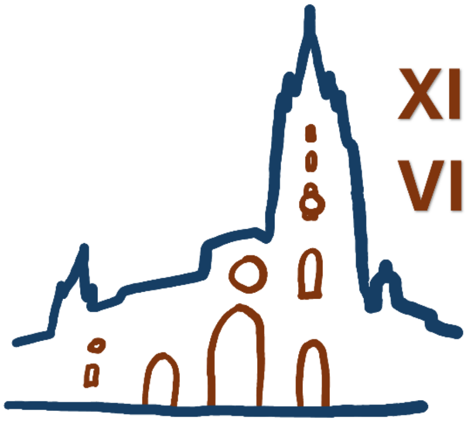

XI AUSE Conference
VI ALBA Users Meeting

Oviedo
2 – 6 September 2024

Book of Abstracts

2-6 September 2024, Oviedo, Spain

AUSE ASOCIACIÓN USUARIOS SINCROTRON ESPAÑA
Universidad de Oviedo
ALBA
Spain XFEL HUB

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Welcome Message

Dear Participants,

As Director of the ALBA Synchrotron, President of the Spanish Synchrotron User Organization - AUSE - and Chair of the Local Organizing Committee, **we want to invite and welcome you to the joint 11th AUSE conference and VI ALBA User Meeting** at the town of Oviedo. In a time of new funding opportunities and the need to address the big challenges of our time, the meeting will give you a platform for celebrating great scientific success, exchange and align within the community and learn more about what is planned to make your X-ray research infrastructures ready for your needs. Perhaps even more important, it will give you a voice to communicate your needs as well as ideas to improve the services.

This conference joins, as usual in the last decade, the **biannual AUSE meetings** with **the ALBA User meeting,** reflecting a long success story of community growth and community engagement with its X-ray research infrastructures. Alternating between hosting universities and ALBA as venue, the meeting series is a symbol and expression for the great integration of the facility with its user community and the open welcome culture to new communities.

In 2024 we will profit from the renown hospitality of the town of Oviedo, where we expect to deliver a full and exciting program. This is a very interesting period for ALBA to meet with the community, since the foundations for its future, ALBA II, are being laid down, and the constructive exchange with its users is of outmost importance.

The meeting is connecting three building elements: the first one dedicated to the **AUSE activities**, including an update on the **Spanish ESRF CRG beamline SpLine**, the second focused on **ALBA and its upgrade ALBA II,** and the last session dedicated to the **Spanish XFEL Hub activities**.

The conference is expected to bring together a group of about 150 researchers, where we anticipate a high number of students and post-docs who will be shaping the future of X-ray science. Several plenary lectures by key-note speakers, a significant amount of oral contributions as well as extended poster sessions are planned. Several prizes will be awarded to the best posters presented by young researchers.

Looking forward to meeting you all in Oviedo!

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Day 1, 2nd September 2024

PLENARY Lecture I

Diamond Light Source: Science and updates on the Diamond-II upgrade #174

Author: *Gianluigi Botton (Diamond Light Source)*

In this presentation a review of some of the recent science highlights from the facility will be given. Then, examples of the science drivers that motivated a 4th generation diffraction-limited storage ring upgrade for Diamond will be provided, with timelines and current state of upgrade. The facility will see three new flagship beamlines, maximizing the benefits of improved brightness and coherence, then several additional upgrades to multiple beamlines and new software architecture to enable new scientific experiments and new data acquisition and processing pipelines.

SESSION A - Soft Condensed Mater

Applications of synchrotron radiation in polymers science: techniques and sample environments #192

Author: *Mari Cruz García-Gutiérrez (IEM-CSIC)*

The great potential of synchrotron light is of special interest for dealing with the intrinsically weak X-ray scattering power of soft condensed matter in general and polymers in particular, associated to the low level of order and to its small electron density contrast. Scattering techniques have been widely used for studying the structural heterogeneity of polymer systems. Simultaneous Small Angle X-ray Scattering (SAXS) and Wide Angle X-ray Scattering (WAXS) is a well suited technique for investigating phase changes or conformational rearrangements on a length scale ranging from 1 to 500 nm. Static or time resolved measurements can be performed with simultaneous SAXS and WAXS area detectors. Investigation of thin films at different length scales is also possible by using grazing incidence geometry, GISAXS and GIWAXS. Modern application of synchrotron radiation to polymer science has expanded significantly. Besides scattering and diffraction, new techniques have been incorporated showing that synchrotron light in polymer science can go well beyond traditional structural characterization. By using soft X-rays (Eph < 2500 eV) and tuning the X-ray photon energy close to and within the fine structure of a constituent element absorption edge, unique chemical sensitivity among components in a heterogeneous material is obtained. Resonant soft X-ray scattering methods have been developed combining conventional scattering with soft X-ray spectroscopy, thus offering enhanced and tunable scattering contrast.

Examples of the application of the above techniques, as well as different sample environments, will be shown: in-situ scattering techniques to deal with structure-property relationship in fully biobased polymers for packaging [1] and with the fluorescence changes of regenerated silk fibroin induced by deformation, the use of nano-focus techniques to deal with confinement effects of conjugated polymers [2], the investigation of nanostructured thin films by grazing incidence X-ray scattering [3] and the use of resonant soft X-ray scattering to deal with nanophase separation in P3HT/PCBM bulk heterojunctions [4].

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Structure development during “in-operando” 3-D printing using simultaneous Small and Wide-Angle X-ray Scattering measurements #122

Authors: *Esther Rebollar (Instituto de Química Física Blas Cabrera, IQF-CSIC)*
Co-Auhtors: *Tiberio Ezquerra, Aurora Nogales, Mari Cruz García-Gutiérrez, Esther Rebollar, Igors Sics, Marc Malfois*

The performance of simultaneous SAXS/WAXS with synchrotron radiation can be used to characterize structure development during “operando” 3D printing Fused Filament Fabrication (FFF). The performance of the set-up can be successfully applied to evaluate the crystallization of 3D printed thermoplastic polymers. The real time evolution of the crystallization of different parts of the printed specimen can be evaluated with resolution below 50 μm. Thus, simultaneous SAXS and WAXS experiments can be performed across the lines (roads) and welding zones with a resolution of 50 μm. Here we will show experiments in different thermoplastic polymers indicating that crystallization/solidification proceeds faster at polymer-air interfaces than in other parts within the printed line. The final crystallinity can be position dependent being typically lower at polymer-air and welding zone. Orientation of the crystalline lamellae can be higher at both interfaces suggesting higher shear rate than in the bulk of the printed line where elongational flow is dominant. The final crystallinity levels at different locations within the printed line can be relatively low for standard printing conditions. This can be attributed to the fast crystallization kinetics involved in the solidification of the printed lines. This evaluation can be useful in order to minimized structural modifications due to the post printing crystallization that eventually will proceed during storage in the case of polymers with glass transition below room temperature.

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This work has been funded by the Spanish State Research Agency (AEI) and by the European Regional Development Fund (ERDF) “A way of making Europe” through projects TED2021-129845B-I00, PID2022-138635NB-I00 and PID2019-106125GB-I00. SAXS/WAXS experiments were performed at NCD-SWEET beamline at ALBA Synchrotron with the collaboration of ALBA staff.

Monitoring the structural transformation on thin films of an Asymmetric Benzothieno[3,2-b][1]-benzothiophene Derivative #120

Author: *Alba Cazorla Moreno (Institut de Ciència de Materials de Barcelona (ICMAB-CSIC))*
Co-authors: *Carmen Ocal, Shunya Yan, Esther Barrena, Eduardo Solano*

In recent years, there has been significant interest in the use of π-conjugated organic semiconductors for organic electronic devices, such as organic field effect transistors (OFETs). Particularly, [1]benzothieno [3,2-b]benzothiophene (BTBT) derivatives have emerged as promising candidates for p-type OFETs, exhibiting excellent performance under ambient conditions with mobility values exceeding 10 cm2V-1s-1, comparable to silicon devices. Despite the latest advancements, structural control is still a challenging aspect for optimizing the devices performance.

In this work, we focus on an asymmetric BTBT derivative named 7-decyl-2-phenyl[1]-benzothieno [3,2-b][1]benzothiophene (Ph-BTBT-10). The asymmetry of the molecule causes crystallization with a bilayer herringbone structure, where the molecules stack on top of each other in a head-to-head (or tail-to-tail) fashion, resulting in a bilayer stacking in the single-crystalline form. However, this molecule exhibits different crystal packings in thin films, which critically determines the performance of the OFETs. [1] The high carrier mobility in OFETs based on Ph-BTBT-10 thin films has been linked to the structural transformation from the metastable thin-film phase to the thermodynamically stable BL stacking via thermal annealing [2]. However, crystal structure is not the only aspect to consider since inhomogeneous strain on the film has been also reported to impact the performance and reliability of the devices [3]. Here, we employ a combined investigation by Atomic Force Microscopy (AFM) and Grazing Incidence Wide Angle Scattering (GIWAXS) to monitor *in situ* the structural transformation induced during thermal annealing.

By growing the thin films on different substrates, we aim at clarifying the effect that the surface has on interfacial strain and thermal expansion.

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Structure and pressure gap in the NO reduction on Rh studied by ambient-pressure HAXPES #119

Author: *Fernando Garcia Martinez* (Deutsches Elektronen-Synchrotron, DESY)
Co-authors: *Sabine V Auras, Patrick Loemker, David Degerman, Christopher Goodwin, Bernadette Davies, Sara B. Bibi, Markus Soldemo, Frederik Schiller, Anders Nilsson, Christoph Schlueter, Jose Enrique Ortega*

The reduction of NOX towards N2 plays a significant role in decreasing the emissions of pollutants from combustion engines to the atmosphere [1]. To allow the reaction and subsequent product formation, NO molecules must dissociate at the surface of a Rh or Ir catalysts. In addition, Ir is selective to N2, while a small amount of N2O is also formed when Rh is used [2]. NO reduction literature is not abundant at ambient pressures (≥1 mbar, AP) due to the difficulties of high-pressure experiments, and therefore several aspects remain largely unknown.

We report an AP-HAXPES (AP - Hard X-Ray Photoemission Spectrostopy) investigation of the NO reduction on Rh surfaces. Experiments were conducted in the POLARIS end-station (Stockholm Uni) located at beamline P22 (DESY, Hamburg). This unique instrument is the first in the world that routinely gathers spectra in the bar range pressure, several orders of magnitude higher than conventional AP-XPS (X-Ray Photoemission Spectrostopy) experiments carried out at lower photon energy [3].

To evaluate a possible pressure gap, we exposed a Rh(111) sample to 150 mbar, 1:1 CO:NO mixture and heated the sample. The aim is to initiate the reaction, and trigger the transition from a NO-covered, inactive Rh surface towards a N-covered, active Rh surface [2]. We show the active stage of the reaction for Rh(111) at two different total pressures. Sizeable XPS signals arising from N2O and N2 are detected at 150 mbar. In contrast, very low N2O production is observed at 1 mbar in previous experiments. In order to delve into the structure dependence of the NO reduction, we compared the ignition of a flat Rh(111) and stepped Rh(112) surfaces. We observed that, under the same temperature range, the Rh(112) surface yields a significantly larger amount of N2 as compared to the Rh(111) surface, showing the higher activity of the stepped surface. The N2O production is lower than that of N2 for both surfaces, yet the Rh(111) yields a larger amount of this gas. Our results clearly demonstrate how the NO reduction on Rh is both structure and pressure dependent, and emphasizes the need of high-pressure experiments.

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Session B - Advanced Materials for Energy & Sustainable Applications

Understanding physical and chemical properties of energy storage materials with synchrotron radiation #197

Author: *Enrique G. Michel (IFIMAC, UAM)*

Understanding the physical and chemical properties of materials involved in energy storage, in particular in Li-ion and Na-ion batteries, is of paramount importance given the need to store the energy produced by renewable energy sources and the critical need to decarbonize transportation.

In this talk, I will review the use of synchrotron radiation in three cases that span several different techniques and both fundamental and applied research. For conventional liquid electrolyte batteries, the delithiation process of LiCoO2, a common cathode material, and the role of phase coexistence in determining its behavior will be described using ARPES, XAS and PEEM. The degradation processes of solid-state batteries and the strategies to mitigate them are considered in the second example, where HAXPES and XPS are used to evaluate the properties of a solid electrolyte interface and to develop a strategy to mitigate degradation. Finally, the zero-excess Na-ion battery approach is described, including its potential advantages with respect to other technologies, and the application of PEEM and LEEM to understand the in-situ formation of the Na anode and how to overcome the problems associated with it.

Controlling the number of coordinatively unsaturated iridium oxide active centers for optimize the electrocatalytic oxygen evolution reaction #202

Author: *Juan J. Velasco Vélez (ALBA Synchrotron)*

Water splitting is considered one of the most important electrochemical processes for the storage of renewable energy by the cathodic production of molecular hydrogen, which is used as a carbon-free energy vector. In addition, the electrocatalytic path of molecular hydrogen production yields cleaner stocks compared to other technologies, but at a higher cost. It is, in part, due to the sluggish anodic oxygen evolution reaction (OER) limits significantly the efficiency of this technology and, as a consequence, its economic viability. Moreover, the electrode corrosion under acidic conditions and anodic polarization shortens materials lifetime with the consequence of the increase of the costs. Among all the materials investigated for the OER, iridium and its oxides/hydroxides combine uniquely the lowest overpotential and highest corrosion resistance under harsh anodic acidic conditions. Unfortunately, iridium is also one of the rarest and scarcest elements on earth, thus synthetic strategies for its optimal usage are required.

We reason that determining the electronic structure characteristics responsible for the peculiar reactivity of IrOx during OER is key to extend these unique properties to a more abundant material. Unfortunately, the element specifics analytical techniques able to provide interface information are very limited and hardly compatible with liquids allowing usually only ex situ characterizations methods. This limitation led to a loss of important information as in many cases the reaction intermediates and electrocatalytic actives species cannot be determined in post process analysis.

Using a special membrane electrode assembly to measure operando X-ray absorption spectra and resonant photoemission spectra of iridium mesoporous oxide films (calcined at different temperature to mediate the catalytic activity), together with ab initio simulations, it was possible to unambiguously distinguish the formation/presence of µ2-O (bridging oxygen) and µ1-O (terminal oxygen) in the near surface regions of the catalysts during OER conditions. It was found that the intrinsic activity of iridium oxide scales with the formation of µ1-O (terminal oxygen), meanwhile the peroxo species do not accumulate under reaction conditions. Thus, the µ1-O is the key specie in the formation of O－O bond during the OER.

Synchrotron radiation to solve the puzzle of capacity limitation in Aqueous Zn-MnO2 Batteries #141

Author: *Cheng Liu (Instituto de Ciencia de Materiales de Barcelona, CSIC)*
Co-authors: *Vlad Martin-Diaconescu, Andrea Sorrentino, Laura Simonelli, Tonti Dino*

Manganese-based cathodes such as αMnO2 have been attracting enormous interest for aqueous Zn metal batteries, due to their low-cost, suitable potential and considerable reversible capacity. The electrochemical mechanism is generally believed to mainly consist of the dissolution of MnO2 into Mn2+ with simultaneous precipitation of Zn4SO4(OH)6·xH2O (ZHS) buffering the pH:

MnO2 + 2H2O + 2e- ⇌ Mn2+ + 4OH- (Eq.1)

3Zn2+ + 6OH− + ZnSO4 + 5H2O ⇌ ZnSO4[Zn(OH2)]3 · 5H2O (Eq.2)

However, the electrochemical potential profile of first discharge differs from subsequent discharges, and most importantly, the experimentally delivered capacity is typically below 400 mAh g-1, in stark contrast with the ca. 600 mAh g-1 expected theoretically based on the 2-electrons reaction (Figure 1A-B). We used several X-ray techniques both operando and ex-situ to spot or rule out the possible underlying processes, by determining the involved species qualitatively and quantitatively with time and length resolution (Figure 1C-D). The reaction of active Mn(IV)O2 to Mn2+ in electrolyte was indeed confirmed by X-ray Absorption Spectrum (XAS) in operando, while a possible competing one-electron reaction of Mn(IV) to Mn(III) can be ruled out. In-situ soft X-ray Transmission Microscopy (sTXM) and X-ray Kβ Emission Spectrum (XES) revealed almost no evolution of Mn(III), suggesting that at most it could only be considered a short-lived intermediate. The relationship between Zn and Mn carefully modelled by the edge jump of operando XAS, and MCR-ALS, indicated declining formation of ZHS, which points to a possible MnO2 passivation mechanism.

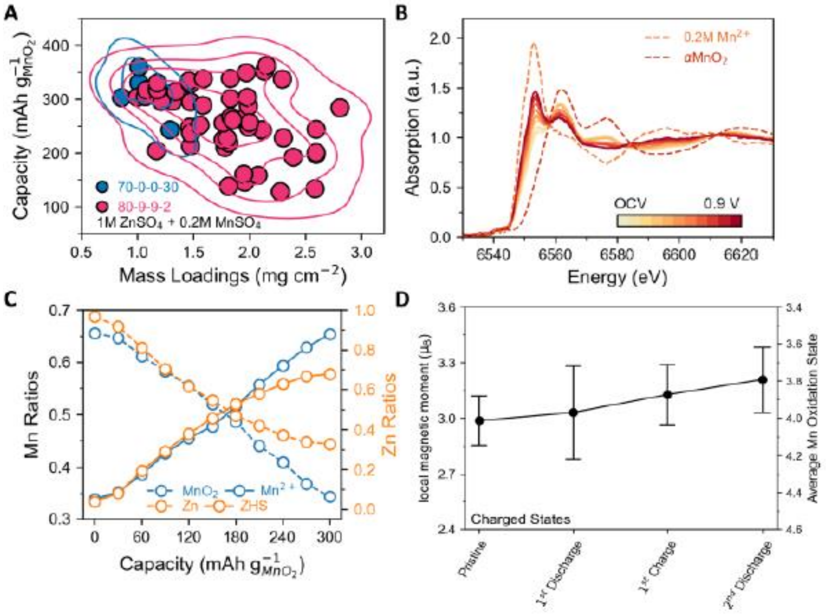

Figure 1. Electrochmical 1st discharge capacity at different electrode loading (A). Operando XANES of the Mn K edge during 1st discharge (B). Comparison of Mn and Zn evolutions obtained from MCR-ALS (C). Magnetic moment and and average Mn oxidation state at different states of charge obtained from ex-situ Kβ emission spectra (D)

The role of the local structural properties in the electrochemical characteristics of $Na_{1-x}Fe_{1-y}Ni_yO_2$ cathodes #123

Authors: *Wojciech Olszewski (Faculty of Physics, University of Bialystok)*
Co-Authors: *Sourav Baiju, Payam Kaghazchi, Carlo Marini, Benoit Mortemard de Boisse, Masashi Okubo, Atsuo Yamada, Takashi Mizokawa, Naurang Lal Saini, Laura Simonelli*

The natural abundance of sodium makes the Na-ion batteries (SIBs) attractive devices in the framework of a global economy [1]. SIBs naturally deliver relatively lower energy density respect to Li-ion counterparts (LiBs), however, their lower cost and fast charge/discharge-ability make them a promising competitor to LiBs to load level the intermittent power from renewable energy sources for smart grids or renewable power stations [2].

The O3-type NaFeO2 is a promising candidate for SIBs cathodes, even if the irreversible structural transition occurring during Na-ion extraction/insertion seriously hinders its practical application [3]. Partial replacement of Fe by Ni significantly improves its electrochemical properties [4]. Therefore, it is crucial to investigate how such substitution affects the local electronic and structural properties and in consequence Na-ion mobility and charge compensation mechanism.

Here, we report the effect of Ni for Fe substitution on the local structural disorder, lattice stiffness and consequent local electronic changes during Na (de)intercalation in O3-$Na_{1-x}Fe_{1-y}Ni_yO_2$ oxide (y = 0 and 0.5) using X-ray absorption and emission spectroscopies. Obtained results are compared with the results of the spin-polarized DFT calculations. The results support the stability of the electronic properties of Fe and Ni as a function of cycling in a partially substituted system, while the decrease of Fe-O covalency and the local disorder changes are at the origin of the improved performances.

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Day 2, 3rd September 2024

PLENARY Lecture II

XChem: High throughput crystallographic fragment screening and structure enabled fragment progression #171

Author: *Blake Balcomb (Diamond Light Source)*

Crystallographic fragment screening and high-throughput structural biology, powered by the XChem facility at Diamond Light Source, is central to hit identification and lead optimisation drug discovery campaigns. Part of performing such large-scale structural biology experiment is based upon the premise of structurally enabling a target which is critical for efficient fragment hit-to-lead and lead optimisation. Literature surveys of known fragment-to-lead medicinal chemistry campaigns over the past eight years have shown that nearly 70-80% of all campaigns generated a structure for either hit or lead chemical matter. With 100% of all campaigns using structural information during lead optimisation. Beyond a crystallographic fragment screen, remains the question as to how one can reliably and efficiently progress fragment hits with the goal of on-scale potency. This includes retaining the shape and colour of the base compound in lead generation.

In this presentation I will highlight the value of having structurally enabled drug discovery campaigns with a focus on how high-throughput structural biology and interaction-driven algorithmic methodologies can accelerate fragment progression strategies and ameliorate the design-make-test cycle.

