



# Journée Grands Instruments

Mardi 28 novembre 2023

D420

## Programme

09h00 – 09h25	Accueil des participant.e.s
09h25 – 09h30	Introduction
09h30 – 10h10	<b>Nicolas Martin</b> (Laboratoire Léon Brillouin) <i>“Small-Angle Neutron Scattering, a mesoscope for condensed-matter”</i>
10h10 – 10h35	<b>Stefania Pizzini</b> (Institut Néel) <i>“Electric field manipulation of magnetic properties of Pt/Co/oxide thin films”</i>
10h35 – 11h00	<b>Chafic Fawaz</b> (Institut Néel) <i>“High temperature superconducting oxychlorides: A light element model for cuprates”</i>
11h00 – 11h25:	Pause
11h25 – 12h05	<b>Guillaume Morard</b> (Institut des Sciences de la Terre) <i>“Time-resolved X-ray diffraction of geomaterials under high pressure and high temperature”</i>
12h05 – 12h30	<b>Edmond Chan</b> (Institut Néel) <i>“Neutron diffraction in <math>MnSb_2O_6</math>: coupled chiralities in a polar magnet”</i>
12h30 – 14h00 :	Déjeuner / Buffet
14h00 – 14h40	<b>Flora Yakhou</b> (ESRF) <i>“ID32, the soft X-ray spectroscopy beamline at ESRF”</i>
14h40 – 15h05	<b>Amit Pawbake</b> (LNCMI) <i>“Magneto-optical sensing of the pressure-driven magnetic ground states in bulk CrSBr”</i>
15h05 – 15h30	<b>Abdallah Nasserredine</b> (Institut Néel) <i>“Réacteur à cellule haute-pression/température pour l'étude des catalyseurs par XAS operando au Synchrotron”</i>
15h30 – 15h55:	Pause
15h55 – 16h20	<b>Ursula. B. Hansen</b> (ILL) <i>“IN20 upgrade and polarization analysis with PASTIS3”</i>
16h20 – 16h45	<b>Antoine Cornet</b> (Institut Néel) <i>“Unraveling the atomic dance in metallic glass-forming liquids and glasses in the multi-GPa range”</i>
16h45 – 17h30	Echanges sur l'activité de l'axe

Résumés (pages suivantes)

## **Small-Angle Neutron Scattering, a mesoscope for condensed-matter**

Nicolas Martin

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Small-Angle Neutron Scattering (SANS) is a ubiquitous technique in condensed-matter physics, with applications spanning a broad number of scientific areas. The main strength of this method is its ability to probe structures with typical sizes in the 1-100 nm range. This makes it an ideal tool to bridge the gap between diffraction and microscopy. Although traditionally devoted to the study of soft-matter and biophysical systems, SANS is now also considered as a method of choice for understanding the properties of magnetic meso-structures.

I will introduce the principles of SANS and illustrate its potential through various examples. Possible extensions of the method will then be briefly discussed. Finally, I will describe the SAM project for a fourth SANS instrument at the Institut Laue Langevin (ILL). SAM is currently under construction through a collaboration between the Laboratoire Léon Brillouin (LLB, Saclay) and the ILL. It should receive its first neutrons by the end of February 2024 and, soon after, welcome its first users.

## Electric field manipulation of magnetic properties of Pt/Co/oxide thin films

Cristina Balan<sup>1</sup>, Aymen Fassatoui<sup>2</sup>, Laurent Ranno<sup>1</sup>, Jan Vogel<sup>1</sup>, H  l  ne B  a<sup>2</sup>, Capucine Gu  neau<sup>2</sup>, Johanna Fischer<sup>2</sup> and **Stefania Pizzini**<sup>1</sup>

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The manipulation of magnetism with a gate voltage, and in particular by magneto-ionics - where the magnetization is controlled by an electric field driving the migration of ionic species - is a fast developing research field which opens the perspective of energy efficient magnetic devices. Magnetoionic effects under micropatterned electrodes in solid-state devices allow modifying magnetic properties locally, in a nonvolatile and reversible way. In this work, we illustrate some of the results obtained on Pt/Co/AlO<sub>x</sub> thin films using HfO<sub>2</sub> as a high-k dielectric layer as well as a ionic conductor. We demonstrate that tuning magnetic anisotropy, magnetization and Dzyaloshinskii-Moriya interaction with gating allows modifying "at will" the dynamics of nontrivial magnetic textures such as skyrmions and chiral domain walls. Through hard x-rays photoelectron spectroscopy (HAXPES) measurements carried out at SOLEIL synchrotron, we show that the change of magnetic properties can be directly attributed to the modification of the oxidation state of the cobalt layer via electric field driven oxygen ion migration.

