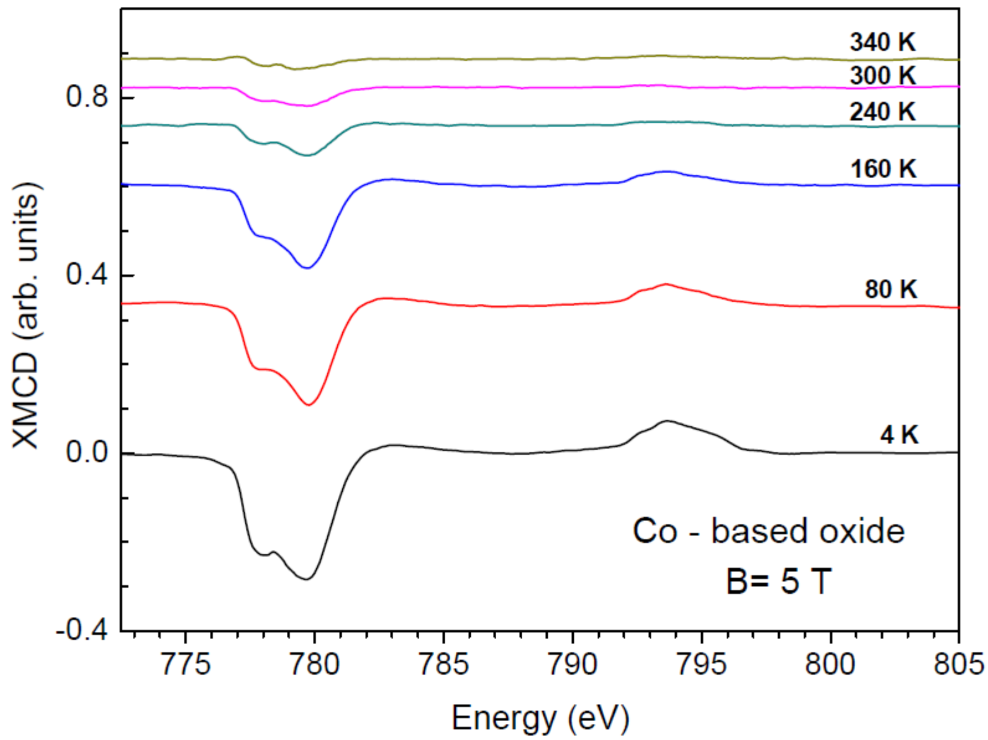

Figure 13: MCD spectra at different temperatures from a ferromagnetic bulk Co-oxide sample under an applied field of 5 Tesla in the HECTOR cryomagnet.

The variable temperature regulation of HECTOR cryostat allows performing temperature-dependent XMCD experiments. In Figure 13 we can see an example of the XMCD signal of Co-oxide sample as a function of temperature and collected during the last run of the in-house research program.