SESSION A - Drug Discovery

Time-resolved Crystallography: A New Frontier in Drug Discovery #165

Author: *José Manuel Martín García (Institute of Physical Chemistry Blas Cabrera (IQF-BC), CSIC)*

Structural biology, especially X-ray crystallography, has significantly advanced our understanding of diseases by revealing the 3D structures of proteins. However, traditional X- ray crystallography encounters challenges, such as difficulties in obtaining suitable protein crystals and studying protein dynamics. X-ray free-electron lasers (XFELs) have transformed this field with their bright and brief X-ray pulses, allowing for high-resolution structures of radiation-sensitive and hard-to-crystallize proteins. With XFELs, structural biology is also shifting its focus from determining static structures to exploring the dynamic aspects of protein function. A key tool for investigating these dynamics is time-resolved crystallography (TR-SX), which has emerged as a transformative approach in drug discovery, offering unprecedented insights into the dynamic processes of protein function. Traditional X-ray crystallography provides static snapshots of protein structures, which, while valuable, do not capture the full complexity of molecular interactions and conformational changes critical to drug efficacy. TR- SX, leveraging the capabilities of XFELs and advanced synchrotron sources, allows researchers to observe proteins in action at atomic resolution and in real-time. This technique employs ultra-fast, intense X-ray pulses to trigger and capture transient states and intermediates in biochemical reactions, providing a dynamic view of protein-ligand interactions, enzyme mechanisms, and conformational changes. These insights are crucial for the rational design of drugs with improved specificity and efficacy. In my talk I will review the recent advancements in time-resolved crystallography, highlights its impact on understanding protein dynamics, and discusses its potential to revolutionize drug discovery by enabling the design of next-generation therapeutics. The challenges and future directions in the broader application of this method to a wide range of biologically relevant proteins and drug targets are also outlined.

Structural approaches to identify novel inhibitors of IMP dehydrogenase enzymes #138

Author: *Rubén Martínez-Buey (Universidad de Salamanca)*

Inosine 5'-monophosphate dehydrogenase (IMPDH) is an evolutionarily conserved enzyme that catalyzes the first committed step in the *de novo* GTP biosynthetic pathway and plays a pivotal role in nucleotide metabolism. IMPDH is tightly regulated through various mechanisms, including transcriptional control, allosteric modulation, enzyme filamentation, and post-translational modifications.

IMPDH is a key target for several clinical drugs, including immunosuppressive agents like mycophenolate mofetil and mizoribine, and antiviral agents such as ribavirin. These drugs inhibit IMPDH activity, reducing the guanine nucleotide pool and subsequently inhibiting cell proliferation.

Our research focuses on deciphering the structural dynamics and regulatory mechanisms of IMPDH enzymes, paving the way for the development of more effective therapeutic agents. In this presentation, I will discuss the structural approaches employed in our laboratory, particularly X-ray small-angle scattering and X-ray crystallography, to identify novel inhibitors targeting both the catalytic and allosteric sites of IMPDH.

Fostering innovation in drug discovery by automated data collection and X-ray based fragment screening at ALBA #194

Author: *Stefano Pernigo (ALBA Synchrotron)*
Co-Authors: *Roeland Boer, Xavi Carpena Vilella, Isidro Crespo García, Fernando Gil-Ortiz, Guillem Prats Ejarque, Aleix Tarrés Solé, Núria Valls Vidal*

Fragment based drug discovery (FBDD) is a well-established approach to develop new drugs in which libraries of small organic molecules are tested for their ability to bind macromolecular targets. Subsequently, the identified hits, are extensively improved for their properties (i.e. affinity, potency, solubility) with the aim to develop a lead compound. Fragments typically have a molecular weight smaller than 300 Da, no more than three freely rotatable bonds, and no more than three H-bond donor/acceptor atoms. Compounds with these features are potentially able to bind several macromolecular targets albeit with low affinity. As a result, they need to be screened for their binding properties with a highly sensitive method.

X-ray crystallography is a suitable technique for FBDD thanks to recent technological advances such as sensitive detectors with high frequency readout, high brilliance monochromatic beams, robotics for sample handling and automated data collection pipelines. This enables to screen hundreds of crystals in a day, which is suitable for High-Throughput screening.

We were able to accomplish FBDD at ALBA, at BL13-XALOC, on three in-house targets. First, crystals were optimized focusing on their tolerance to DMSO using the crystal farm. Then, soaking experiments were accomplished on the DMSO-tolerant crystals with the aid of a mosquito (SPT Labtech) which allowed to dispense quickly and reliably minute volumes of fragment solutions (e.g. 100-250 nl). After incubation the soaked crystals were screened for their diffractive properties. The development of an in-house automated data collection (ADC) pipeline combined with the fast read-out detector PILATUS3 X 6M (and high-brilliance beam) allowed a fast collection of diffraction datasets at BL13-XALOC (12 crystals per hour). ADC is also a useful tool to optimize the human resources of ALBA or of the industrial/academic users. Identifications of binding fragment was accomplished by Pan-Dataset Density Analysis (PanDDA), which has been proven to be far more effective than conventional crystallographic methods for this task.

Recently a crystal shifter (Oxford Lab Technologies) was installed as equipment with the aim of automating crystal harvesting and annotation. In addition, we aim in the near future to install FragMAXapp, a piece of software that allow an easy management of fragment screening projects. We envisage that these improvements will streamline FBDD at ALBA and increase its throughput.

Combating Friedreich's Ataxia with Nanobodies: Structural Characterization of complexes with Frataxin. #130

Authors: *Alba Garay* Álvarez *(IQF Blas Cabrera-CSIC)*
Co-Authors: *Martín Aran, Juan Antonio Hermoso, Lorena Itatí Ibañez, Rafael Molina, María Florencia Pignataro, Javier Santos*

Friedreich's ataxia (FRDA) is an inherited neurodegenerative disease caused by mutations in the fxn gene, which encodes the protein frataxin. Frataxin plays a crucial role in mitochondrial functioning, and its deficiency in FRDA leads to iron accumulation, mitochondrial dysfunction and progressive neurological symptoms. However, tiny, persistent antibody fragments called nanoantibodies that are generated from camelids have shown great promise in the development of tailored treatments for a range of illnesses.

Our research aims to improve this process by using these tiny protein fragments called nanoantibodies that can interact with frataxin, specifically with the frataxin G130V variant involved in FRDA, this mutation affects the stability of the protein and results in low levels of mature protein. This suggests that the primary goal of the nanobodies would be to stabilize the mature protein and function as a chaperone so that it may attach to the cysteine desulfurase complex, which is in charge of the biogenesis of Fe-S clusters. To understand why these nanobodies bind so tightly to frataxin, we determined the three-dimensional structure of the nanobody-frataxin complex by X-ray crystallography. This technique allowed us to analyze the structures of complexes formed by nanobodies: 16C10, 6B1, 4A7, and 29F7. Additionally, we included previous predictions for these complexes by the AlphaFold3 tool, as well as the complexes formed by 5A8 and 28F6.

More importantly, X-ray crystallography helped us identify the exact binding site of the nanobodies on frataxin. By analyzing all the investigated complexes, we were able to categorize them into three binding types and associate these binding types with the affinity previously studied by our collaborators. This technique has also provided insights into how nanobody binding might affect the formation of the complex and the regulatory function of iron-sulfur cluster assembly.

SESSION B - In-situ and In-operando Processes

Femtosecond soft X-ray photoelectron spectroscopy on solids at the European XFEL #191

Author: *Manuel Izquierdo (European XFEL)*

X-ray Free electron lasers produce intense coherent femtosecond laser-like pulses in a large energy range, extending the capabilities of laser-based sources. The soft X-ray regime is of paramount importance since it allows resonant studies of the most abundant chemical elements, that serve as the base for materials with exciting scientific properties and very many prospective technological applications addressing relevant societal challenges. Photon-in/photon-out techniques at XFELs have been successfully implemented to study ultrafast dynamical processes in solids. In some cases, the entanglement between the relevant degrees of freedom in the material has been understood in a single experiment. Generalizing these observations to any class of material requires the use of multi-messenger techniques like photoelectron spectroscopy. The technique has been key to understand the properties of complex materials, surfaces and interfaces. It provides simultaneous understanding of the electronic, spin, chemical and structural properties. The measured spectra generally contain information on the excitation spectrum and the excitation probabilities thanks to the many-body nature of the photoemission process. Furthermore, the intrinsic time scale of the photoelectric effect enables the study of the fundamental material dynamics. The technique has been successfully implemented down to the pico-second time resolution at synchrotrons. Femtosecond experiments are routinely done using high harmonic generation up to around 100 eV. The extension of the technique to higher photon energies is only possible using superconducting acceleration technology that produces femtosecond pulses at MHz
repetition rates. In this contribution, the implementation of time resolved experiments above 1 keV at the SXP instrument will be presented.

Catalytic oxidation of CO by step and kink sites of curved Au crystal #158

Authors: *Anna Makarova (Helmholtz-Zentrum Berlin)*
Co-Authors: *Fernando Garcia Martinez, Jesús Redondo, Sabine V Auras, Elmar Kataev, Sabrina Gericke, Lisa Rämisch, Yuri Hasegawa, Ignacio José Villar García, Virginia Pérez Dieste, Edwin Lundgren, Jose Enrique Ortega, Frederik Schiller*

Curved crystalline surfaces allow to compare chemical reactions across a sequence of vicinal crystal planes with increasing density of steps, all exposed to the very same reacting conditions. This provides essential clues to understand catalytic processes, and, in particular, to reveal the role of low-coordination active sites. Within the present study, we have explored the formation of oxidic species across different crystallographic planes of the Au curved crystal and their further behaviour for CO oxidation reaction by means of UHV- and NAP-XPS. In order to oxidise gold we have followed three approaches: use of atomic oxygen, ozone and activating molecular oxygen by intense X-ray beam of the synchrotron. We have observed the formation of oxide at all the studied crystallographic planes, both at room and elevated temperatures, upon all applied approaches. Reduction of atomic oxygen chemisorbed on gold was revealed already at partial CO pressure of 6×10-9 mbar. Our analysis of photoemission intensities of O 1s species implies lower efficiency of the CO oxidation reaction at the flat (111) surface compared to the stepped surfaces.

In situ powder diffraction as a key for understanding materials behavior in non-ambient atmosphere #187

Author: *Alexander Missyul (ALBA Synchrotron)*

MSPD beamline at ALBA [1,2] provides access to the synchrotron X-ray powder diffraction experiments. High flexibility of the beamline is ensured by a broad photon energy range, 3 different detectors and large set of sample environments including setups for in situ and operando studies. Among others, capillary gas cell allows performing experiments under regulated gas flow or under predefined gas pressure. The cell can be also combined with temperature and humidity control setups to broaden the range of available conditions. The talk will include several demonstrative examples of using in situ diffraction data for understanding peculiarities of the materials behavior that cannot be directly observed ex situ.

Significant difference was observed in gas sorption capability of ZIF-8 metal-organic framework for nitrogen and alkanes. In the second case, amount of absorbed gas is almost 4 times less. Analysis of the diffraction data revealed existence of the strong non-covalent bonding between alkane molecules and ZIF-8 framework limiting the amount of absorbed gas to the interaction sites.

ZIF-65 is considered to be a promising material for CO2 storage. In situ diffraction studies showed that its crystal structure significantly changes depending on the CO2 pressure and temperature. Some of the changes are reversible, while another result in complete reconstruction of the framework and resulting structure remains unchanged even after CO2 is completely removed.

One of the important mechanisms causing degradation of the perovskite-based solid oxide fuel cells is interaction of the active material with CO2 yielding SrCO3. In situ studies of this process as a function of temperature, time and gas pressure opens a way to better understanding of this process and conditions required to ensure prolonged SOFC life.

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The role of anharmonicity on the thermoelectric properties of half-Heusler alloys #125

Authors: *Cristina Echevarria Bonet (Universidad de Oviedo)*
Co-Authors: *José Luis Garrido, Javier López-García, Óscar Juan Durá, Matthias Schrade, João Elias F. S. Rodrigues, Jesús A. Blanco*

In recent years, thermoelectric (TE) materials have garnered significant attention because they offer a versatile and promising solution for various energy-related challenges, contributing to advancements in energy efficiency, sustainability, and development of new technologies. The performance of TE materials is demonstrated by their unique ability to directly convert heat into electricity and vice versa [1]. This TE performance of materials is greatly influenced by the microstructures and fabrication methods. The spark plasma sintering (SPS) technique is particularly beneficial, providing rapid heating and electric current activation for efficient material densification and producing well-controlled microstructures that optimize the TE properties of the material [2]. Half-Heusler (HH) compounds are promising candidates for TE conversion, having an ABC cubic crystal structure that allows for various elemental combinations such as A=Ti,Zr,Hf; B=Co,Ni; and C=Sb,Sn, among others [3].
High-resolution synchrotron radiation X-ray powder diffraction was used to structurally characterize HH alloys with compositions MNiSn (where M=Ti,Zr,Hf or a mixture) and ZrNiSn1−xSbx. Alloys were synthesized by arc melting, thermal annealing, and a subsequent ball milling for 5, 10 or 15h. After that, powders were consolidated by SPS. Rietveld refinements of the room temperature patterns of the unmilled samples provided important crystallographic information consistent with existing data. For the milled alloys, particle sizes were reduced below 100 nm. The linear coefficient of thermal expansion (α) for all the investigated phases was less than 10−5 K−1. Additionally, the Debye temperature (θD) was estimated by analyzing the temperature dependence of the isotropic atomic displacement parameters in terms of a Debye model. For these HH alloys, a lower θD corresponds to a lower α. The strong agreement between the temperature dependence of the Debye temperature and the calculated values using the Thirring-Stern expansion highlights the significant role of anharmonicity: the lighter the compound, the lower the anharmonicity. Furthermore, we discuss the implications of anharmonicity on the thermal conductivity of these HH alloys [4].

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PLENARY Lecture III

Synthetic Control of Compositional Complexity in Reticular Frameworks #183

Author: *Carlos Martí-Gastaldo (Instituto de Ciencia Molecular, Universidad de Valencia)*

Reticular chemistry has boosted the design of thousands of metal and covalent organic frameworks for unlimited chemical compositions, structures, and sizable porosities. The ability to generate porous materials at will based on geometrical design concepts is responsible for the rapid growth of the field and the increasing number of applications derived. Despite their exceptional stability, the synthesis of targeted homo- and heterometallic titanium-organic frameworks amenable to these principles is relentlessly limited by the high reactivity of this metal in solution that impedes the controlled assembly of titanium molecular clusters. We will describe our recent results in the synthesis of new titanium organic frameworks by using high throughput methodologies. This approach permits producing porous crystals at high scale, that can help implementing new concepts towards energy conversion with molecular frameworks due to their unique combination of high surface area, crystallinity, photoactivity and tailorable catalytic activity.

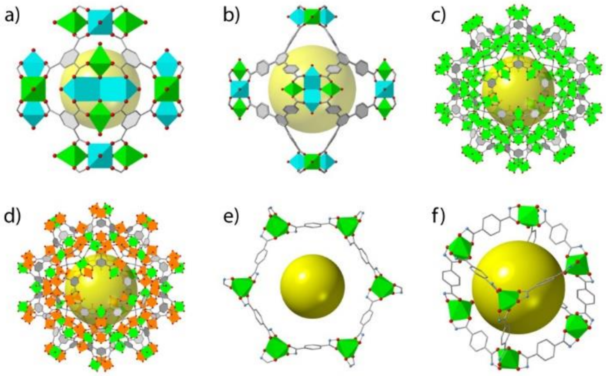

Figure 1. Structure of homo- and heterometallic Ti-MOFs: a) MUV-10, b) MUV-12, c) MIL-100(Ti), d) MUV-101(M), e) MUV-11 and f) cMUV-11. MUV stands for Materials of the Universidad de València.

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KEYNOTE Lecture I

BM25-SpLine at the ESRF: Status and perspectives #208

Author: *Juan Rubio-Zuazo (Spanish CRG beamline BM25-SpLine at the ESRF)*
Co-authors: *A. Garcia-Prieto, A. Gutierrez-León, M. Montero-Aranda, E. Sebastiani-Tofano*

The main goal of the interdisciplinary and multipurpose Spanish CRG BM25-SpLine beamline at the ESRF is to satisfy the needs of the use of synchrotron radiation in the region of hard X-rays of the Spanish scientific community, with a broad range of interests crossing very different research areas: physics, chemistry, materials science, biology, environmental sciences, and cultural heritage. The Spanish CRG BM25-SpLine beamline is dedicated to structural investigations using hard X-ray scattering mostly in materials science, specialized on the combination of diffraction and spectroscopy techniques.

With the advent of the new EBS-ESRF machine the beamline has been fully upgraded enabling state-of-the art experiments using Grazing incidence X-ray Diffraction, Hard X-ray Photoelectron Spectroscopy and X-ray Absorption Spectroscopy techniques. The increase of brilliance offered by the EBS-ESRF and the upgraded optics and experimental stations enable high energy and angular resolution measurements with low acquisition times, being especially suitable for in-situ and in-operando experiments.

In this talk, an overview of the present and future capabilities of the BM25-SpLine beamline will be presented.

AUSE PhD Thesis Award

Advanced Quantitative Synchrotron X-ray Imaging of Cement Micro- and Meso- Structures #162

Author: *Shiva Shirani (Universidad de Malaga)*

This research includes observations on hydration kinetics, microstructural changes and the effect of curing temperatures on cement properties. It also investigates the development of innovative methods for measuring cement hydration in situ, with the aim of improving the understanding and optimising the use of cementitious materials. The 4D (3D+time) tomographic study was reported with an astonishing spatial resolution of 250 nm and excellent contrast between the different components. The hydration of alite particles, the formation of C-S-H gel shells surrounding every alite grains and the passage of water through these shells were observed in the tomograms. Many interesting features have been identified in this 4D multi-modality X-ray imaging study, from laboratory micro tomography to synchrotron phase contrast and synchrotron ptychographic nanoimaging; such as spatial dissolution rate of anhydrous particles, etch-pits growth rate of alite particles, C-S-H gel shell growth rate, etc.

Inhomogeneity and disorder in ultrafast phase transitions #207

Author: *Daniel Perez Salinas (ALBA Synchrotron)*

In recent decades, there has been a growing interest in the emergent phenomena in strongly correlated materials. These systems are challenging to understand due to the complex interplay between various degrees of freedom, such as electronic, structural, and spin, which interact in non-trivial ways. A significant result of these interactions is the diversity within the phase diagrams, often featuring exotic states that are not yet fully understood. The study of phase transitions is then crucial for uncovering the complex microscopic mechanisms at play. Experimental techniques tracking these transitions have become increasingly precise, revealing how previously overlooked aspects like inhomogeneity and disorder play an important role.

This complexity is particularly relevant in ultrafast studies of correlated materials, a relatively young field exploring non-equilibrium situations. This thesis presents novel experimental techniques aimed at assessing disorder and inhomogeneity in non-equilibrium phase transitions while accurately tracking the dynamics of the involved degrees of freedom. These techniques are applied to two systems: La0.5Sr1.5MnO4, a prototypical layered manganite, and VO2, a well-known correlated material.

For La0.5Sr1.5MnO4, an all-optical tabletop pump-probe setup is introduced, allowing for precise tracking of the ultrafast melting of charge and orbital order. It is shown that the transition is incoherent, fitting the order-disorder paradigm, with spatial phase separation playing a significant role. For VO2, large-facility X-ray techniques are used to image phase inhomogeneity during the metal-to-insulator transition. These experiments reveal no evidence of previously suggested intermediate phases, such as the monoclinic metal VO2. Additionally, the thesis presents the first spatially-resolved observation of the ultrafast phase transition in VO2 with nanometer resolution, showing a global, prompt change in the domain pattern on the femtosecond timescale.

PLENARY Lecture IV

Investigation of Three Dimensional Magnetic Nanomaterials using Synchrotron Techniques #184

Author: *Amalio Fernández-Pacheco (Head of Physics of 3D Nanomaterials Institute of Applied Physics, TU Wien)*

The expansion of nanomagnetism to three dimensions provides exciting opportunities to explore new physical phenomena and opens great prospects to create 3D magnetic devices for green computing technologies [1].

In this talk, I will present some of our recent works dedicated to the investigation of three dimensional artificial magnetic materials. The talk will mainly focus on works using X-ray synchrotron techniques, which includes the study of 3D printed nanostructures with complex geometries [2-4], and multilayered heterostructures with spin chiral interactions [5-7].

Acknowledgements

I would like to thank all my group members and collaborators contributing to the work I will be presenting. This work is supported by the European Community under the Horizon 2020 Program, Contract No. 101001290 (3DNANOMAG).

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KEYNOTE Lecture II

Neutron scattering as a complementary tool for X-Ray Synchrotron Radiation techniques #185

Author: *Antonio Urbina* (Universidad Pública de Navarra (UPNA) / SETN)

Neutron scattering provides a tool to explore the structure and dynamics of materials that is highly complementary to X-ray Synchrotron Radiation scattering techniques. Neutron wavelengths at a length scale similar to atomic structure, spin detection, deuteration tools (to tune scattering cross section of hydrogen rich samples), a broad span of spectroscopic energies (time scales), provide a powerful tool that will be illustrated with examples of polymer sciences to demonstrate this complementarity [1,2]. The current availability of neutron sources will also be presented [3], as well as plans for its upgrading and for the construction of new compact sources [4], including information about Sociedad Española de Técnicas Neutrónicas (SETN) [4].

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Session A - Biotechnology and Biological Sciences

Advancements in Biological Sciences at the European Synchrotron #107

Author: *Montserrat Soler Lopez (ESRF)*

Biology's intricate complexity poses an ongoing challenge to understanding life's fundamental processes. Integrating molecular, cellular, and organ/systems biology is essential for a comprehensive grasp of the intricate relationship between structure and function. The ESRF stands at the forefront of pioneering unique biological technologies within an internationally collaborative, visitor-oriented environment, fostering an ideal setting for cutting-edge research.

Among the diverse research lines at the ESRF, a significant focus is on unravelling the pathogenesis of Alzheimer's disease, a debilitating neurodegenerative condition. By probing bioenergetic mechanisms within neuronal circuits of the brain, we aim to pinpoint potential root causes of the disease [1]. The facility's array of beamlines, dedicated to structural biology [2,3], X-ray imaging [4], and spectroscopy [5], enables detailed investigations into biological processes across various spatial and temporal scales.

This presentation will emphasize the pivotal role of interdisciplinary approaches in biological research, highlighting the remarkable advancements made possible by the state-of-the-art facilities and collaborative environment at the ESRF. By investigating the enigmas of life, at the ESRF we are not only expanding our understanding but also paving the way for potential breakthroughs in the diagnosis and treatment of complex diseases such as Alzheimer's.

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FaXToR: Fast X-ray TomogRaphy Beamline for ALBA #109

Author: *Alessandra Patera (ALBA Synchrotron)*
Co-authors: *Federico Cova, Llibert Ribó Mor, Victorien Bouffetier, Alberto Mittone, Josep Nicolàs Roman, Judith Juanhuix Gibert*

Synchrotron radiation-based X-ray tomographic microscopy is nowadays a powerful technique for non-destructive, high-resolution investigation of a broad range of materials. Taking advantage of the high-brilliance and coherence of the third-generation synchrotron radiation facility ALBA, the new µ-Computed Tomography (µCT) beamline (BL) called FaXToR will allow micrometer, quantitative, three-dimensional imaging with sub-second temporal resolution and extend the traditional absorption imaging technique to phase-sensitive measurements. With its advent, ALBA will fully develop a multi-scale, multi-technique approaches to investigate the morpho-structural changes in biological samples from the cellular up to the tissue level or even whole small organisms. Similarly, the upscale approach and the capabilities of the BL will be beneficial in a wide range of applications, from material science to cultural heritage to paleontology. FAXToR will provide the worldwide scientific community with a flexible design enabling morphological and dynamic studies at the micron scale in cm-sized volumes using a large variety of experimental setups. The BL gets photons from an in-vacuum 2.5T multipole wiggler with a critical energy of 14.8 keV. The main optical element is a Double Multilayer Monochromator (DMM) which covers an energy range between 8 and 50 keV with a bandwidth ΔE/E down to 1.5%, thus keeping minimal number of optical elements in order to guarantee the best beam quality in terms of stability and homogeneity. The BL will also provide filtered white beam with energies ranging from 10 to 70 keV and a maximum beam size of 35×12 mm2 (HxV) at the sample position, located 36.5 m from the source. At the end-station, simultaneous acquisition at two different spatial (within µm-scale) and temporal (from sub-seconds to minutes) resolutions will enable in-operando tomography while zooming-in at very small details inside the samples. In particular, time resolved µCT with a temporal resolution < 1 s (per scan) will be possible with state-of-the art CMOS fast detectors with a data streaming of 100 Gb/s. This high data throughput will be matched with on-the-fly 3D reconstruction and visualization pipelines, currently being developed. This work will describe the BL design and commissioning, from the optical elements to the end-station, and will give an overview of capabilities accomplishing the user community which make intense use of synchrotron-based µCT.

Synchrotron-based phase contrast tomography for cardiovascular applications: from rodents to humans #199

Author: *Hector Dejea (ESRF)*

Cardiovascular diseases (CVDs) affect the structure and function of the heart leading towards heart failure and are thus leading cause of death worldwide. The heart can be considered a hierarchical object, in which whole organ, tissue level and cell level structures integrate for optimal heart function. In that way, diseases altering each structural level will have an effect across all scales, so that multi-scale techniques are required for their investigation. Synchrotron-based phase contrast tomography has proven in recent years to be a very valuable technique to investigate the structural changes caused by CVDs. This research has been carried out mainly in ex-vivo rodent hearts with great success. This presentation will initially focus on rodent research, to then show how technological advancements and the advent of 4th generation synchrotrons have allowed to move towards dynamic imaging of beating hearts and even the possibility to scale up and scan whole adult human hearts.

Session B - Electronic and Magnetic Mater

Unveiling the magnetic secrets of cylindrical nanowires with X-ray microscopy #166

Author: *Lucas Perez (Dept. Fisica de Materiales Universidad Complutense de Madrid)*

In the past year, 3D magnetic systems and curvilinear nanostructures have emerged as promising alternatives for the development of novel spintronics applications. Among these, nanowires are some of the most studied curvilinear systems. In these systems, it is possible to stabilize complex, topologically protected domain walls, giving rise to novel effects such as the suppression of the speed limit for domain wall movement, known as Walker Breakdown. The study of these new effects, which may form the basis of novel spintronics devices, requires new approaches and techniques. In this talk, I will revisit our efforts in understanding the spin textures and magnetization dynamics of ferromagnetic nanowires using synchrotron-based x-ray microscopy techniques.

Spin polarized bulk bands with large energy splitting due to Dresselhaus effect in ZnTe(110) #151

Author: *Ji Dai (ALBA Synchrotron)*
Co-authors: *Andrés Santander-Syro, Massimo Tallarida, Ruben Weht*

ZnTe is a face-centred cubic p-type II-VI semiconductor with a direct gap of 2.26 eV [1] and a wide range of applications, such as green light-emitting diodes, THz generators/detectors, or substrates for CdTe self-assembled quantum dots [2-5]. Despite its importance in modern electronic applications, to this day there is surprisingly no high-resolution angle-resolved photoemission spectroscopy (ARPES) data allowing a complete understanding of its electronic structure.

Recently, using high-resolution ARPES and DFT calculations, we found that spin-orbit coupling (SOC) plays a crucial role in determining the band structure of ZnTe. More importantly, due to the lack of inversion symmetry in the ZnTe bulk unit cell, a Dresselhaus spin-splitting with a large spin polarisation comes into play. According to our calculations, this effect, never observed so far in this technologically important material, is larger along the ΓK direction in ZnTe's bulk electronic structure.

Taking advantage of LOREA, the ARPES end station integrated with a spin detector in ALBA, we manage to measure those bands along ΓK direction and characterize their spin texture in ZnTe(110). Large band splitting, up to 300 meV, has been observed. These splitting bands are indeed spin-polarized, as demonstrated by our spin-ARPES data.

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Structural Analysis by High-Resolution X-Ray Diffraction of Ferromagnetic Lu2Fe17 Melt-Spun Ribbons #132

Author: *José Luis Garrido Álvarez (Universidad Oviedo)*
Co-authors: *María Lourdes Areguín Hernández, Jesús A. Blanco, Cristina Echevarria Bonet, Pedro Gorria, Javier López-García, José Luis Sánchez Llamazares, Pablo Álvarez Alonso*

Binary R2Fe17 (R = rare earth) intermetallic alloys exhibit phenomena with great potential for applications, such as the magnetocaloric effect or magneto-volume anomalies. Among them, the Lu2Fe17 compound represents an interesting example because it crystallizes into a non-conventional Th2Ni17-type crystalline structure. In this structure, Lu atoms occupy Wyckoff crystallographic sites 2b (0, 0, 1/4) and 2d (1/3, 2/3, 3/4), while the Fe atoms occupy sites 4f (1/3, 2/3, z), 6g (1/2, 0, 0), 12j (x, y, 1/4), and 12k (x, 2x, z) [1]. However, only around 66 % of the 2b sites are occupied by Lu atoms, and some Fe atoms appear near these “unoccupied-Lu-positions” along the z-axis in 4e sites (0, 0, z) [2].

We have fabricated Lu2Fe17 melt-spun ribbons and performed a precise structural and magnetic characterization. Rietveld refinement of the high-resolution synchrotron X-ray diffraction patterns ($\lambda = 0.4428$ Å), measured at ALBA synchrotron (Spain), allowed us to confirm the crystalline structure and to extract detailed crystallographic information. One important observation is the anisotropic broadening of the diffraction peaks as a consequence of a grain growth that varies depending on the ribbon direction, which is typical in melt-spun samples [3]. Additionally, from neutron thermo-diffraction patterns collected at the Institut Laue-Langevin, we have observed an anomalous temperature dependence of the cell parameters and the cell volume, which is related to the magnetovolume effects characteristic of these alloys. The temperature derivative of magnetization for the melt-spun ribbons, dM/dT (inset in Fig. 1), reveals the existence of two magnetic transitions: first, from a paramagnetic to an antiferromagnetic (AFM) state at 276 K, and second, from AFM to a fan magnetic structure at 252 K [4]. Nevertheless, the melt-spun ribbons exhibit a high saturation magnetization MS = 131 A·m2·kg−1 at 2 K, which is compatible with the reported AFM-to-ferromagnetic transition induced by magnetic field.

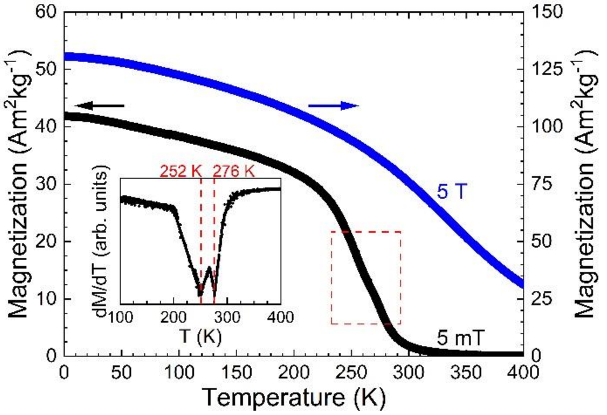

Figure 1. Thermomagnetic curves measured under static magnetic fields at 5 mT and 5 T for Lu2Fe17 melt-spun ribbons.

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Experimental observation of a magnetically induced warping transition in a topological insulator mediated by rare-earth surface dopants using angle-resolved photoemission spectroscopy #129

Authors: *Beatriz Muñiz Cano (IMDEA Nanociencia)*
Co-Authors: *Julio Camarero, Ji Dai, Yago Ferreiros, Adriana I. Figueroa, Kevin García-Díez, Francisco Guinea, Vera Marinova, Rodolfo Miranda, Aitor Mugarza, Pierre A. Pantaleón, Jose Angel Silva-Guillén, Massimo Tallarida, Miguel Angel Valbuena, Sergio O. Valenzuela*

Magnetic topological insulators are a novel class of materials where the topologically protected band structure coexists with long- range ferromagnetic order, which can lead to the breaking of time-reversal symmetry (TRS), introducing a bandgap in the Dirac cone-shaped topological surface state (TSS) [1-4]. In this work [5], we demonstrate novel spectroscopic evidence of TRS breaking at the TSS using angle-resolved photoemission spectroscopy, measured at the LOREA beamline at the ALBA Synchrotron. For this purpose low concentrations of magnetic rare-earth, namely Er and Dy, have been deposited on the ternary three dimensional prototypical topological insulator Bi2Se2Te. Specifically, the expected bandgap opening at the Dirac point is accompanied by a distortion in the TSS, evolving from its typical hexagonal warped shape to a trigonal one. Moreover, increasing Er coverage results in a tunable p-type doping of the TSS. As a consequence the Dirac point and therefore the magnetically induced bandgap can be both tuned towards the Fermi level as the RE concentration is increased, thus fulfilling the two necessary prerequisites for the realization of the quantum anomalous Hall effect (QAHE). The experimental results are rationalized by a theoretical model where a magnetic Zeeman out-of-plane term is introduced in the Hamiltonian governing the TSS band dispersion. Our results offer new strategies to control magnetic interactions with TSS based on a simple approach and open up viable routes for the realization of the QAHE and their implementation in new spintronic devices.

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Day 3, 4th September 2024

PLENARY Lecture V

ALBA and its future, ALBA II #195

Author: *Caterina Biscari (ALBA Synchrotron)*

Over the past decade, the ALBA Synchrotron has become an important pillar of the Spanish and European Research Area, providing research capabilities and a wide range of state-of-the-art instrumentation to a community of more than 8000 academic and industrial users. With its eleven operational experimental beamlines, while three more are being put into operation, and with the creation, in collaboration with other institutions, of an electron microscopy center, it is an essential tool to address society's most urgent challenges.

ALBA is preparing the leap towards the 4th generation, aiming at increasing its performance, reinforcing its role on the user community and on the industrial use of the instrumentation, as well as its educational vocation and training capacity. ALBA II, which is planned to be fully operative in 2032, will combine the upgrade to low-emittance storage ring with the construction of new fully-optimized beamlines, the refurbishment of part of its instrumentation, and the evolution of the data infrastructure.

The talk will highlight the performance of the facility and its strategy for the future.

SESSION - Science at ALBA

Photopharmacology: light and molecules for dynamic structural crystallography #143

Author: *Amadeu Llebaria (MCS, iQAC-CSIC)*

Azobenzene photoisomerization can be chemically implemented in protein ligands to actuate on biological receptors and to manipulate their activity with light. Azobenzene small molecule photoswitches can be designed and synthesized to serve for real-time regulation of receptors with high spatiotemporal accuracy using specific illumination patterns. The basis for this is a different interaction mode of the ligand isomers with the biological receptor. Therefore, light can be used for a precise control of physiology, on/off drug activation and targeting localized organs in free behaving animals. Strikingly, the photomolecular isomerization can also be employed in structural studies. Photoswitchable ligands co-crystallized with biomolecules can be used for triggering molecular actions in the crystal upon illumination. The bound ligand can be very fast photoisomerized, sharply generating a new state, which induces a receptor rearrangement that can be experimentally measured. This light switch in the crystallized receptor, which is reminiscent of some photon activated endogenous receptor systems, opens unprecedented possibilities to measure structural changes at atomic resolution and at very short-time scale. This approach will involve cooperative work of chemists, biologists, physicists and engineers, and may open a new perspective in dynamic studies of biological processes that can change our understanding of life and applied to invent radically new therapeutic approaches.

How ARPES can contribute to advanced materials research? #204

Author: *Arantzazu Mascaraque (UCM)*

Angle-resolved photoemission spectroscopy (ARPES) has emerged as a powerful technique in solid-state physics, providing direct observation of electronic structures with unprecedented energy- and momentum-resolved information. Since its technical development in the 1990s, ARPES has played a fundamental role in the study of electronic structures, bridging the gap between fundamental physics and practical applications. The insights gained from ARPES have had far-reaching implications, enabling technological advances such as bandgap engineering in semiconductor heterostructures for high-efficiency LEDs and laser diodes, or facilitating the design of improved catalysts for fuel cells and batteries, exemplifying its critical role in addressing today's energy challenges.

In this talk we will present the current methods and recent advancements in the experimental determination of electronic structure over the past decades. We will also highlight some examples of ARPES and its contributions to our understanding of relevant condensed matter systems, such as (i) the role of ARPES in advancing our knowledge of molecular hydrogen absorption, (ii) its insights into the effects of high entropy on the conductivity of oxide perovskites, and (iii) its impact on the design of new nickelate-based sensor devices. Finally, with an eye to the future, we will also consider possible advances in the field.

Crystal Chemistry of Important Polyanionic Materials used as Cathodes in Sodium-Ion Batteries: the decisive impact of Synchrotron X-Ray diffraction #203

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Polyanionic materials (phosphates in particular) are of special interest as positive electrodes for Li-Ion or Na-ion batteries since they offer competitive performances compared to sodiated or lithiated transition metal oxides [1,2]. They are based upon stable frameworks which provide long-term structural stability thanks to a unique variety of atomic arrangements.

The fluorinated vanado-phosphate $Na_3V_2(PO_4)2F_3$ possesses quite extraordinary features in terms of performances at very high rates and for extensive electrochemical cycling and is now developed by the company TIAMAT as their cathode material in commercialized batteries. Very early preliminary studies conducted in ALBA Synchrotron gave us the opportunity to determine its real crystal structure and to reveal subtle phase transformations upon Na+ extraction [3-4].

The NASICON structural family, on the other hand, with its large panel of compositions, $Na_xMM'(PO_4)_3$ ($0 < x < 4$; M,M' = Ti, Fe, V, Cr, Mn) is among the most widely investigated due to its 3-D framework structure which generates high Na+ mobility [1]. Among them $Na_3V_2(PO_4)_3$, $Na_4MnV(PO_4)_3$ and $Na_4FeV(PO_4)3$ are of particular interest [5-10]. We will present several new structures that we determined, from pristine powders or for intermediate compositions spotted by operando synchrotron XRD.

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Two-dimensional Layered Hydroxides for Energy Applications #206

Author: *Gonzalo Abellan (ICMol, UV)*

The alkaline oxygen evolution reaction (OER) is crucial for green hydrogen production using water electrolysis. However, its industrial implementation at high current densities is restricted by the limitations of current commercial electrocatalysts with respect to scalability, overpotential, and stability. One of the most promising alternative electrocatalysts are those based on layered hydroxides (LH) due to their outstanding electrochemical properties. In this talk, we will show the latest advances carried out in the 2D-Chem research group (www.icmol.es/2dchem) in the synthesis and characterization of novel two-dimensional (2D) LH based on abundant transition metals. Among others, we have developed an industrially scalable room-temperature, atmospheric pressure homogeneous alkalinization synthetic pathway to achieve an optimized NiFe layered double hydroxide (NiFe-LDH). By using the nucleophilic attack of chloride over an epoxide ring, a low-dimensional, highly defective NiFe-LDH with pronounced cation-clustering can be obtained, exhibiting excellent electrochemical performance. Spectroscopic studies including XANES, EXAFS and SAXS, together with ab-initio calculations revealed the important role of Fe-clustering in minimizing the energy pathway for the electrochemical performance of the catalyst. Moreover, we will show how in-situ XAS and PXRD experiments can be also used to optimize the synthesis of these layered materials, allowing a new use as precursors of metallic nanocomposites of interest in energy storage.

In-situ X-Ray Diffraction revealing the reaction mechanism of ultrafast Transient Liquid Assisted Growth of superconducting thin films #201

Author: *Elzbieta Pach (ICMAB-CSIC)*
Co-authors: *Diana Franco, Emma Ghiara, Cristian Mocuta, Ona Mola, Xavier Obradors, Cornelia Pop, Teresa Puig, Lavinia Saltarelli, Daniel Sanchez, Carla Torres, Roxana Vlad*

Cuprates, REBa2Cu3O7-δ (REBCO, RE = Y or rare earth) are the most important class of High Temperature Superconducting (HTS) materials, due to their outstanding properties. They are the superconductors with the highest operational temperature and highest operational magnetic field, hence, nowadays the materials of choice for Coated Conductor (CC) technology in most of the high current applications. The synthesis of REBCO-type superconducting films is compatible with standard chemical solution deposition methods (CSD) and pulsed laser deposition growth (PLD). However, recently, the development of a novel synthesis approach promises the reduction of the cost/performance ratio in the CC field. The, so called "Transient Liquid Assisted Growth" (TLAG) method [1-3] is a high-throughput, ultra-fast, non-equilibrium, kinetic process of growth of REBCO type superconducting films and coated conductors compatible with industrial applications. The understanding of the TLAG process requires application of advanced tools and techniques for its characterization and optimization. Therefore, development of a specialized instrumentation for the characterization and optimization of TLAG process in real time and in real conditions (operando) by synchrotron radiation based in-situ X-Ray Diffraction, was achieved. This new instrumentation allows to follow the crystalline precursors of reaction, the generation of the intermediate phases and the dynamic growth of the superconducting phase while acquiring the resistance of the sample in real time, by in-situ XRD. Epitaxial REBCO TLAG growth at 1000 nm/s has been reached in superconducting films of 3 MA/cm2 at 77 K. Furthermore, TLAG growth method was shown to be compatible with introduction of inorganic nanoparticles as vortex pinning centres to increase the performance of such films. In this presentation, I will mainly report on the present understanding of the TLAG process based on the results gathered from the in-situ XRD-MS-R experiments.

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SESSION - ALBA Beamlines Upgrade Program

XAIRA: Advancing macromolecular crystallography with low background diffraction and micron-sized beam #134

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The new microfocus beamline XAIRA at ALBA will host the first macromolecular crystallography (MX) experiments in fall 2024 with the aim to offer an optimal solution for some major factors limiting the quality of MX data, namely the crystal size, the radiation damage and the background of diffraction patterns.

Micron-sized crystals will be optimally illuminated by the 3×1 µm² beam at focal position. The beam spot will be enlarged and tailored to match crystal dimensions by either defocusing the optics or moving the sample along the beam axis. Radiation damage will be minimized in rotation experiments using helical collection strategies enabled by a custom-made sub-100nm run-out goniometer, as well as in serial experiments, using a fixed target SSX setup compatible with several commonly used standards and custom supports.

Nevertheless, the landmark feature of XAIRA aims to be the exceptionally low background in diffraction images, achieved by enclosing the entire end-station in He atmosphere, which includes the sample environment, diffractometer, cryostream and detector, while remaining compatible with automated sample mounting, standard cryocrystallography sample formats and operation in air.

The high flux, above 1013 ph/s at 12keV, and the EIGER2 X 9M HE fast detector will enable fast oscillation experiments (~1 s), raster and helical scans. X-ray-based raster scans and AI algorithms are being developed to locate the micron-sized crystals.

The two MX beamlines at ALBA, XAIRA and XALOC, will share resources to tailor project needs through a joint proposal submission system. A new Dewar shipment system and a new data portal to provide a single access point, automated processing and a catalog identification for all data acquired will be in service soon to improve user experience.

Lensless coherent imaging of nanoscale magnetic domains in 2D van-der-Waals materials #136

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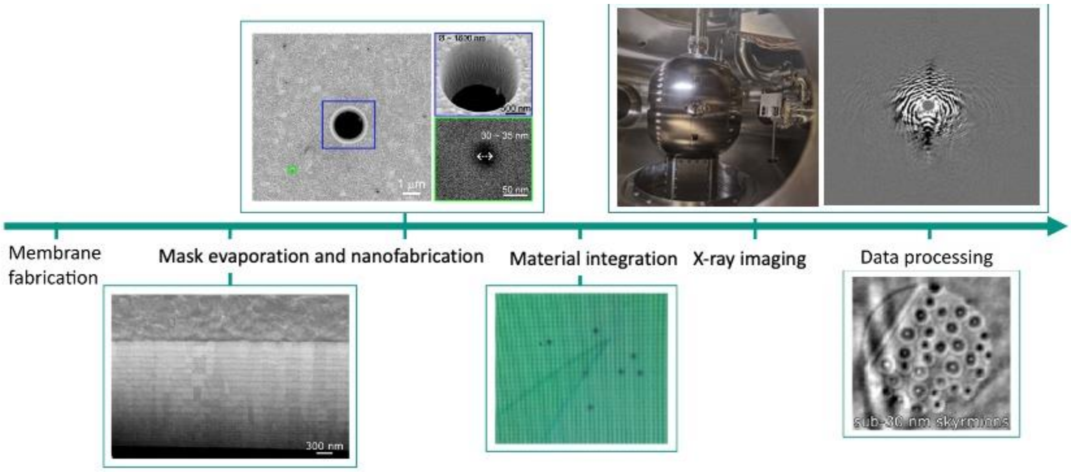
Here we report lensless coherent soft x-ray nanoscale imaging [1] of magnetic domains in 2D van- der-Waals materials (vdW) [2] at low temperatures and under large applied magnetic fields. With the integration onto clean-room nano-fabricated holography masks of micron-sized flakes of 2D materials via (i) deterministic transfer inside an inert-air glove box or (ii) focused ion beam prepared lamella, we extend the application of x-ray holography to this novel class of materials. The developed methodology, summarized in Figure 1, is of general applicability and also streamlines the investigation of air sensitive materials. We will showcase its use in the investigation of the intricate magnetic domains and non-collinear configurations occurring in various 2D vdW materials under variable temperatures down to 20K and high applied magnetic fields up to 2 Tesla. Besides Fourier transform holography [1], subsequent holography-assisted phase retrieval in coherent diffraction imaging [3] has been also applied. Our findings demonstrate that lensless soft X-ray coherent imaging techniques exploiting either circular, linear dichroism contrast or element specific spectroscopic fingerprints, provide distinctive opportunities to unravel the emerging magnetic and electronic properties of 2D magnetic materials and related devices at the nanoscale.



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Updates from the CHEMAT Section, The Beamline Upgrade Program #205

Authors: *Carlo Marini (ALBA), François Fauth (ALBA)*

The Chemistry and Material Science Section (CHEMAT) of ALBA represents MSPD, CLAESS, NOTOS, NCD-SWEET, and CIRCE-NAPP beamlines, i.e. a synergetic mix of scattering and spectroscopic techniques, well exploited and supported by the user community. CHEMAT focuses on bulk and surface science, particularly in operando and in-situ conditions. The three scientific examples, here presented, provide a panoramic view of the current capabilities and future potential of CHEMAT in the field of catalysis (electronic and structural characterization of carbon-doped Ruthenium oxide catalyst) and in energy related materials (beam-induced effects in Li-ion cathodes materials; perovskite solar cell investigation after surface passivation), demonstrating CHEMAT's ability to tackle complex problems through multimodal and coordinated efforts, leveraging advanced analytical instruments and methodologies. The upgrade program of CHEMAT has been shaped on extensive user input since 2021, by means of workshops, meetings, and the section review, which have allowed to define requirements and goals. The section in ALBA-II is undergoing significant upgrades (classified into three categories: necessary, essential, and enabling), aimed to enhance beamline capabilities, improve instrumentation (or refurbish old existing ones), and support advanced research in chemistry and material science. The mission of CHEMAT in In Alba-II will remain the same: to providing robust, highly productive instruments and synergies with microscope platforms for the user community. CHEMAT will continue to focus on energy materials, catalysis, polymers, cement, cultural heritage, and strongly correlated materials, while also offering industrial services. The synergies and complementarities between the different scattering and spectroscopic beamlines will be kept and enforced by the increase of spatial resolution and radiation coherence, as a result of the new accelerator machine. New laboratories and facilities will support sample preparation, data processing, and analysis, fostering collaborations with academic and industrial partners.

SESSION A - Materials Science

Building a Materials Acceleration Platform (MAP) for battery materials research: Automated module for the synthesis of inorganic compounds #142

Author: *Iciar Monterrubio*
Co-authors: *Montse Casas Cabanas, Marine Reynaud, Amaia Saracibar, Javier Carrasco*

The global demand for sustainable rechargeable batteries is expected to increase drastically in the next decade as applications continue to take off. Li-ion batteries (LIBs) are the most powerful of the high-capacity rechargeable batteries on the market today, but they have almost reached their energy density limit and, therefore, exploration of new systems and chemistries is mandatory.

Taking up this challenge requires new disruptive approaches and better tools to accelerate the discovery and understanding of new electroactive materials. Several strategies have been proposed by different groups to theoretically identify new potential candidates more efficiently (Jain et al. 2011, Rousse et al. 2013, Katcho et al. 2019), but the experimental validation stage often reveals to be the limiting step of the new material discovery process (Carey et al. 2011, Adhikari et al. 2020, Benavad et al. 2021).

In this work, a novel automated high-throughput experimental module for the preparation of lab-scale samples of electrode materials will be presented. This module is designed to handle and mix solutions of precursors in appropriate stoichiometries that will be evaporated before a solid-state annealing treatment. This approach enables to build a set-up compatible with a wide range of synthesis routes such as sol-gel, Pechini, co-precipitation-based syntheses, etc, which in turn enable for size and morphological control and/or in situ carbon coating of the particles to enhance the ionic conductivity. This automated high-throughput module for inorganic synthesis has been employed to explore high-voltage spinel materials, which will be thoroughly discussed in this talk and that have been characterized at ALBA synchrotron.

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Near ambient pressure oxidation of silver in the presence of steps: electrophilic oxygen and sulphur impurities #124

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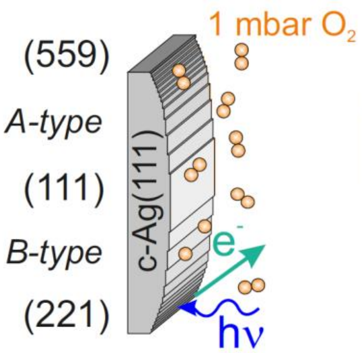
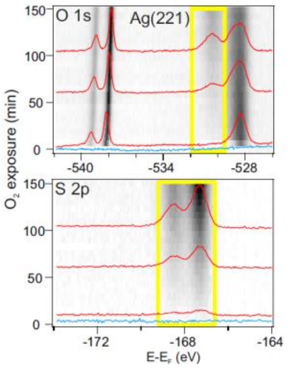
Co-Authors: *Khadiza Ali, Anna Makarova, Sabine V Auras, Fernando Garcia Martinez, Alaa Mohammed Idris Bakhit, Rodrigo Castrillo Bodero, Ignacio José Villar García, Jose Enrique Ortega, Virginia Pérez Dieste*

The oxidation of Ag crystal surfaces has recently triggered strong controversies around the presence of sulphur impurities that may catalyze reactions, such as the alkene epoxidations, especially the ethylene epoxidation. A fundamental challenge to achieve a clear understanding is the variety of procedures and setups involved, as well as the particular history of each sample. Especially, for the often used photoemission technique, product detection or photoemission peak position overlap are problematic. Here we investigate the oxidation of the Ag(111) surface and its vicinal crystal planes simultaneously, using a curved crystal sample and operando X-ray photoelectron spectroscopy at 1 mbar O2 near-ambient pressure conditions to further investigate surface species. The curved geometry allows a straightforward comparative analysis of the surface oxidation kinetics at different crystal facets, such as to precisely correlate the evolution of different oxygen species, namely nucleophilic and electrophilic oxygen, and the build up of sulphur as a function of the crystal orientation. We observe that emission from both surface and bulk oxide contribute to the characteristic nucleophilic oxygen core-level peak, which arises first and rapidly saturates below temperatures of 180 ∘C. The electrophilic oxygen peak appears later, growing at a slower but constant rate, at the expenses of the surface oxide. Electrophilic oxygen and sulphur core-levels evolve in parallel in all crystal facets, although faster and stronger at vicinal surfaces featuring B-type steps. Our study confirms the intimate connection of the electrophilic species with the formation of SO4, and point to a higher catalytic activity of B-type stepped silver surfaces for alkene epoxidation or methane to formaldehyde conversion.





4D nanoimaging of Portland cement hydration” #105

Author: *Miguel Ángel García Aranda (Universidad de Malaga)*
Co-authors: *Emmanuel Brun, Ana Cuesta, Ana Diaz, Mirko Holler, Bratislav Lukic, Alexander Rack, Shiva Shirani, Pavel Trtik*

Portland cements are environmentally contentious, accounting for ≈8% of the anthropogenic CO2 emissions. Understanding the cement hydration reactions (dissolution and precipitation processes) is important to contribute to developing cements with lower CO2 footprints.

We are pushing 4D (3D+time) cement hydration nanoimaging within a multiscale framework. Full-field laboratory X-ray micro Computer Tomography (microCT) is widely used to study cement hydration. Still, the best spatial resolution is about 2 microns for a Field of View (FoV) of ≈1×2 mm (H×V) with polychromatic measurements taking hours. Moreover, the contrast between the different components is poor. Full-field propagation-based phase-contrast synchrotron X-ray microCT can study similar FoVs ≈1×2 mm with better spatial resolution, ≈0.50 microns. The monochromatic measurements are fast, i.e. 5-10 minutes. Unfortunately, the contrast is only slightly better. Cement hydration can be studied with much better contrast and spatial resolution by scanning near-field ptychographic nano-computed tomography (nCT). In this case the FoV could be ≈200×30 microns with spatial resolution, close to 250 nm, and excellent component contrast. Even air and water can be differentiated. However, these nCTs takes about 3-4 hours in optimized beamlines (BL) at third generation synchrotrons.

We will present here our latest 4D nanoimaging results (Shirani et al. (2023) Nature Comm. 14:2652) from data taken at cSAXS beamline of SLS-PSI for a commercial Portland cement 52.5R with a water-to-cement mass ratio of 0.40. In the period 4-19h, the alite spatial dissolution rate, for particles smaller than 3 microns, has been measured as ~100 nm/h. In the same period, alite particles with average sizes larger than 10 microns dissolved with a spatial dissolution rate of ~33 nm/h, i.e. three times more slowly. In the period 19-47h, the alite spatial dissolution rate, for particles larger than 10 microns, is ~25 nm/h. Moreover, in the same 19-47 h time interval, the etch-pit growth rate, for particles larger than 10 microns, was ~40 nm/h. These values highlight the importance of particle size for determining the kinetics of cement hydration processes.

Moreover, in the period 4-19h, the C-S-H gel growth rate has been estimated as ~33 nm/h. C-S-H gel shell densifies with time and its average electron density evolves from 0.47 e-Å-3 at 19 h to 0.53 e-Å-3 at 47 h. Some other details will be discussed.

The barocaloric effect in the FeII(5NO2-pap)2 spin-crossover complex via low-pressure synchrotron powder X-ray diffraction #133

Author: *Gloria Subias-Peruga (INMA-CSIC, Universidad de Zaragoza)*
Co-authors: *Javier Blasco, Marie-Laure Boillot, Emmanouil Charkiolakis, Vera Cuartero, Marco Evangelisti, Talal Mallah, Catalin Alexandru Popescu, David Gracia Alcalde*

Fe(II) complexes with spin-crossover (SCO) transitions are known for their giant and tunable barocaloric responses, which are of interest for the development of more efficient and environmentally friendly cooling technologies [1]. The barocaloric effect (BCE) is associated with a low-spin (LS, S=0) to high-spin (HS, S=2) transition for Fe(II) (3d6) upon heating, which involves significant lattice volume changes that can be tailored with pressure [2-4]. Here, we address the barocaloric potential of FeII(5NO2-pap)2, whose SCO transition takes place near room temperature at ambient pressure in ref. [5]. We present synchrotron powder X-ray diffraction (PXRD) measurements performed at the MSPD beamline at ALBA synchrotron, using the low-pressure-range diamond anvil cell at 300, 310, 320 and 330 K, to quantify the isothermal volume changes at the SCO transition on increasing (P) and releasing (PR) pressure (0 – 6 kbar). At ambient pressure, all PXRD patterns agree with a monoclinic C2/c cell as described in [5]. At T = 300 K, the LS phase is stable with pressure and we observe the contraction of the lattice volume. For T ≥ 310 K, a volume collapse takes place at the HS-LS transition as the pressure increases, the average volume change being ΔV = 76 ± 2 Å3. These findings are supported by our heat capacity (HC) experiments under pressure (Fig. 1, left panel). The transition temperature spans from 308 K at ambient pressure to 350 K at 2 kbar on heating/decompression, being accompanied by significant thermal hysteresis, which increases under applied pressure. The remarkable and reversible BCE of FeII(5NO2-pap)2 is characterized by an isothermal entropy change of 75 J kg−1 K−1 and an adiabatic temperature change of ~ 26 K (Fig. 1, right panel).

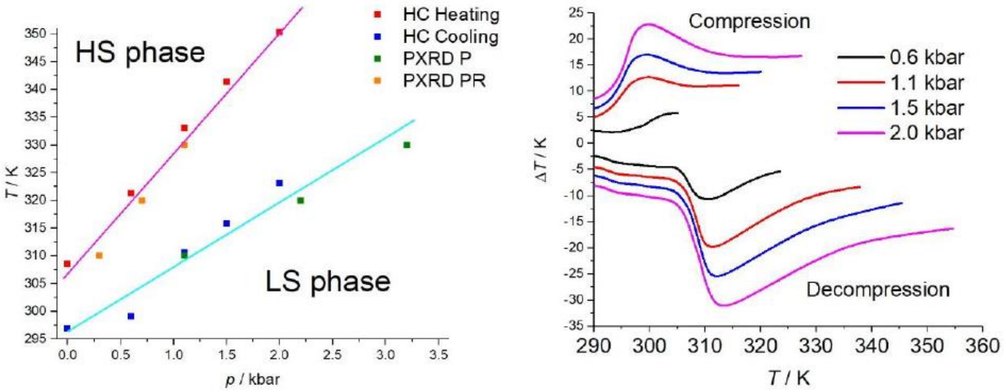


Figure 1. Left: Temperature-pressure phase diagram from PXRD and HC measurements. Right: Adiabatic temperature change ΔT, driven by pressure

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SESSION B – Life Sciences

Multiscale connectomics for neuroevolution: X-Ray tomography and FIBSEM #135

Author: *Marc Corrales (MRC-LMB (Laboratory of molecular biology) Cambridge, UK)*

The structure of a neural circuit tightly constrains its function.

Obtaining synaptic level connectomes (i.e the complete set of neurons constituting a circuit and its connections in the form of synapses) is the first step towards understanding their physical implementation. Like any other biological entity, nervous circuits evolved by natural selection, i.e a process of random modification of preexisting structures that become predominant if they confer a reproductive advantage to the individual. Since the modifications have to be compatible with the viability of the organism, "innovations" are constrained and the end product (e.g. brains like ours) result from the accumulation of gradual changes and module repurposing. The comparison of homologous circuits from animals along the tree of life is therefore key to interpret and understand the principles of nervous system organization.

Homologous circuits in closely related species teach us how complexity emerges by addition and combination of preexisting circuit motifs while analogous circuits in distant species show different implementations to solve similar problems, like perception or motor control.

Taking advantage of the small size of the pygmy squid *Idiosepius* hallami we acquired a whole animal (0.5*0.5*2mm) X-Ray tomography volume at 125 nm/px, allowing us to follow nerve bundles between all the brain lobes and organs. This allows us to then use our modified FIBSEM to acquire targeted synaptic resolution volumes that can be cross-registered into the whole squid volume.

As our first proof of concept, we have imaged a volume of the eye and optic lobe at 8*8*30 nm/px containing the photo-receptors and the first layers of visual processing of the animal and are currently reconstructing the circuits at synaptic resolution. Combining the 2 scales afforded by each of the techniques we can start to move beyond the insect model organisms such as Drosophila and C. elegans while retaining the ability to work at synapse resolution, key for understanding the structure function relationship of nervous systems: the basic requirement in biological neural network research.

Macromolecular and structural changes in glioblastoma cell line after carbon dots and riluzole treatments: Synchrotron-based FTIR and X-ray Microscopy approach #198

Author: *Tanja Ducic (ALBA Synchrotron)*
Co-authors: *Manuel Algarra, Milena Ninkovic, Elena González-Muñoz*

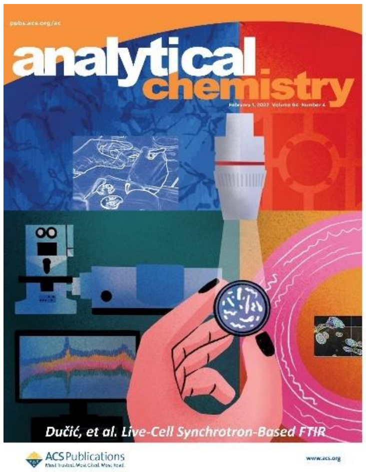
Glioblastoma (GBM) is characterised by short median survival and an almost 100% tumor-related mortality, whose mechanisms are not yet fully understood. Differences in elemental concentrations, and organic, cytoskeleton cell morphology, by using X-ray fluorescence and full-field soft X-ray microscopy were observed. The bio-macromolecular changes explored by live cells SR-FTIR spectro-microscopy focus on the biochemical profiling of live GBM cells and provide new insight into tumor heterogenicity [1].

Aditionally, we tested riluzole treatment, a medicament with potential anticancer properties. In addition, we synthesized and characterized carbon-based nanoparticles of 2-acrylamido-2-methylpropanesulfonic acid (AMPS-CDs), which showed biocompatibility with different cells and tested them as a carrier for the drug riluzole (AMPS-CDs@riluzole). The AMPS-CDs have been extensively characterized by techniques to obtain the exact composition of their surface by using FTIR, XPS, and NMR spectroscopy, and cryo-transmission electron microscopy. The surface of the AMPS-CDs is covered mainly by sulfonated, carboxylic, and substituted amide groups, which make them suitable as a carrier for drug delivery.

The AMPS-CDs@riluzole system was used and the cell viability was observed in cancer and non-cancerous cells. Although the AMPS-CDs nanoparticles themselves were not toxic, when combined with riluzole, they had a positive impact on promoting cell death on GBM.

The effect of CDs as carriers for the riluzole system on glioblastoma cells was studied using live-cell SR-FTIR micro-spectroscopy to monitor biochemical changes of single cells in situ. After applying AMPS-CDs@riluzole, we have observed changes in all bio-macromolecules, including the nucleic acids and protein conformation in particular, α-helix and β-sheet conformation and lipids metabolism in GBM cells [1, 2].

Combining all imaging techniques, including different synchrotron-based microscopies, allows us deeper structural and molecular insight into modification on the cellular and organelles level. The study indicates that AMPS-CDs could be a useful nano-carrier for delivering riluzole and targeting proteins in glioblastoma cancer cells, without the same effect on the non-cancerous cells [3].



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Day 4, 5th September 2024

PLENARY Lecture VI

Design of processable polymeric carbon nitride materials for catalytic applications #159

Authors: *Santiago Garcia-Granda (University of Oviedo)*
Co-Authors: *Christiane Adler, Radim Beranek, Igor Krivtsov, Dariusz Mitoraj, Ashish Vazirani*

Polymeric carbon nitride (PCN) is a unique material in the polymer world; It is low-cost, easy to prepare and possesses outstanding thermal and chemical stability. Most importantly, however, it shows an ability to efficiently drive photocatalytic oxidation and reduction reactions under UV-blue light irradiation, which made it one of the best candidates for the role of photo(electro)catalyst for solar-fuels production. Although the material itself is not a new discovery, it has been known since times of Liebig, its photocatalytic properties were only discovered little more than a decade ago[1]. Since then, the properties of this material have been thoroughly scrutinized and a number of synthetic approaches for the preparation of PCNs with tuneable characteristics was developed. This, however, has not allowed overcoming a poor processability of bulk PCN impeding its use in advanced catalytic processes and in solar-fuel producing systems like photoelectrochemical (PEC) cells. A recent discovery of ionic PCN also known as poly(heptazine imide) (PHI) followed by the developed in our group method for water-soluble PHI synthesis[2] has made a huge step in that direction. Water-soluble PHI, apart from its high photocatalytic activity in alcohol photoxidation, also proved itself as an efficient stabilizer for ultra-small metallic nanoparticles. On the other hand, the sol-gel processing of the PHI nanoparticles enabled the first robust PCN-type photoanodes for solar alcohols reforming even under bias-free conditions[3]. It also made possible obtaining an unprecedented insight in photogenerated charge dynamics in PHI demonstrating that light-induced accumulation of electrons leads to its photodoping, resulting in a significant improvement of PEC performance [4].

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As a preamble to the talk, the plans for the renewal of the International Union of Crystallography (IUCr), which on reaching its 75th anniversary has redefined its Vision/Mission and Values as a common home for researchers in Structural Science, will be presented.

We gratefully acknowledge the financial support provided by Spain´s Agencia Estatal de Investigación, PID2020-113558RB-C41.

SESSION A - Magnetism

Searching for monolayer flakes at LOREA #152

Authors: *Massimo Tallarida (ALBA Synchrotron)*
Co-Authors: *Ji Dai, Jordi Prat Albert, Pierluigi Gargiani, Iolanda Di Bernardo*

LOREA beamline at ALBA is characterised by high energy resolution and large energy range, which makes the ARPES beamline highly competitive. Another important property of LOREA is the beam spot size on the sample, which is about 20µmx15µm (HxV) and allows to measure very small samples, including small flakes, which can be optically observed with a microscope pointing onto the sample. However, for monolayer flakes, which are optically not active, a different strategy is needed. In this case it is possible to scan the sample position in a reproducible way to detect features which are related to the monolayer. This method was employed to detect monolayer MoSe2 flakes, produced at ALBA by mechanical exfoliation of a MoSe2 bulk crystal onto a Au/mica substrate in Ar atmosphere. After the exfoliation in the glove box, the sample was moved to LOREA in a UHV suitcase to avoid air contamination. We present here the full characterisation of the monolayer MoSe2 with ARPES, and core level spectroscopy, showing the capability of studying this class of materials at LOREA.

The transition of magnetic textures from quasi 2D to 3D microstructures #146

Author: *Alicia Estela Herguedas Alonso (Universidad de Oviedo)*
Co-authors: *Salvador Ferrer Fábregas, Aurelio Hierro Rodriguez, José Ignacio Martín Carbajo, Eva Pereiro López, Carlos Quirós, Andrea Sorrentino, María Vélez*

In Nanomagnetism, thin films and heterostructures are frequently used in magnetic memories, where the magnetization is confined in two dimensions [1]. While non-trivial magnetic textures, such as vortices, can appear in thin films [2], the drive for new functionalities and the capability to grow three dimensional magnetic nanostructures necessitate exploring how these textures change in these materials when the thickness exceeds the exchange length [3].

We have studied the transition of magnetic configurations from 2D to 3D of a 150 nm Py (Permalloy, Fe20Ni80)/30 nm Cu/50 nm Py rectangular microstructure by increasing the thickness of its simulated geometry starting from 40 nm Py using Mumax3. This simulation allows to visualize the behavior of the magnetization while growing the sample's thickness, mimicking the process of sputtering. Simulations start with a cross-tie magnetic configuration, which was found to be one of the possible initial states of an experimental sample with similar dimensions. This cross-tie pattern is characterized by a series of vortex, antivortex and vortex distributed along the length of the structure. As the thickness is increased, the perpendicular magnetic anisotropy begins to manifest, and a stripe domain pattern appears. This pattern resembles the shape of an eight, where the stripes are concentric around the initial vortices. During this growth process, the system increases the period of the stripes, expelling them at the borders. When the structure is fully grown, the upper Py layer of the magnetic system relaxes into a vortex surrounded by stripes, which can be analogous to a target skyrmion [4].

Finally, we have studied the final state of the simulation by performing magnetic tomography of a sample grown with magnetron sputtering similar to the simulated heterostructure. Magnetic images were acquired at the Fe L2 edge (719.9 eV) using the Transmission X-ray Microscope available at MISTRAL beamline in ALBA [5]. The reconstruction of the magnetization was computed with the algorithm designed in [6]. This configuration exhibits the stripe domain pattern observed in the simulated system, suggesting that its initial state may correspond to the initial cross-tie of the simulation evolving into a more complex configuration in the 3D regime.

Engineering of organolanthanide sandwich complexes by on-surface synthesis #113

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Co-Authors: *Shanmugasibi Mathialagan, Maria Tenorio, Lenka Černa, Daniel Moreno, Beatriz Muñiz-Cano, Cristina Navío, Manuel Valvidares, Miguel Valbuena, José Urgel, Pierluigi Gargiani, Rodolfo Miranda, Julio Camarero, José Martínez, José Gallego, David Écija*

Lanthanide-based organometallic chemistry is an expanding research area with high potential for applications in fields as efficient catalysis and molecular magnetism [1,2]. Organolanthanide complexes are promissing candidates for single molecule magnets since they can present properties as large magnetic anisotropy, slow relaxation times and high blocking temperatures [3,4]. Usually these materials are synthesized in solution following wet chemistry protocols. We report an alternative approach to engineer lanthanide-based organometallic sandwiches by on-surface synthesis. This approach allows the achievement of a 2D regular array of single molecular magnets with long range ordering.

A new family of lanthanide sandwiches is introduced, based on partially deprotonated hexahydroxybenzene molecules and Dy or Er metallic atoms [5]. The sandwich structure of these complexes is ideal for the design of molecular magnets on surfaces since it isolates the metallic atom and reduces its interaction with the surface. The organometallic sandwiches were investigated by scanning probe microscopy and spectroscopy, X-ray photoelectron spectroscopy, X-ray absorption spectroscopy, X-ray linear and X-ray magnetic circular dichroism. The experimental results were complemented by thermochemical atomistic simulations based on density functional theory calculations. Our results show that the Dy and Er sandwiches assembly in hexagonal-packed islands and present similar electronic structures. On the other side, they display different magnetic properties, with the Er sandwiches presenting isotropic magnetization while the Dy sandwiches have in-plane magnetic anisotropy.

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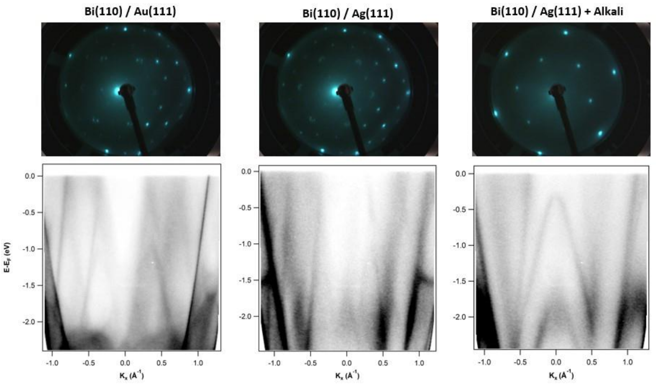
Electronic properties of Bismuth monolayers grown on metallic substrates #153

Author: *Kevin Garcia Diez* (ICN2 / ALBA Synchrotron)
Co-authors: *Ji Dai, Ji Dai, Jorge Lobo Checa, Aitor Mugarza, Jose Enrique Ortega, Jose Enrique Ortega, Massimo Tallarida, Massimo Tallarida, Aitor Mugarza*

Bismuth is the most metallic element of group V, with strong spin-orbit-coupling (SOC) and high carrier mobility, especially at its surfaces, making it unique. In the last years, thin films have attracted a lot of interest due to the relation between the atomic structure and their topological properties [1,2]. Bismuth is an interesting case of topological material, with predicted unconventional high order topology [1], and several film orientations exhibiting topological 1D states at the edges. At the monolayer limit, two different Bismuthene allotropes have been grown (α and β), both showing topological edge states [2,3].

We grew Bi monolayers on Au(111) and Ag(111) substrates and studied their atomic structure by Scanning Tunneling Microscopy (STM) and Low Energy Electron Diffraction (LEED), and their electronic structure by Angle Resolved Photoemission Spectroscopy (ARPES). We found that the growth of Bi on both substrates is very similar, with the formation of a (110)-like atomic structure modulated by a 1D Moiré pattern imposed by the registry between substrate and Bi lattices. The investigation with ARPES suggests a strong coupling between the monolayers and the substrate, that hides the observation of the electronic bands of Bi monolayers. In order to decouple the Bi layer from the metallic substrate we used alkali elements



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SESSION B - Multi-modal Studies

Multi-scale and multimodal imaging combining NanoSIMS and Synchrotron techniques #114

Author: *Iris Henríquez Valido* (UAB-IPREM)
Co-authors: *Maria Angels Subirana, Alessandra Monteiro, Etienne Gontier, Maria Maares, Claudia Keil, Martin Senz, Patrick Ollivier, Dirk Schaumlöffel*

Multi-scale and multimodal imaging techniques have transformed the study of environmental and biological samples, providing unprecedented insights into their structure, composition, and functionality. This work combines NanoSIMS with synchrotron radiation techniques (µFTIR, µXRF, and µXAS) applied to three different samples, yielding key information in three research areas:

1) Studies on trace element supplementation of laying hens have shown improved shell and bone quality, but their uptake remains uncertain. NanoSIMS and µFTIR analyses of eggshells correlate the spatial distribution of the supplemented metal with the distribution of organic matter, providing a better understanding of metal adsorption metabolism and eggshell quality assessment.

2) Zinc deficiency is a global public health concern. Zinc-enriched yeast (ZnYeast) is a promising biological source with Zn-integrated structures. Safety evaluations by EFSA require comprehensive data on bioavailability and clarification of its Zn species. NanoSIMS examined subcellular Zn distribution in Saccharomyces strains, while µXAS evaluated the speciation, which is crucial for understanding Zn homeostasis and future legal evaluations of ZnYeast as an organic zinc supplement.

3) Sargassum algae influx onto Caribbean beaches poses ecological and economic threats, with tons of algae washing ashore annually, containing pollutants like arsenic. Proper disposal is crucial to prevent arsenic leaching during degradation. NanoSIMS revealed arsenic localization in algae cells, while µXRF and µXAS determined arsenic speciation, providing vital insights for Sargassum algae treatment strategies.

The study showcases the effectiveness of combining NanoSIMS with synchrotron radiation techniques (µFTIR, µXRF, and µXAS) for analyzing environmental and biological samples, highlighting the significant potential of these imaging techniques in solving complex research problems.

XPS, XAFS, XRD and FTIR Operando Studies of a Vanadium based (H2V3O8) Lithium-ion Battery at ALBA beamlines. #126

Author: *Ignacio José Villar García (CEU San Pablo University)*
Co-authors: *Virginia Pérez Dieste, François Fauth, Carlos Escudero, Ibraheem Yousef, Vlad Martin-Diaconescu, Angelica Duarte Cárdenas, Chandrasekar Mayandi Subramaniyam, Alois Kuhn, Flaviano Garcia Alvarado, Jose Angel Martínez González*

The performance of a battery relies on the electrodes' ability to reversibly store charge quickly and for extended periods. In metal-ion batteries, this involves multiple processes that influence the efficiency of charge transfer and the intercalation of metal ions at the electrode's surface and within its bulk. Critical processes, such as the chemical and structural modifications of the electrode and the formation of stable solid electrolyte interfaces (SEI) on the anode and cathode electrochemical interfaces (CEI), are highly dependent on the properties of both the electrode and electrolyte materials and how they interact. Given the significant changes that battery components undergo during operation, there is widespread agreement that more in-situ and operando studies are necessary to improve our understanding of these processes and to enhance future battery systems rationally.

In light of this, we are focusing significant efforts on developing a comprehensive operando approach utilizing a selected range of synchrotron-based techniques: XPS, FTIR, XRD, and XAFS to study electrode materials under working conditions. In this presentation, we will present the latest results obtained at the MSPD, NOTOS, CLAESS, CIRCE, and MIRAS beamlines at the ALBA synchrotron on the H2V3O8 cathode material. H2V3O8, a layered vanadium oxide hydrate, has been investigated by our group for lithium-ion batteries, achieving stable capacities of approximately 300 mAh g-1 for the developed nanobelt structured electrodes [1]. The use of operando techniques has enabled us to conduct a comprehensive study of the key chemical, structural, and electronic changes at both the bulk and the surface of this electrode during the discharge and charge stages of the battery cycle.

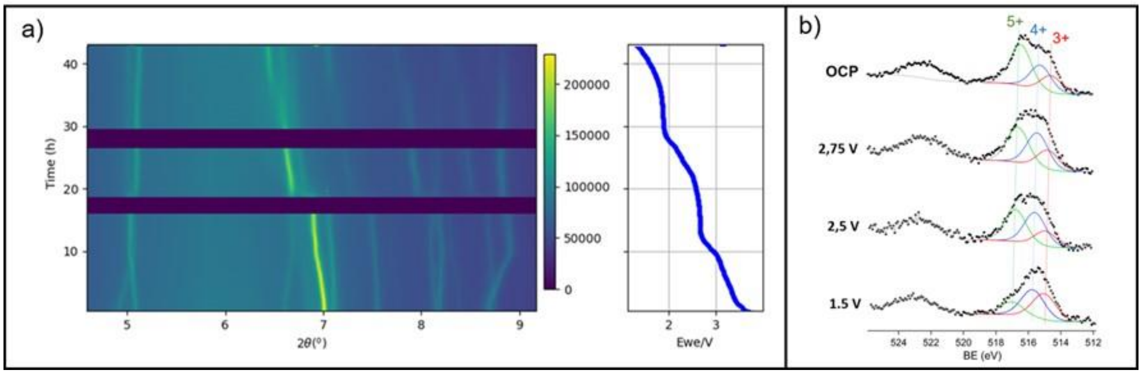

Figure 1. a) Contour plot of X-ray diffraction patterns collected operando during electrochemical cycling of H2V3O8 in a lithium cell (MSPD) and b) operando XPS data of the V2p spectrum at different voltages showing the evolution of V3+, V4+ and V5+ states at the V2p3/2 at different potentials during discharge (CIRCE).

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Ni and Fe Doped Graphene Aerogels Prepared by Freeze-Casting: Investigating the Effect of Fe through In Situ Synchrotron Characterization #117

Authors: *María González Ingelmo (Instituto de Ciencia y Tecnología del Carbono (INCAR-CSIC))*
Co-authors: *Pablo Rodríguez Lagar, Jonathan Ruiz Esquius, Freddy E. Oropeza, Ibraheem Yousef, Miriam López García, Patricia Álvarez, Clara Blanco, Ricardo Santamaría, Victoria G. Garcia Rocha*

Transition metal oxide-based catalysts represent a promising alternative to noble metals for oxygen evolution reaction (OER). Notably, Ni and Fe layered double hydroxides are considered benchmark catalysts for this reaction in alkaline media. The good synergy between these two metals is also observed as an enhancement in the activity of Ni-based catalysts when are tested in an electrolyte containing Fe impurities. Therefore, studying the interactions between Ni and Fe is crucial to understanding and improve the catalyst design.

In this work, graphene aerogels doped with Ni and Ni-Fe based nanoparticles were prepared. To do so, an aqueous solution of graphene oxide was mixed with metal precursors and processed by freeze-casting. After a subsequent reduction (650 °C, 1h, N2), hierarchical structure materials of partially reduced graphene oxide (rGO) doped with 10 wt.% loading of highly dispersed metal nanoparticles were obtained. Additionally, the Ni-Fe ratio in the precursor was modulated to obtain aerogels without Fe (rGO-Ni) and with 2, 10, and 25% Fe relative to the total metal content.

The OER activity of these samples was studied in 1M KOH Fe-free and containing 10 µM of Fe. It was observed that the presence of Fe positively affects the catalytic performance after an activation process (CP 10 mAcm-2 12h), particularly for samples with lower intrinsic Fe content. Therefore, rGO-Ni experiences the greatest improvement in activity (10 times the initial activity) when cycled in the presence of Fe, representing a remarkable enhancement compared to other reported results, due to the low concentration and good dispersion of the nanoparticles in the graphene network.

These characteristics make these materials suitable for in situ characterization studies, which could provide valuable insights into the Fe incorporation during the OER. Firstly, operando infrared spectroscopy has been used with the aim to identify and monitor the formation of intermediate species involved in the catalytic mechanism, offering a deeper understanding of the reaction pathway. This also presents a significant challenge as it requires the design and fabrication of an electrochemical flow cell compatible with the beamline requirements, including a ZnSe window close to the sample and at an appropriate focal distance for the microscope. Secondly, X-ray absorption spectroscopy has been employed to characterize the oxidation state of the metal species and its coordination environment.

Impact of Co-doping on Pd/Al2O3 Catalysts for Methane Oxidation: A Characterization Study #118

Author: *Laia Pascua Solé (Universitat Politècnica de Catalunya)*
Co-authors: *Ilaria Lucentini, Xin Xin Hu, Alba Garzon Manjon, Edgar Eduardo Villalobos Portillo, Carlos Escudero, Carlo Marini, Núria J. Divins, Jordi Llorca*

Pd on alumina is regarded as one of the most active catalysts for methane oxidation. Nonetheless, its tendency to sinter and deactivate in the presence of water poses a challenge in stabilizing Pd nanoparticles from forming hydroxides. The synergy between Pd and Co_3O_4 as a support has been investigated, suggesting that cobalt oxide may preserve palladium as an oxide and prevent hydroxide formation. This study explores the interaction of cobalt in a Pd/Al_2O_3 system using a novel synthesis technique based on a mechanochemical milling of the precursors with the support. Conventionally, cobalt forms a spinel structure with alumina, $CoAl_2O_4$; however, this physical milling approach may inhibit its complete formation.

In this study, two catalysts were synthesized using the mechanochemical technique: (i) Pd monometallic on Al_2O_3 (Pd/Al_2O_3) and (ii) Pd and Co on Al_2O_3 ($PdCo/Al_2O_3$). Two tests were conducted on both samples: (i) four consecutive cycles from room temperature to 900 °C and back under CH_4 and O_2, and (ii) one cycle under the same conditions followed by a 24-hour step at 450 °C with water. $PdCo/Al_2O_3$ was characterized using operando XAS and XRD in BL-16 NOTOS in ALBA synchrotron and TEM from JEMCA, also, in ALBA synchrotron and ICN2.

The tests conducted under a dry atmosphere reveal a slight hindrance in activity for the Co-doped catalyst; however, under a wet atmosphere, $PdCo/Al_2O_3$ exhibits a slight improvement in activity compared to Pd/Al_2O_3.

XAS and XRD experiments under dry conditions indicate that Pd in $PdCo/Al_2O_3$ is not easily reduced, while for Pd/Al_2O_3, the reduction from PdO to Pd metallic is complete. Additionally, Co is predominantly transformed into $CoAl_2O_4$. TEM images reveal PdO particles around 20 nm, consistent with XRD measurements. These characteristics may account for the reduced activity of the Co-doped catalyst under dry conditions. Nonetheless, the enhanced activity under wet conditions is noteworthy. XAS measurements could suggest the possible presence of hydroxide in Pd/Al_2O_3, whereas $PdCo/Al_2O_3$ primarily remains as PdO.

PLENARY Lecture VII

Using LCLS to study ultrafast structural and electronic changes in bio and condensed phase chemistry systems #172

Author: *Roberto Alonso-Mori (SLAC National Accelerator Laboratory)*

The Linac Coherent Light Source (LCLS), the world's first hard X-ray Free Electron Laser, provides unprecedented opportunities to study ultrafast processes, down to the femtosecond timescale, in real-time across a diverse range of scientific disciplines. This presentation will highlight the transformative impact of LCLS on the fields of condensed-phase chemistry and biochemistry. I will present the current status of the LCLS, and its LCLS-II high repetition rate upgrade. The capabilities of XCS, one of the 8 LCLS instruments, now include a versatile platform that supports pump-probe condensed-phase chemistry experiments in the liquid phase. The MFX instrument is dedicated to bio and biochemistry experiments in air. Both instruments offer multimodal approaches, where hard X-ray spectroscopy techniques are used in conjunction with scattering methods (e.g X-ray diffraction and X-ray solution scattering), making it possible to observe chemical and biochemical reactions and associated electronic and geometric changes simultaneously and in real-time with fs time resolution. These methods together with recent and upcoming advances in hard X-ray spectroscopy and instrumentation optimized for the time structure and high flux of the LCLS will be presented.

Case studies will demonstrate the application of these instruments in capturing femtosecond to picosecond dynamics, providing insights into chemical reactions. Finally, I'll also introduce future spectroscopic developments projected for the near and mid future of LCLS including new instrumentation and opportunities in the soft and tender X-ray range offered by the LCLS-II upgrade with an emphasis on the scientific opportunities enabled by these developments.

SESSION -XFEL

Capturing ultrafast molecular motions and lattice dynamic response in a spin crossover thin film using femtosecond electron diffraction and X-ray diffraction #178

Author: *Yifeng Jiang (European XFEL)*
Co-authors: *Fernando Ardana Lamas, Carsten Deiter, Tobias Eklund, Katarzyna Jarzembska, Radosław Kamiński, Maciej Lorenc, Christopher Milne, Gábor Molnár, Henrike Müller-Werkmeister, Fengfeng Qi, Karl Ridier, Robin Schubert, Doriana Vinci, Dao Xiang, Peter Zalden, Renske van der Veen*

Solid-state materials based on photoinduced spin crossover (SCO) complexes provide a particularly attractive platform due to their functional design and tuning versatility in the crystal structure. SCO refers to a reversible phase transition between a low-spin state and a high-spin state that can be triggered by changes in pressure, temperature, and magnetic field or by photoexcitation. Dynamical processes induced by an ultrafast laser pulse in SCO materials are intrinsically multi-scale in time and space. Among the SCO systems, a novel SCO materials $Fe(HB(tz)_3)_2)$ (HBTZ) nanocrystalline thin film shows attractive features: it has an extremely abrupt first-order, isostructural spin transition above the room temperature with an high resilience upon repeated photoswitching. HBTZ can be deposited on different substrates to prepare smooth, dense, highly oriented nanocrystalline films with the orthorhombic c-axis normal to the surface. These unique properties of HBTZ open a promising perspective for fast response photo-switches at room temperature.

In this contribution, we will discuss the MeV-UED and the femtosecond XRD results as complementary tool to the MeV-UED for having a better understanding of the molecular dynamics in the HBTZ thin film. Our team has performed MeV-UED and ultrafast XRD experiments on HBTZ nanofilms to investigate the photoinduced SCO dynamics with ~100 fs temporal resolution. Due to the epitaxial orientation of the thin film with the c-axis perpendicular to the surface and the high electron energy (MeV), only Bragg peaks of the hk0 family have been detected. This renders it impossible to study the lattice volume expansion along the c-axis, which requires Bragg peaks hkl ($l \neq 0$). We further carried out the time-resolved XRD experiments at the FXE end-station of the European XFEL with the same experimental geometry. Thanks to the high q-resolution in low q-range of X-rays, we unambiguously show that this local structural rearrangement occurs within a constant unit-cell volume of the ground state on the ultrafast timescale. The molecular switching and the lattice heating are responsible for the lattice volume changes, but their contributions are different in multiple timescales after photoexcitation.

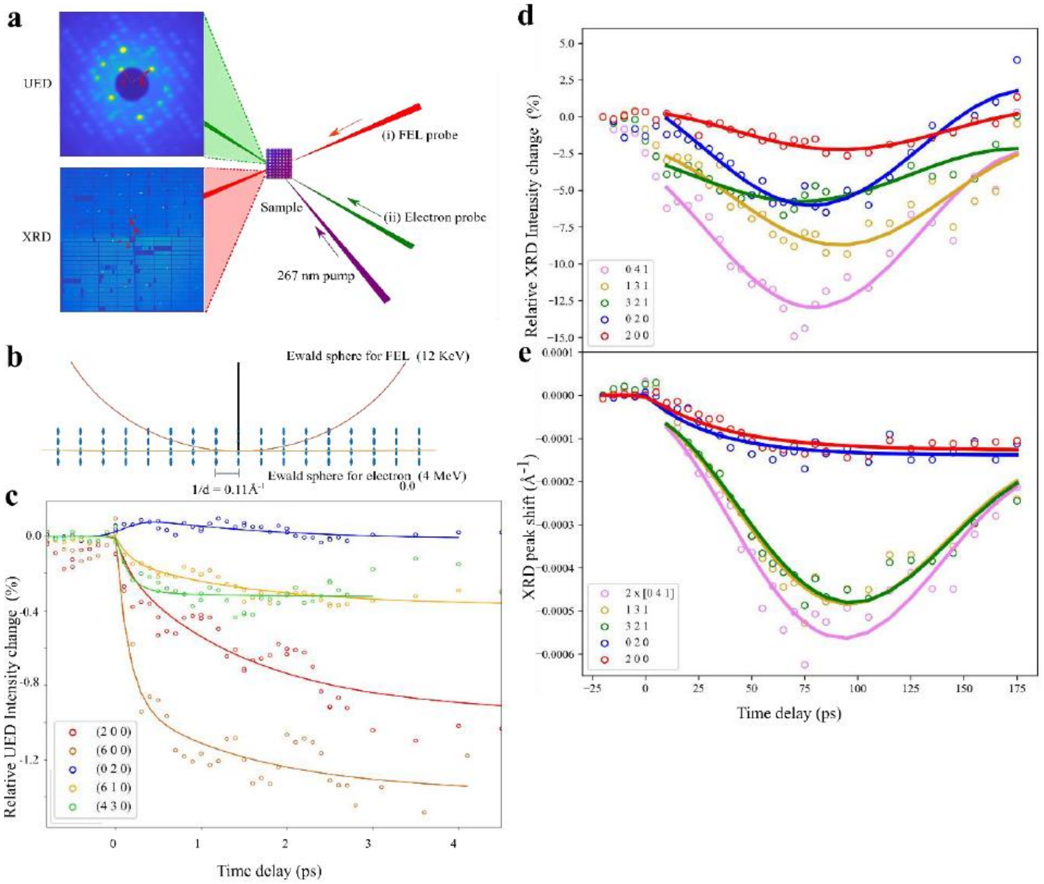

Figure 1. a) Scheme of the UED and XRD experiment. b) Ewald sphere of FEL (12 KeV) and MeV electrons (4 MeV). c) Time-resolved change of the UED experiment. d) Time-resolved XRD intensity change. e) Time-resolved XRD peak shift

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Investigation on electronic structure and reactivity in heterometallic proteins using femtosecond X-ray absorption spectroscopy #189

Author: *Rebeca Gomez Castillo (Max Planck Institute CEC)*

Enzymes with bimetallic carboxylate cofactors catalyze numerous essential reactions and are commonly found to be homometallic. However, the existence of bimetallic cofactors with heterometal active sites was recently established. RNR-like enzyme R2lox2 enzyme contains an FeMn heterometallic cofactor is capable of O2 activation while also demonstrating an unusually efficient photodecarboxylation upon irradiation, a rare photoactivity for natural enzymes outside of photosynthesis. These observations prompt questions about how the MnFe influence the enzyme's reactivity. Particularly, what is the identity of the metal in the MLCT that triggers the photodecarboxylation and which are the initial steps involved in this mechanism? These questions have been addressed by Fe and Mn femtosecond X-ray absorption spectroscopy (XAS) performed on R2lox enzyme, allowing to track the electronic and structural changes associated with photochemical decarboxylation.

Ultrafast soft X-ray investigations of solution phase molecular photochemistry and photophysics at EuXFEL #160

Author: *Benjamin Van Kuiken (European XFEL)*

The EuXFEL routinely provides ~4,000 X-ray pulses/s for time-resolved experiments at the soft X-ray SASE3 branch. Here we outline the possibilities for solution phase soft X-ray spectroscopy at the Spectroscopy and Coherent Scattering (SCS) instrument, which has a dedicated endstation for experiments requiring liquid jet sample delivery. This setup is integrated with either round nozzles (Ø = 20 – 50 µm) for scattering geometries or flat jets (1-4 µm thickness) for transmission-mode measurements. The liquid jet sample delivery has been commissioned together with two standard spectroscopic techniques: resonant inelastic X-ray Scattering (RIXS) and X-ray absorption spectroscopy (XAS). RIXS measurements are carried out using the hRIXS spectrometer which currently enables experiments up to 10,000 resolving power.1 XAS measurements are implemented within the BOZ-XAS scheme where a diffractive optic is used to generate both signal and reference beams, which are both detected on the same shot-resolved detector.2 Early experiments have monitored intersystem crossing and charge transfer dynamics and demonstrate the possibility of experiments on dilute (~10 mM) samples.

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Ultrafast biological imaging at the European XFEL, without crystals #161

Author: *Filipe Maia (Uppsala University)*

The dream of imaging single molecules was instrumental to the construction of X-ray free-electron lasers (XFELs). The extremely bright and short pulses provided by XFELs make it possible to collect the diffraction pattern of a particle before its destruction (Neutze et al. 2000) which was successfully proved at FLASH more than a decade ago (Chapman et al. 2006). Since then, the method of flash X-ray imaging (FXI) has been used to image live cells (van der Schot et al. 2015), cell organelles (Hantke et al. 2014) and in particular the giant Mimivirus in both two dimensions (Seibert et al. 2011) and three dimensions (Ekeberg et al. 2015). The inauguration of the European XFEL marked the beginning of the high-intensity, high-repetition-rate and high data-rate era of XFELs, and it has been shown that FXI can take full advantage of those rates (Sobolev et al. 2020). Yet, FXI has not yet fulfilled its promise of high-resolution sub-nanometer imaging. In this talk, I will present our latest results from single-particle imaging experiments at the SQS beamline at the European XFEL. I will discuss the future of single-particle imaging, how we are tantalizingly close to our goals and how several new technologies can help us get there. I will also present our results from time-resolved solution scattering experiments and how ultrathin liquid sheets show great promise.

Expanding drop on fixed target approaches using droplet addition #170

Author: *Jos J. A. G. Kamps (Diamond Light Source)*
Co-authors: *Pierre Aller, Philip Hinchliffe, Allen M. Orville, Robin L. Owen, Tiankun Zhou*

Over the past decade, serial femtosecond crystallography (SFX) and serial synchrotron crystallography (SSX), have emerged as powerful techniques for investigating time resolved and dynamic crystallography under ambient conditions. To facilitate these experiments, a variety of sample delivery methods have been developed. Among these, fixed target approaches, where crystals are distributed over a grid of cavities at predictable locations on a solid support, have gained adoption at numerous light sources due to their relatively efficient sample consumption.

The concept of fixed target sample delivery has been combined with various pieces of equipment, such as lasers for light activated systems, to enable time resolved crystallography. More recently, work by Mehrabi et al. demonstrated the feasibility of liquid addition on fixed target systems. This innovative approach has been adapted for use on the microfocus beamline I24 at Diamond Light Source.

This talk will provide insights into the challenges associated with this approach and emphasise the importance for the full integration of rigorous controls when conducting drop on fixed target experiments.

The long path to extend transient grating spectroscopy into the X-rays #177

Author: *Cristian Svetina (Instituto Madrileño de Estudios Avanzados en Nanociencia - IMDEA)*

Nonlinear wave-mixing based on table-top laser sources, have provided a major advance in experimental and theoretical physics, chemistry and biology [1]. A special case of wave-mixing is the Transient Grating (TG) spectroscopy which employs two crossed laser beams interacting at the sample generating an interference pattern. A third time-delayed probe beam is used to monitor the time evolution of the induced dynamics that transiently changes the sample's index of refraction. TG is routinely used to gain information on transport and diffusion processes and quasi-particle dyamics [2]. Optical wavelengths inherently limit the reachable spatial and temporal resolutions while extension to higher photon energies - i.e. extreme ultraviolet and X-rays - allows to potentially reach nanometer and atto-femtosecond scales adding element and chemical specificity being able to access to K, L and M resonances [3]. In the last years a large international community has formed to study extension of TG in the X-rays [4, 5]. In the talk I will describe and advances and status of the Long Term Proposal "Dynamics of nanoscale phenomena in solids and liquids studied with X-ray Transient Gratings at European XFEL" [6] showing the latest results and look at the future activities. I will conclude introducing the COST Action NEXT for the development and application of nonlinear wave-mixing methods in the extreme ultraviolet and X-rays [7].

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Imaging femtosecond laser-induced lattice distortions inside crystalline semiconductors by Ultrafast Dynamical Diffraction #115

Author: *Angel Rodriguez Fernandez (European XFEL)*
Co-Author: *Jan Siegel*

Material processing with femtosecond lasers has attracted enormous interest over the past decades due to the countless potential applications in technology and industry. One key aspect of their performance is the reduced thermal load, enabling the fabrication of smaller and sharper feature sizes, as well as enabling surface and sub-surface processing of transparent materials [1,2]. This advantage can be implemented by using laser pulses that are shorter than the time it takes for the strongly excited electron subsystem to transfer its energy to the lattice (typically a few ps). Such pulses can generate highly non-equilibrium states and trigger structural changes within a few hundred fs[3]. The processes involved are almost instantaneous non-thermal melting of a surface layer of several tens of nm [3,4], followed by the inward propagation of a sharp melt-front, as well as shock wave propagation into the material.

Time-resolved X-ray diffraction has been successfully used to study non-thermal melting in semiconductors thin films (e.g. [4]). But, most “real-world” processing applications deal with bulk materials. Therefore, the development of new x-ray tools that will enable to measure fast and ultrafast laser-induced structural changes as a function of depth inside bulk materials is highly desired.

We have developed a novel technique for imaging ultrafast deformations along the depth in thick crystalline Si wafers, based in the dynamical diffraction process observable in quasi perfect crystals[5]. To this end, we have recently performed a fs laser pump (800nm) - fs X-ray probe (9 keV) experiment at the Materials Imaging and Dynamics instrument of the European XFEL facility, exploiting the extraordinary flux, high spatial coherence and ultrashort duration of the generated X-rays pulses. During the experiment, Si and Ge wafers with different thicknesses (up to 300 um) were investigated as a function of pump laser fluence and delay time. First results clearly demonstrate the high sensitivity of the technique to reveal the dynamics of laser-induced processes over a wide temporal window (from tens of fs up to hundreds of ns) even at fluencies below the modification threshold.

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Day 5, 6th September 2024

Workshop on CoDI

Coherent Diffraction Imaging Beamline at ALBA II

Leveraging x-ray coherence to probe the complexity of materials under realistic conditions #108

Author: *Irene Calvo (University of Zaragoza)*

The advent of the world's first coherent hard X-ray sources, i.e. 4th generation synchrotrons, in France (ESRF), the USA (APS), and very soon Spain (ALBA-II) represents an unprecedented opportunity for the spatio-temporal visualization of nanoscopic systems evolving under realistic conditions.

In this talk, I will start with a general overview of the new capabilities offered by 4th generation synchrotrons. These new radiation sources generate a highly brilliant beam characterized by a coherence flux that is between 100 to 1000 times greater than that produced by 3rd generation X-ray sources.
In the context of these new experimental facilities, I will introduce the different techniques based on coherent X-ray (CX) scattering, emphasizing their suitability to investigate the complexity of materials, i.e., the coupling between dynamics, structure, and specific material properties (e.g., mechanical, optical, electronic, chemical, etc.) at the nanoscale. This strength is due to the great versatility of hard CX, which combines highly penetrating X-rays (with energies > 8 keV) with the exquisite sensitivity of CX diffraction, particularly in its fine-scale features called speckles, to 3D structural features like defects, crystalline distortions, etc., of nanoscale objects. These properties form the basis of a new class of microscopy approaches uniquely adapted to in situ and real-time studies on the response of all sorts of materials to a wide variety of reactive environments.

The power of these new microscopy methods to address materials complexity under realistic conditions will be illustrated by the different talks in the workshop, through multiple examples: e.g., the investigations on cement hydration, studies in Earth and Planetary Sciences, the imaging of the internal structure of quantum materials, the in-situ studies of photo-electrocatalysis or the coupling between structure and performance in optoelectronics materials. I will contribute with the example of the study on the chemical reactivity of mineral interfaces in contact with liquid solutions (i.e. crystallization and dissolution in liquid medium).

To conclude, I will outline a roadmap of the challenges these new CX microscopy methods face in realizing their great potential.

Bragg Coherent Diffraction Imaging: from simple particles to complex crystalline materials #164

Author: *Virginie Chamard (University of Marseille)*

Coherent diffraction imaging in the Bragg geometry has been demonstrated as a powerful method to investigate the crystalline properties of material in 3D with a high spatial resolution (10-50 nm) and a high sensitivity to lattice distortion (from a few 0.0001 to a few 0.01). This phase retrieval method makes use of coherent diffraction intensity patterns to retrieve detailed maps of the sample scattering function, overcoming the limits induced by the lack of efficient focusing x-ray optics.

Since its advent in the early 2000s [Pfe06], Bragg coherent diffraction imaging (BCDI), which exploits the finite size of the crystalline particle, has filled the gap between direct microscopies (AFM, SEM, TEM) and reciprocal-space based x-ray Bragg diffraction analysis approaches. It is now a mature microscopy tool, widely developed at third and fourth generation synchrotron sources, and used by a large community of scientists for material science related problems [Ulv15, Atl23].

In the early 2010, Bragg ptychography has been proposed as a mean to investigate large and complex crystalline materials [God2011, Mas2017, Hru2017], pushing further the limits of BCDI [Li2021, Li2022].

In this talk, I will first detail the principles of BCDI and Bragg ptychography and review their successive developments. I will further highlight their recent advances, in the perspectives offered by 4th generation synchrotron sources, in terms of temporal resolution and strain analysis. I will finally describe the efforts developed to integrate these methods at synchrotron beamlines for enabling their use to a larger x-ray community.

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CoDI, Coherent Diffraction Imaging BL at ALBA II - technical details #169

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Co-Authors: *Lucia Aballe Aramburu, Miguel Anaya, Jesús A. Blanco, Irene Calvo, Ana Diaz, Javier Ignacio Diaz Fernández, Carlos Frontera, Miguel Ángel García Aranda, Cristián Huck Iriart, Allan Johnson, Catalin Alexandru Popescu, Angel Rodriguez Fernandez, Jose Solla-Gullon, Eduardo Solano Minuesa, Víctor Antonio de la Peña O'Shea*

Coherent Diffraction Imaging (CoDI) beamline, will have as primary mission the study of in situ and operando processes with nano-focus X-rays beams in thick samples (< 500 µm), covering a large number of nanostructured materials for different technological areas. The energy range 10-30 keV has been chosen to increase the penetration depth through the thick nanostructured materials under study. Along this energy range sub-50 nm X-ray beams will be provided. The X-ray beam transport from CoDI will be capable of delivering the high coherent properties of the X-rays from ALBA II. The percentage of coherent photons will be maximized with a pre-focusing scheme based on focusing mirrors and a secondary source located around 50 m from the source. The main focusing technique will be a set of Kirkpatrick–Baez(KB) mirrors located 230 m from the X-ray source that will provide a focused spot down to 32x32nm2 at the energy of 20 keV with a working distance of 150 mm. This will allow the installation of relatively big cells to perform in situ and operando experiments in nanostructure systems. The expected flux in the focused beam will range from 2 1011 ph/s for a high degree of coherence to 1012 ph/s with the full flux, at a 0.1% bandwidth at 20 keV. A second focusing method foreseen at CoDI will be a set of two Multilayer Laue lenses (vertical and horizontal) to focus the beam to 6x6nm2 (FWHM). A long sample detector distance (20 m) along the forward direction will allow the use of efficient single photon counting detectors while ensuring sufficient spatial resolution for direct measurements.

With X-ray beam parameters CoDI will be suitable for nano-scanning diffraction and fluorescence studies [1,2]. We propone to implement at CoDI several coherent based imaging techniques both in forward and diffraction directions, with the main focus in tomography-based imaging and Bragg Coherent Diffraction Imaging [3,4]. The setup will be compatible with imaging approaches such as holotomography, ptychography [5,6], Bragg-ptychography [7] and tele-ptychography[8-10]. Phase modulation techniques exploiting the nano-focusing capabilities of CoDI will be available. These methods will allow the control of the incoming beam wavefront, and hence, the distribution of the intensity at the focal plane. Thereby, we will improve the spatial resolution and optimize the measurement efficiency favouring the in situ and operando experiments [11].

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Non-destructive imaging of integrated circuits on the nanoscale by X-ray ptychography #149

Author: *Tomas Aidukas and Ana Diaz (Paul Scherrer Institute, Villigen PSI, Switzerland)*
Co-Authors: *Nicholas Phillips, Emiliya Poghosyan, Elisabeth Müller, A. F. J. Levi, Gabriel Aeppli, Manuel Guizar-Sicairos, Mirko Holler*

The semiconductor industry is capable to produce integrated circuits (ICs) featuring nanostructures in three dimensions with sizes of a few nanometers. Such structures include interconnects, circuits and transistors, made of different materials such as copper, alumina, titanium, etc…, embedded in lower density materials like silicon or silicon dioxide. Electron microscopy is a common technique to characterize these nanostructures, however the low penetration depth of electrons requires to cut the samples to allow for inspection. X-ray nano-tomography is an attractive alternative to perform a 3D characterization of integrated circuits non-destructively. In this presentation we show how a technique called ptychographic X-ray computed tomography (PXCT) [1,2] can achieve sufficient 3D resolution to characterize integrated circuits with critical dimensions down to about 20 nm in volumes of more than 1000 cubic microns [3,4]. After a brief introduction to X-ray ptychography and the required instrumentation, we will show examples of 3D images where the structures of ICs were revealed with a resolution of 15 nm. Finally, we will give an outlook regarding the inspection of ICs with even smaller critical dimensions, and its extension to larger volumes, in context with 4th generation synchrotron sources.

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X-ray nanoimaging of cement hydration #106

Author: *Miguel Ángel García Aranda (Universidad de Malaga)*

The hydration of Portland cements (PC) is complicated as there are many components with multi length scale heterogeneity. Thus, 3D nanoimaging techniques with high spatial resolution and scanning large fields of view are needed. Here, synchrotron X-ray near-field ptychographic tomography is used to investigate the hydration in 4D (3D + time) of a Portland 52.5R cement paste. Data were taken at the cSAXS beamline of SLS-PSI and recently published: Shirani et al. (2023) Nature Comm. 14:2652.

Ptychotomography yields electron density and absorption coefficient tomograms and the combined histograms are instrumental for characterizing amorphous components. The spatial resolution of ~250 nm attained with very good contrast allowed us to identify nanofeatures including densities and spatial distributions of amorphous constituents.

Details about the dissolution processes (of alite) and the growth processes (of C-S-H gel) will be reported and discussed. The particle size dependent alite spatial dissolution rates will be given. In the acceleration period, the alite spatial dissolution rate, for particles smaller than 3 microns, is ~100 nm/h. In the same period, alite particles with average sizes larger than 10 microns dissolved at ~33 nm/h, i.e. three times more slowly. In the deceleration period, the alite spatial dissolution rate, for particles larger than 10 microns, is ~25 nm/h. Moreover, the etch-pit growth rate, for particles larger than 10 microns, in the same interval was ~40 nm/h. Particle sizes are crucial for determining the kinetics of cement hydration processes.

Moreover, in the acceleration period 4-19h, the C-S-H gel growth rate has been (indirectly) estimated as ~33 nm/h. C-S-H gel shell densifies with time and its average electron density evolves from 0.47 e-Å-3 at 19 h to 0.53 e-Å-3 at 47 h. Some other details will be discussed including the requirements to directly studying the acceleration process. This could be carried out at CoDI beamline.

X-ray nanodiffraction as a probe for structural dynamics in Earth and Planetary sciences #182

Author: *Catalin Alexandru Popescu (ALBA Synchrotron)*

X-ray diffraction remains the main technique to provide structural information for understanding the physical properties of the matter. It is the most extensively employed experimental method to investigate a wide range of materials from crystalline, liquids, disordered to nanoscale materials in various sample environments. In Alba landscape MSPD beamline was one of the first public beamlines open at Alba synchrotron in 2012 and it was designed for powder diffraction and microdiffraction. Future developments aim at the installation of a new-state-of-the-art beamline with an endstation that offers improved performance in terms of focusing capabilities. With the ongoing Alba II upgrade, X-ray nanodiffraction fits in the big picture of diffraction program. Alba characterization capabilities will be enhanced through the new proposed Coherent Diffraction Imaging beamline offering submicron X-ray beam for nano-characterization. Here, I will discuss about Earth and planetary materials which are highly heterogenous and may encompass submicron mineral inclusions. Such samples bear materials from Earth, Moon and other planets and their crystal structure information provides important insights into the origin and evolution of the Earth and solar system.

Coherent X-ray Imaging for Quantum Materials #209

Author: *Allan S. Johnson (IMDEA Nanociencia)*

Quantum materials are expected to underpin many next-generation technologies due to their emergent and sometimes astonishing properties. On the other hand, the delicate balance of interactions and sensitivity to external stimuli that leads to these properties also makes the materials difficult to understand and integrate successfully in devices. Imaging the in-situ operation of quantum devices is thus essential to understanding their functionality and limitations [1]. For example oxide memristors can be based on the stochastic but non-volatile switching of nanoscale phase domains in quantum materials, but how such processes proceed remains controversial and is likely highly sensitive to defects and other external factors [2]. In this talk I will present an overview of the use of X-ray imaging methods to understand quantum materials and devices based upon them, with a special emphasis on our recent work applying coherent imaging methods to nanoscale phase transitions [3-5]. I will discuss the potential for coherent imaging with hard X-rays like those anticipated at the CoDI beamline to reveal functionality in such systems.

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X-ray nanoimaging in photo-electrocatalysis #137

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In recent years, the development and application of in situ and/or operando methods in photo-electrochemical systems is receiving increasing attention to gain better understanding and new insights into the phenomena and processes taking place in the photo-electrocatalysts under realistic working conditions. Thus, it is possible to perform real-time monitoring of photo-electrodes and to properly study dynamic changes under relevant working conditions. In this regard, nanofocused X-rays methods have proven be powerful tools to locally probe the composition, chemistry, and structure of different materials. In particular, focused coherent X-ray beams, now available at modern synchrotrons, can be used for advanced imaging techniques, which can give phase and absorption contrast images via ptychography and high-resolution 3D strain maps via Bragg coherent diffraction imaging (BCDI) and Bragg ptychography. Among these, BCDI appears as a promising three-dimensional imaging technique for operando studies of the shape and strain of the crystalline nanoparticles in various photo-electrochemical contexts. In this work, we will summarize some of the recent advances and remaining challenges on the use of BCDI for in operando studies in photo-electrochemical applications.

Unveiling New Frontiers in spintronics through Multi-Length Scale Imaging and Correlative Microscopy #167

Author: *Sandra Ruiz Gómez (ALBA Synchrotron)*

Three-dimensional nanomagnetic systems, featuring novel and unconventional spin textures, offer an exciting platform to explore new magnetic phenomena and possibilities for the development of more efficient, capable, and multifunctional technologies. However, harnessing these effects requires an understanding of the fundamental properties and behaviors.

The experimental study of such intricate systems poses a considerable challenge. Until recently, techniques available for probing magnetic materials were confined to flat surfaces or films, rendering the exploration of 3D magnetic systems nearly impossible. As a result, significant efforts have been made in the past few years to develop state-of-the-art imaging techniques across a range of length scales—from transmission electron microscopy to X-rays—that allow the visualization of magnetization in three dimensions.

This presentation reviews the developed techniques, showcasing examples that demonstrate how the synergies between CoDI and the other image capabilities of ALBA will allow the integration of multiple imaging approaches. This integration not only facilitates the study of 3D nanomagnetic systems but also opens avenues for in-operando characterization devices.

ABSTRACTS of the POSTER Contributions

Large segregation to the surface of the rare earth in NdCo5 amorphous thin films with perpendicular magnetic anisotropy probed by hard X ray photoemission #110

Author: *Javier Ignacio Diaz Fernández (Universidad de Oviedo)*
Co-Authors: *Jonathan Rodríguez-Fernández, Juan Rubio Zuazo*

Binary amorphous alloys made of rare earth and cobalt (RECo) are used in the form of thin films in several magnetic functional systems due to the easiness to change their magnetic properties by simply changing the type of RE and its concentration in the alloy. Depending on the RE, RECo alloys can be soft or hard ferromagnets, ferrimagnets, antiferromagnets, and they can have in-plane (IP) or out-of-plane (OOP) magnetic anisotropies. Sample deposition of these films have little requirements, they are currently deposited at RT using regular magnetron co-sputtering techniques. Interestingly, all magnetic RE have a similar chemical interaction with Co. NdCo5 amorphous alloys are ferromagnetic. They are interesting due to their OOP magnetic anisotropy which, at certain thickness, give rise to stripe domains. This property is used, for instance, in spin wave devices due to the regular spacing of these stripe domains, what makes the system behave as a 1-dimensional magnonic crystal whose symmetry axis can be field oriented. The OOP anisotropy is manifested above a thickness threshold of about 30 nm. Below this threshold, the films are magnetically soft with a weak IP anisotropy. Magnetometry, X Ray reflectivity and Hard Xray photoemission spectroscopy (HAXPES) were used to characterize several films with the same RE to Co concentrations (NdCo5) but with different thickness (5 nm, 10 nm, 50 nm and 65 nm). HAXPES experiments were done at the ESRF in the Spline beamline (BM25) at a fixed electron detection angle. Different depth probes were tested by changing the incident photon energy to 7 keV, 10 keV and 13 keV. The obtained Co to Nd concentration ratios were well below (about 2:1) the nominal concentration used in the evaporation process (5:1), indicating a substantial RE segregation to the surface of the films, explaining its thickness dependent magnetic behavior.

X-ray transmission microscopy of dipolar-coupled bilayers with crossed anisotropies for reconfigurable spin wave transport #111

Author: *Javier Ignacio Diaz Fernández (Universidad de Oviedo)*
Co-authors: *Alicia Estela Herguedas Alonso, Luis Manuel Alvarez-Prado, Simone Finizio, Javier Hermosa, Aurelio Hierro Rodriguez, Carlos Quirós, Silvia Tacchi, María Vélez*

Scanning transmission X-ray magnetic resonant microscopy (STXMRM) is used to study the magnetization reversal mechanisms of its stripe domains in dipolar-coupled layers (Py/Al/NdCo7) used for reconfigurable spin wave transport. The element specificity of X-ray permitted to observe the evolution of the stripe-domains as a function of the external magnetic field in each layer and determine the correlations between layers. This is key for the understanding of the hysteretic behavior observed in the spin wave transmission of the permalloy (Py) when it is dipolar coupled to the stripe domain textured NdCo7 layer. The images obtained show that the stripe domain imprinting on the Py layer is undetected in the range of fields where hysteretic properties in the dynamics of the Py magnetic moments are present, indicating that this effect must be determined by the stray field of the NdCo substrate without significantly affecting the static magnetic configuration of the Py layer.

Materials Imaging and Dynamics Instrument at the European XFEL Facility #116

Author: *Angel Rodriguez Fernandez (European XFEL)*
Co-author: *Anders Madsen*

This contribution presents the Materials Imaging and Dynamics (MID) instrument of the European XFEL Facility.
MID is specialised in coherent experiments perform in the Hard X-ray regime (5-20 keV). In a weekly basis different experimental configurations (SAXS, WAXS, XPCS, BCDI, Pump-probe,...) are used to study ultrafast dynamics in a large spectrum of samples from Proteins to single crystals. The AGIP detector, capable of working at 4.5 MHz with a pixel size of 200 um, can be moved in the horizontal plane (0-52 degrees) to cover a large range of Q angles. The sample detector distance range from 40 cm up to 8 m. Other detectors with smaller pixel size (Jungfrau, Epix, Andor Zyla,..) working at lower repetition rates are also available.

A selection of different experiments perform in the last 5 years of operation at the MID instrument are presented as example of the scientific scope and capabilities of the instrument.

Effects on the topological surface state induced by Er magnetic doping in the prototypical topological insulator Bi2Te3 studied by photoemission spectroscopy. #127

Author: *Beatriz Muñiz Cano (IMDEA Nanociencia)*
Co-Authors: *Julio Camarero, Ji Dai, Adriana I. Figueroa, Kevin García-Diez, Francisco Guinea, Rodolfo Mitanda, Aitor Mugarza, Jose Angel Silva-Guillén, Massimo Tallarida, Miguel Angel Valbuena, Sergio O. Valenzuela*

Magnetic topological insulators are a novel class of materials where the topologically protected band structure coexists with long-range ferromagnetic order. This coexistence can break time-reversal symmetry (TRS) and introduce a bandgap in the Dirac cone-shaped topological surface state (TSS). This effect is at the origin of the long-sought quantum anomalous Hall effect (QAHE), characterized by purely spin-polarized, dissipationless currents with significant applications in spintronics. However, realizing the QAHE experimentally is very challenging and has only been achieved under extreme temperature or doping conditions [1]. Therefore, further magnetic doping strategies must be investigated. Following our recent findings of spectroscopic evidence of TRS breaking by magnetic rare-earth surface dopants (Er and Dy) on a prototypical topological insulator Bi2Se2Te [2], we continued our investigation by replacing the substrate. Erbium dopants were deposited on the Bi2Te3 topological insulator, which has a more warped Fermi surface of the TSS compared to the former material. We present angle-resolved photoemission spectroscopy (ARPES) results measured at LOREA beamline in ALBA Synchrotron on the evolution of the TSS after Er surface doping and rationalize these results in terms of magnetic interactions with the TSS.

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The potential of in situ XAS to reveal the role of aliovalent dopants in TiO2 nanocrystals for advanced energy storage and smart windows #131

Author: *Mariam Barawi Moran (IMDEA Energia)*
Co-authors: *Miguel García-Tecedor, Ignacio José Villar García, Giulio Gorni, Marta Liras, Víctor Antonio de la Peña O'Shea*

Smart windows can dynamically adjust their optical properties to regulate sunlight and heat, increasing energy efficiency by reducing energy costs and greenhouse gas emissions. Electrochromic materials stand out given their ability to dynamically change their optical properties in response to an applied voltage. Metal oxides are the most investigated materials due to their low cost, high stability and low toxicity but must face limitations as low coloration speed, low electrical conductivity and lack of selectivity control. Aliovalent-doped TiO2 nanocrystals (NCs) have demonstrated improved electrical conductivity, advanced electrochemical energy conversion properties, and an ability to modulate optical transmittance further and in a more selective manner. In particular, V- and Nb-doped TiO2 NCs excel in energy storage applications, although they appear attractive for energy-efficient smart window devices. However, some aspects of the behavior of these materials in these applications remain to be explained in depth.

Advanced in situ characterization techniques, particularly X-ray Absorption Spectroscopy (XAS), play a crucial role in understanding of these materials in smart windows. XAS provides real-time insights into electronic and structural changes during operation, enabling to observe dynamic processes, study element-specific roles, and determine local atomic arrangements.

This study employs a combination of advanced in situ spectroscopy techniques, including XAS, spectro-electrochemistry (SEC), and Electrochemical Impedance Spectroscopy (EIS), to provide insights into the electrochemical reaction mechanisms within these doped nanocrystals. The experimental results demonstrate significantly improved charge storage capacities, particularly in the pseudo-capacitance storage mechanism. In situ SEC and XAS analyses reveal a more effective reduction of Ti4+ ions during the electrochemical process in doped NCs, while measurements of the V K-edge and Nb edge unveil the real active role of the dopants in the reduction process. This characteristic had not been seen until now, the active participation of the dopant in the TiO2 reduction process.

In summary, this research highlights the potential of XAS to investigate the significant potential of V- and Nb-doped TiO2 nanocrystals in advanced energy storage and smart window applications, while emphasizing the critical role of advanced in situ spectroscopy techniques in elucidating their complex mechanisms.

RIANA - Research Infrastructure Access in NAnoscience & nanotechnology #139

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RIANA is a Horizon-Europe-funded project to assist research in nanoscience and nanotechnology. Coordinated by DESY, the project runs from March 2024 for 4 years.

We support curiosity-driven research in nanoscience with open research questions for long-term impact and challenge-driven research in nanotechnology with targeted research questions for short- and mid-term impact.

At the core of the RIANA consortium is the ARIE (Analytical Research Infrastructures in Europe, arie-eu.org) network, which comprises European analytical networks with a focus on large scale research infrastructures (RIs).

Preliminary Studies for the Use of Sludges from the Petroleum Industry as Agricultural Fertilizers. Characterization by XANES #140

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Our research group RNM-160 has a research line focused on the valorization of waste. In this study, we focused on water clarification sludge, generated as waste from the water treatment system used for steam production and cooling in the refineries. These sludges are a complex mixture of suspended solid particles, organic matter, and other components dissolved in the water. Despite their industrial origin, these sludges are classified as non-hazardous waste according to the LER 190902 regulation for water clarification sludges. Currently, these wastes are disposed of in landfills.

Characterization of the sludge by ICP-MS (Inductively Coupled Plasma Mass Spectrometry) indicates a high iron content, suggesting potential reuse in the agricultural fertilizer industry, as iron is an essential micronutrient for plant growth. However, the bioavailability of iron critically depends on its oxidation state.

This work presents the XANES (X-ray Absorption Near Edge Structure) characterization performed at ALBA, which allowed the determination of the iron oxidation state in the amorphous phase of our sludges, laying the groundwork for studying an appropriate valorization protocol as fertilizers. Results indicate that the predominant mineralogical phase of the sample is ferrihydrite $(Fe^{3+})_2O_3\cdot 0.5H_2O$. XANES pre-edge deconvolution reveals that the sludge sample contains exclusively Fe (III), evidenced by the presence of a single peak in the fit, which logically aligns with the predominance of ferrihydrite in the major phase. X-ray diffraction (XRD) characterization did not detect the ferrihydrite phase, as this technique is more sensitive to well-defined crystalline phases. Since ferrihydrite in the clarification sludges is in an amorphous form, XRD may not be able to detect it. In contrast, XANES is more sensitive to amorphous phases, focusing on the absorption of X-rays by specific atoms and allowing detailed information on element speciation.

Currently, we are developing ways to valorize these sludges, which would not only contribute to the sustainability of the petroleum industry but also offer an innovative and ecological solution for agricultural fertilization.

Morphology and dynamics of domains and domain walls through asymmetric hole density-graduated 2D-arrays #144

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The manipulation of magnetic domain walls (DW) opens novel strategies to design spintronic devices with potential high impact in data storage, nonvolatile logic, and magnonic applications, which may include reservoir/neuromorphic computing, data storage, and sensors among others. For the implementation of several of those applications, asymmetric DW motion controlled by electrical voltages or currents and magnetic fields is needed. The prototypical asymmetric DW motion mechanism is the so-called ratchet effect, which consists of an easier propagation of domain walls in one direction than in the other thanks to an asymmetric pinning potential. Different mechanisms have been demonstrated to favour ratchet effect, being patterning into an asymmetric configuration one of the preferred options. 2D micropatterned arrays of asymmetric holes (2D-AAH) on magnetic thin films have been reported to show ratchet effect. In the large size regime, the DWs have been approximated as elastic lines of zero width that can distort (i.e., bend) throughout their length when pushed by a magnetic field. This bending was attributed to be the main factor determining DW propagation. However, in 2D-AAH, the optimum hole size for the occurrence of ratchet effects is twice the DW width. Therefore, when matching the hole size to the DW width of the particular magnetic thin film used, the DWs cannot be considered 1D objects and their morphologies play a very important role. Additionally, in regular arrays of asymmetric holes the very fine control of the ratchet effect can be challenging.

In order to tackle these two issues, we have fabricated 2D-AAH with different hole shapes (triangles and arrows), sizes (from 2.5 to 6 µm), and pattern structure (regular and with different gradients in density of holes in the array) and study them combining Kerr microscopy and X-Ray PhotoEmission Electron Microscopy. The gradient strategy has allowed us superior control over the movement of the DWs along the array, enabling us to write various and specific magnetization states, which can be observed as a whole in the Kerr microscope due to the large field of view of this technique (see the lower part of the attached figure). On the other hand, zoomed-up images taken with the PEEM at ALBA synchrotron have revealed a much more complex magnetization domain and DW configuration than predicted, as can be seen in the illustrative Figure 1. A full analysis of these phenomena will be presented at the conference.

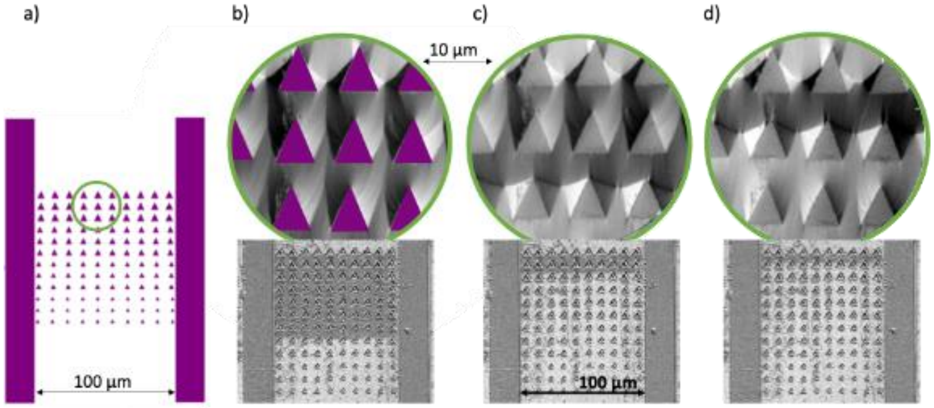

Figure. Left (a): Diagram of one of the studied arrays, with a gradient in the size of the triangles ranging from 3 to 6 µm, and separated by 7 to 4 µm respectively. Right (b, c, and d): PEEM-XMCD (top) and Kerr microscopy (bottom) images corresponding to 3 different magnetization states in the array. In all 3 cases, there is a DW pinned at the top of the array and another DW pinned in the middle (b), in the third-to-the-top line of triangles (c), and in the second-to-the-top line of triangles (d). PEEM-XMCD images correspond to the area marked by a green circle in (a), where the bases of the triangles (6 µm long) are separated 4 µm. Purple triangles in (b) are a guide for the eye.

GUI for magnetic imaging and tomography in MISTRAL #145

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Magnetic vector tomography allows visualizing the three-dimensional magnetic configuration of materials, therefore describing magnetic textures and states for further control and use in spintronics and magnetic storage devices [1]. However, it requires a long data processing that involves normalization, accurate 2D and rotation alignments of the angular projections and the use of specific reconstruction algorithms to finally calculate the 3D magnetization vector [2].

At the full-field Transmission soft X-ray Microscope available at MISTRAL beamline in ALBA [3], it is possible to perform magnetic vector tomography (MVT), using X-ray Magnetic Circular Dichroism (XMCD) as contrast mechanism. We have developed a Graphical User Interface (GUI) to simplify the analysis of the images from the alignment and averaging of single projections and normalization, to the reconstruction of the magnetization in three dimensions.

In MVT, two orthogonal angular projection series are acquired at different tilt angles since the sensitivity to the magnetization depends on its relative orientation to the X-ray beam. Images must be acquired for circular right and left polarizations along with their corresponding incident beam flat-field images (without the sample) to normalize each projection. The GUI processes the angular projections and outputs the transmittance images for each angle grouped by polarization. The 2D alignment of the transmission images between polarizations is crucial for an accurate computation of the XMCD signal. The GUI offers different alignment algorithms with pixel-wise and subpixel precision, adaptable to different sample shapes and dimensions (eg. nanostructures or multilayers). Absorption and magnetic signals are calculated for all the angular projections and, finally, these are aligned to a common rotation axis to perform the tomographic reconstruction. The GUI includes two reconstruction algorithms: for quasi-2D materials (thickness smaller than axial resolution) the algorithm described in [4] has been implemented, while for 3D samples, the method described in [2] is used. Both algorithms allow recovering the 3D vector magnetization within the sample volume.

Here, we demonstrate the GUI's capabilities using a synthetic dataset of 150nm Py/30nm Cu/ 50nm Py, revealing its 3D magnetization configuration. We also show that it is possible to have in-situ magnetic images from the acquired data probing that it is a valuable tool for MISTRAL users.

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Normalization for visualization of weak magnetic signals at MISTRAL beamline #147

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Magnetic 2D van der Waals materials have become interesting for the spintronics community due to their properties for magnetic devices, such as large Spin Orbit Coupling or the ability to control their magnetic properties externally [1]. One of their most important properties is that they can exhibit ferromagnetic order even when thinned down to a monolayer [2].

Transmission X-ray microscopy probes the magnetization at the nanoscale with high spatial resolution and element sensitivity. In the full-field Transmission X-ray Miscroscope (TXM) available at MISTRAL beamline in ALBA [3], the sample is shined with polarized X-rays to get magnetic contrast. To compute the X-ray magnetic circular dichroism (XMCD), transmission images with circular right (C+) and left (C-) polarizations with their corresponding flat-fields (FF) must be acquired. FFs are images of the incoming beam without the sample used for normalization. Images are normalized to obtain the transmittance and the XMCD signal. MISTRAL beamline provides an excellent magnetic contrast and it operates at low temperatures, making it an ideal technique for visualizing 2D van der Waals materials.

We measured mechanically exfoliated Fe3GeTe2 flakes at the Fe L3 edge (706.8eV) below their Curie Temperature (110K), with thicknesses from 100 nm to 5nm. However, the weak magnetic signal coming from the thinnest flakes is very sensitive to fluctuations or drifts of the incoming beam which can mask the physically relevant information. To overcome this, we characterized the FFs fluctuations in order to extract a phenomenological model to predict them, and then, develop a normalization to compensate for these fluctuations.

To quantify these fluctuations in the transmission images, we measured a high number of FFs and varied polarization over time. Our measurements show that the incoming flux decays (increases) exponentially for C+ (C-) polarizations. Taking this into account, we normalized each projection with a synthetic FF generated by fitting the intensity to an exponential function. In this way, we have a FF for each sample projection at the same intensity level, which mitigates the drifts or variations of the incoming beam.

In our poster, we compare our approach with the conventional normalization method for magnetic imaging and show the XMCD images of the Fe3GeTe2 flakes. We demonstrate that measuring low magnetic signals from quasi-2D materials as thin as 5nm is feasible with the TXM at MISTRAL beamline.

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In operando XRD study of Bi2O3, h-MoO3 and MnO2 electrodes during charge/discharge processes of Zn batteries #148

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Zn-ion batteries (ZIBs) are considered as a good candidate to replace Li-ion batteries. Analysis of intercalation/deintercalation mechanism of Zn2+ in the host material is a very important goal for improving the number of charge/discharge cycles of this battery kind. Zn-Air and Zn/Host (with Host at positive electrode being: MnO2, Bi2O3, h-MoO3) have been fabricated and the evolution of the electrodes has been tested "in operando" at the BM-25 ESRF beam line. Different electrolytes have been used, an ionic liquid-based gel polymer electrolyte and two different aqueous solutions: ZnSO4/NaSO4 and ZnTf2. Complementary HAXPES and XANES spectroscopy has also been carried out on the samples (results shown in references [1] to [3]).

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Tuning the magnetic properties of epsilon-Fe2O3 nanoparticles by Cr-doping #150

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Co-Authors: *Naureen Khanam, Javier Herrero-Martin, Zheng Ma, Nico Dix, Marti Gich*

Ferrites with a large magnetic anisotropy and a sizeable magnetization are relevant for applications in the field of information technologies. In particular, systems with giant coercivity are appealing for magnetic memories, and display natural ferromagnetic resonance (NFMR) at high frequency, which can be exploited in self-biased non-reciprocal devices for the next generations of wireless communications (6G and beyond).

Among these materials, ε-Fe2O3 is an orthorhombic ferromagnetic iron oxide (MS ~ 100 emu/cm3) with a strong uniaxial anisotropy [1] reflected in an enormous coercivity (HC ~ 20 kOe) at room temperature [2] and NFMR at around 180 GHz. The latter can be modified by substituting Fe3+ by other trivalent metals in a wide NFMR frequency range [3]. For the above-mentioned applications, the magnetic remanence of the ferrite has to be retained for very long periods of time. Since ε-Fe2O3 is metastable and can only be stabilized in form of nanoparticles (NPs), the magnetic relaxation can be an issue in crystals below a critical size [4]. Thus, it is important to control the synthesis of ε-Fe2O3 to obtain crystals above 30 nm but limiting the growth to avoid the transformation to the thermodynamically stable α-Fe2O3.

For Cr-substituted samples (ε-CrxFe2−xO3), we have observed an increase in the NPs anisotropy and we have found chemical ways to keep their magnetic hardness despite a tendency to softening derived from their smaller size compared to the undoped counterpart NPs. Also, using XAS and XMCD, we have (i) confirmed the isovalent character of the Cr substitution by XAS and XMCD, preferentially occupying octahedral sites, and (ii) addressed the question on the origin of the extraordinary coercive field of these NPs, which might be related to a possible nonzero orbital angular momentum of Fe cations at certain crystallographic site(s) in the structure.

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Bridging Science and IT: ALBA's Scientific Data Management Section #154

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Effective scientific data management is crucial for photon facilities like ALBA, enabling efficient data exploration by automatic pipelines and humans. The Scientific Data Management Section plays an unprecedented role at ALBA, by acting as a pivot between scientists and IT services. This poster showcases our section's approach to scientific data management, emphasizing two primary activities:

(1) Implementation of Data Management Software: the ALBA data catalogue (ICAT) provides users with a centralised interface to browse and visualize their data and metadata, as well as the notes taken during their experiment. Our section orchestrates the transition to NeXus format, jointly with the installation and configuration of the data catalogue in all beamlines, taking into account the particularities of each instrument. All together, these novelties will enhance the user experience at ALBA, ensuring compliance with the FAIR principles.

(2) Tool Provision for Data Processing & Analysis: We highlight some of the main software products created and maintained by our section, tailored to meet the diverse analytical needs of researchers at ALBA. These tools span a spectrum from automatic workflows, compression tools, interactive programs and visualization interfaces. Our future plan for Data Analysis as a Service (DAaaS) will also be presented.

Overall, we aim to illustrate how the Scientific Data Management Section contributes to the institution's research excellence by providing on-demand data analysis and management resources adapted to each instrument's needs.

Dynamic re-shaping of single Au NRs in colloidal suspension observed with ultrafast coherent diffractive imaging #168

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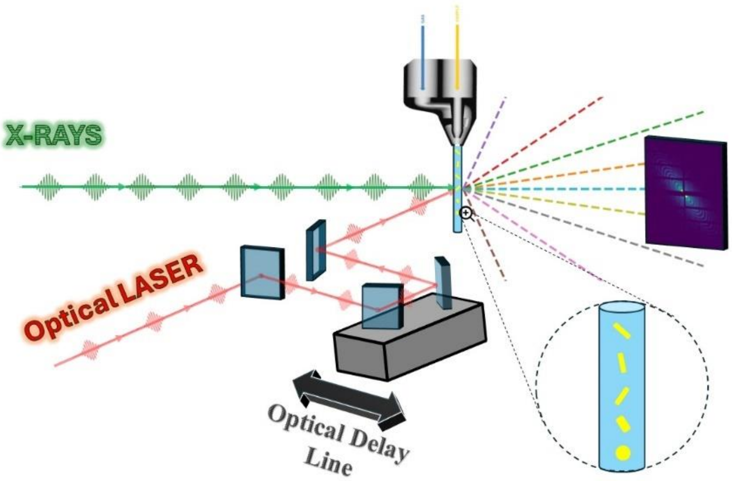
In recent years, coherent diffractive imaging (CDI) has emerged as a powerful technique for resolving atomic structures and shapes of individual large biomolecules and nanoparticles (NPs), offering unprecedented spatial resolution capabilities beyond the diffraction capability of conventional microscopy. The CDI technique, utilizing an X-ray source, becomes particularly viable when employed with X-ray free-electron lasers (XFELs). With the advent of XFELs, CDI of isolated nano-sized objects has become possible due to the unprecedented spatial and temporal characteristics of this radiation. Single particle imaging (SPI) has been reached and applied to study nanoclusters, gas-phase NPs, and large biological systems. Simultaneous development of improved reconstruction algorithms and more efficient sample injectors (including ultrathin liquid jets) has enabled the possibility of obtaining full 3D ensembles of NPs with varying shapes in the same sample. Recently, it became possible to combine SPI techniques at XFELs in pump-probe geometry using an ultrafast optical laser as a photoexcitation source (Fig. 1).

By combining ultrashort XFEL pulses with femtosecond optical lasers it became possible the study the structural dynamics in systems like gold nanorods (Au NRs), well-known for their unique optical characteristics. When gold nanorods particles are irradiated with near-infrared (NIR) laser pump excitation, their shape and size undergo significant changes. These alterations in length and width modify their aspect ratio, thereby affecting the localized surface plasmon resonance and, consequently, their optical properties. Here, we employed an NIR pump/hard X-ray probe experiment measuring transient Small Angle X-ray Scattering (SAXS) signals at SPB/SFX instrument of the European XFEL to track these dynamics in real-time.

We have carried out SAXS measurements in the SPI limit, collecting diffraction patterns corresponding to single Au NR at different time delays. The polarization, the fluence and the pulse duration of the NIR pump laser play a crucial role in these experiments, as it directly influences the excitation efficiency and the observed structural dynamics. We have successfully collected CDI signals varying those parameters for several NPs, including Au NRs, Au-Pt NRs and Au nanospheres. The preliminary analysis of the results indicates very rich structural dynamics occurring on both several picoseconds- and longer timescales (up to 1.2 ns).



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Influence of Crystallographic Structure on the OER Performance of Co & Ni-based Layered Hydroxides #173

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Co-Authors: *Roger Sanchis-Gual, Diego Hunt, Alvaro Seijas-Da Silva, Martin Mizrahi, Carlo Marini, Victor Oetreicher, Gonzalo Abellan (ICMol, UV)*

Water electrolysis stands out as one of the cleanest methods for hydrogen production; however, the oxygen evolution reaction (OER) involved in this process requires high voltages, posing an energetic efficiency challenge.1 Efforts over the past decade have focused on developing cost-effective electrocatalysts, with earth abundant materials gaining prominence as non-precious metal OER catalysts.2 Layered hydroxides (LHs) have proven excellent OER electrocatalysts, offering scalability, affordability, and composition with abundant elements. Despite their electrochemical prowess, fundamental aspects influencing OER, such as the role of LH crystalline structure, coordination environment, and cation distortions, remain understudied.3 Here we study different Co & Ni-based LH phases by in-house physicochemical and electrochemical characterizations, followed by XAS measurements in ALBA Synchrotron of the samples previous and after the OER, revealing that the crystallographic structure significantly impacts OER performance. The α-LH phase, distinguished from layered double hydroxide (LDH), demonstrated superior catalytic behavior in the case of both Co and Ni, introducing a new chemical signature for the rational design of more efficient LH-based electrocatalysts.4,5

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Applying X-ray Absorption Spectroscopy (XAS) at ALBA to the Study of Heavy Metal Pollution in Marine Sediments #176

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The global issue of waste and wastewater discharge into seas and oceans severely impacts coastlines and marine ecosystems, with the Mediterranean Sea, including the Spanish coastline, particularly affected. Polluted rivers, combined sewer overflows, wastewater treatment plants, and large mine dumps contribute to this problem. Off Barcelona (NE Spain), significant quantities of heavy metals, particularly chromium (Cr), have been found in sludge from a wastewater treatment plant accumulating on the inner continental shelf, with levels reaching up to 500 ppm (Palanques, 1994; Cerdà-Domènech et al., 2019). Another severe case is the dumping of approximately 57 million tons of hazardous mine waste into Portmán's Bay (SE Spain), causing persistent contamination and infilling the bay with tailings rich (Baza-Varas et al., 2023) in metals and arsenic (As). X-ray Absorption Spectroscopy (XAS) has been proved a critical tool for studying these contaminated sediments, providing detailed insights into the chemistry of heavy metals. Synchrotron-based X-ray spectroscopy (XANES) at NOTOS has been used to identify the proportions of Cr(III)/Cr(VI), providing essential information for assessing environmental risks and informing policy measures. Similarly, XANES at CLAESS has elucidated the formation of secondary As-bearing minerals, linked to the weathering of arsenopyrite and sulfate-reducing bacterial activity, which reduces As mobility and environmental release. XAS techniques are crucial in identifying how these heavy metals relate to sediment components, tracing their pathways, and understanding their transformation processes. These insights are essential for understanding the sources of heavy metal pollution and developing remediation strategies for these affected areas. Hence, it can be stated that XAS beamlines at ALBA have been effectively used to study heavy metal contamination in Mediterranean marine environments, including the analysis of Cr in marine sediments along the Barcelona coastline and the investigation of historical sulfide mine waste contamination in Portmán's Bay.

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NEXT - An international network for Nonlinear Extreme ultraviolet to hard X-ray Techniques #179

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Extreme Ultraviolet (EUV) table-top sources and soft to hard X-ray Free Electron lasers (XFELs) have opened a new era in science, providing ultrashort, coherent, and tunable pulses that are used to perform cutting-edge experiments in Atomic and Molecular physics, Condensed Matter Physics, Biology and Chemistry. However, most of the reported studies rely on linear light-matter interactions, which are fundamentally limited in the dynamical information they can provide. On contrary, nonlinear radiation-matter interactions have proven to be a powerful tool to unravel hitherto inaccessible properties.

The NEXT collaboration will capitalize on pioneering promising results, reported over the last decade, to create the first concerted experimental and theoretical effort aimed at implementing EUV/X-ray nonlinear spectroscopies at lab-based and large-scale facility short-wavelength sources, especially XFELs.

In this poster, we would like to advertise this new initiative and the recently funded COST Action (CA22148), which is coordinated by IMDEA Nanoscience institute in Madrid and started in October 2023. We would like to particularly encourage the Spanish groups to join the Action and contribute to this new endeavour.

Spain XFEL Hub: A bridge between the Spanish research community and XFEL facilities #180

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Co-Author: *Wojciech Gawelda*

The Spain XFEL Hub (SXH), a collaborative initiative by IMDEA Nanoscience, CSIC, and UAM, aims to establish a national platform with an international reach. This platform integrates diverse research fields, from Chemistry and Physics to Biology and Structural Biology. Its primary goal is to make XFEL technology and science accessible to researchers in Spain, enabling them to leverage the cutting-edge advancements provided by XFEL facilities. Notably, the Hub focuses on maximizing the opportunities available to the Spanish scientific community through the European XFEL laboratory (EuXFEL) near Hamburg, Germany.

The SXH offers essential expertise to:

1) Facilitate access to EuXFEL’s scientific instruments;
2) Assist in the preparation and execution of approved experiments;
3) Engage the Spanish scientific community in developing new methodologies, sample preparation, data analysis, and other technologies necessary for utilizing EuXFEL’s instruments;
4) Provide training and support to enable Spanish researchers to work independently with XFELs;
5) Organize scientific outreach events for both the scientific community and the general public;
6) Disseminate information about proposal calls for EuXFEL access and other international XFEL sources, along with related news, via the website and social media channels.

Electron transfer-induced structural changes in prion proteins studied by ultrafast X-ray absorption spectroscopy #181

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The misfolded isoform of the cellular prion protein PrPc is associated with transmissible spongiform encephalopathies (TSEs), a group of neurodegenerative diseases. It has been observed that the endogenous form of PrPc binds selectively to copper, and there is evidence suggesting that the reduction of this metal may be linked to the development of the disease. This study aims to investigate the redox behaviour of copper bound to prion proteins using the smallest peptide to which copper can bind, HGGGW (OPS), and to demonstrate that tryptophan is responsible for the reduction of Cu(II) to Cu(I).

We have used time-resolved X-ray absorption spectroscopy (TR-XAS) to track in real-time the photoredox reaction at Cu site in Cu-OPS (peptide) complexes in aqueous solution. The study seeks to show that the redox process occurring in the physiological environment can be replicated through electron transfer triggered by the optical excitation of a nearby tryptophan residue, which would clarify the mechanism of copper reduction and its potential structural consequences. A sub-200 fs constant was proposed for the electron transfer process from Trp to Cu(II), and the reverse oxidation process was monitored using time-resolved techniques. TR-XAS and TAS spectra were best fit with a single exponential kinetic model, yielding a time constant of 220±40 fs for TAS and 230±80 fs for TR-XAS, demonstrating that photoestimulation of the tryptophan effectively triggers the reduction of Cu(II).

In-situ micro-Laue x-ray diffraction strain mapping of piezoelectric actuators #186

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The possibility to tune mechanical, optical, magnetic and electric properties of low dimensional systems deposited on top of piezoelectric actuators is being intensively studied in recent years [1,4]. In this context, a detailed characterization of the local structural evolution of the piezoelectric response is important for drawing consistent conclusions about the correlations between the piezoelectric substrate deformation and the properties under investigation.

In this work, we have used micro-Laue x-ray diffraction to gain insight on the local atomic structure changes (5 deviatoric lattice parameters) of [Pb(Mg1/3 Nb2/3)O3]0.72-[PbTiO3]0.28 (PMN−PT) single crystals under external voltages ranging from 0 V to 400 V. Utilizing the experimental setup at BM32 beamline of the European Synchrotron Radiation Facility, we acquire full Laue x-ray diffraction pattern bidimensional maps with a spatial resolution of the order of 500 nm, leveraging a white x-ray beam with energies spanning 5-25 keV. Our results, based on the raster analysis of 10 microns x 10 microns Laue pattern maps reveal that the piezoelectric response is not homogeneous. Instead, it occurs as a combination of several micron-sized local domains that collectively contribute to the overall macroscopic behavior. This finding is significant as it may explain the dispersion sometimes observed in the local properties tuned with piezoelectric actuators, which can be site dependent in a scale of few microns.

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The Methodology Group at ALBA: Bridging Disciplines to Innovate Analysis in Science and Industry #193

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The Methodology Group, established in 2022, is dedicated to advancing multi-modal and multi-scale analysis techniques to meet the needs of both academia and industry. With the growing complexity of modern research, there is an increasing demand for expertise not only in data analysis and computational sciences but also in developing comprehensive workflows. Our group contributes at all stages of the methodological framework, from sample preparation protocols to the integration of experimental techniques and data processing. We ensure our pipelines, algorithms, and tools are tailored to the diverse instrumentation at our facility, supporting research across life sciences, materials sciences, chemistry, and electronic/magnetic systems.

We advance techniques and methods to serve the scientific community, collaborating institutions, and users by providing access to tailored workflows and expert guidance to optimize research outcomes. Working closely with the Interfaces group, which acts as a cross-link between X-ray beamlines, the Joint Electron Microscopy Center at ALBA (JEMCA), scanning probe microscopy (SPM) platforms, and the Scientific Data Management section, we develop and optimize workflows that enhance the facility's capabilities. Our goal is to establish ALBA as a leading facility in targeted research areas.

The Methodology Group fosters multimodal and multi-length scale projects by collaborating with ALBA staff and external scientists. We also initiate pilot projects, aligning research with industrial programs and creating strategic alliances. Moreover, we engage in researching and implementing new technologies while continuously seeking new partners to enhance our capabilities.

Our work is driven by externally funded projects that support the development of cutting-edge methodologies. A notable focus is steel research, where we contribute to significant advancements in understanding third-generation steels, reducing fuel consumption, and mitigating greenhouse gas emissions while improving passenger safety and studying hydrogen's effects on steel strength.

As we expand our expertise and collaborations, the Methodology Group remains committed to pushing the boundaries of data analysis and driving innovation across scientific and industrial domains.

3Sbar: The new HAXPES/SXRD ALBA synchrotron beamline #200

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Surface, structure and spectroscopy at bar (3Sbar) partial pressure beamline, is a hard X ray micro-spot beamline operated between 3 keV and 17 KeV that will perform core level ambient pressure photoelectron spectroscopy (APXPS) and Surface X ray diffraction (SXRD) at grazing incidence angles (~0.5º). Profiting total external reflection conditions, of the X-rays below the critical angle, a signal enhancement and surface sensitivity is achieved which allows depth profiling (with chemical sensitivity to the element and electronic structure) by changing the angle of incidence and photon excitation energy. Simultaneously, elastically scattered X-rays are collected with an off-vacuum 2D detector, sufficiently large to access a wide q-range for SXRD, critical when using tender X-rays allowing measuring strain variations during the operando process, lattice expansions due to insertion of atoms or ions, and crystalline gas condensates etc. 3Sbar beamline is currently construction design in ALBA synchrotron and will be operated at a maximum several bar local partial pressure (Virtual cell operation: the close proximity of sample and analyzer cone-end, where the gas is injected, yields a local high pressure of several bars), and maximum ~50 mbar total partial pressure (back-fill main chamber operation). The endstation will be also compatible with electrochemical experiments. The virtual cell operation, will allow the gas pulses injection in the ~ms range, which allows for the separation of thermodynamic surface properties and gas kinetics in the gas/solid reactions. In addition, the endstation is equipped with on line mass-spectrometer and gas-chromatography for the on-line gas analysis of the feed and gas products.

3Sbar will be a unique beamline aiming to provide fundamental understanding of a broad panel of surface science processes involving solid-solid, solid-liquid and solid-gas interfaces. This is a key aspect in many research topics as (but not only): batteries, catalysis, electrocatalysis, corrosion, sensors, atmospheric science, nitrogen fixation, 2D material fabrication, tribology, etc. Furthermore, 3Sbar beamline is the first beamline in ALBA designed for be fully operational in ALBA II (4th generation light source).