## High temperature superconducting oxychlorides: A light element model for cuprates

Chafic Fawaz<sup>1</sup>, Benjamin Bacq-Labreuil<sup>2</sup>, Victor Porée<sup>3</sup>, Benjamin Lenz<sup>4</sup>, Alessandro Nicolaou<sup>3</sup>, Hervé Cercellier<sup>1</sup>, Laura Chaix<sup>1</sup>, Silke Biermann<sup>2</sup>, Matteo d'Astuto<sup>1</sup>

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The origin of high temperature superconductivity (HTS) in cuprates remains highly controversial in spite of an unprecedented research effort for almost 30 years [1]. HTS appears in close proximity to other symmetry-broken states of matter such as antiferromagnetic (AF) insulator and a still mysterious pseudogap (PG) phase at moderate doping. In this context, the discovery of hole-doped  $\text{Ca}_2\text{CuO}_2\text{Cl}_2$  (CCOC) HTS oxychloride cuprates [2-3], such as  $(\text{Ca}_{1-x}\text{Na}_x)_2\text{CuO}_2\text{Cl}_2$  (Na-CCOC), is very promising to investigate all these phases on common ground. Most notably, it has a simple  $I4/mmm$  1-layer structure, which is stable at all doping and temperatures, and has a strong 2D character due to the replacements of apical oxygen with chlorine [2].

In the PG phase, AF excitations (paramagnons) are a promising candidate for being at the basis of this rich phase diagram: opening the pseudo-gap, and perhaps being the boson exchanged in the Cooper pairing, leading to superconductivity.

Here, I will show Resonant Inelastic X-ray Scattering, at the Cu L3 edge, measured on doped Na-CCOC, up to the optimal doped region ( $x=0.18$ ), showing the evolution of the magnon dispersion with doping, that I will compare to the electronic structure near Fermi energy for the same dopings, measured in Angle Resolved Photo-Emission Spectroscopy.

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## Time-resolved X-ray diffraction of geomaterials under high pressure and high temperature

Guillaume Morard

Understanding the mechanisms and chemistry in play in the deep Earth without direct rock samples is a strong motivation to study the physical properties and phase relations of materials constituting our planetary interior.

As it formed, the Earth experienced a molten state as a result of large impacts, accretional energy and the contribution of short-lived radio-nuclides. This fully molten stage in planetary accretion, the so-called Magma Ocean (Tonks and Melosh, 1993), has strong implications for the subsequent evolution of the planet, from internal processes such as mantle convection and plate tectonics, up to the distribution of chemical elements, and notably the distribution of volatiles between the atmosphere and the interior of the planet (Elkins-Tanton, 2012), and may be different on Venus or other planets. The Earth then formed a differentiated structure, with a rocky mantle and metallic core, which is common among Earth-like planets and asteroids in the solar system. Past differentiation, as the solidification process starts upon cooling, the mechanical properties of the solid in coexistence with the liquid are required to constrain the heat dissipation related to orbital resonances, core-mantle differentiation mechanisms, and the long-term evolution of a mantle heat over the core

Conventional static compression using Diamond Anvil Cell recently allowed in situ X-ray diagnostics for samples over 500 GPa (Dewaele et al., 2018), albeit at ambient temperature. In addition, dynamic compression driven by high-power lasers easily reach pressures over 500 GPa and temperature over 10 000 K for geomaterials. Until recently, however, conventional diagnostics for laser-driven shock compression experiments were limited to optical measurement of pyrometry and velocimetry. The advent of new X-ray sources, such as Free Electron Laser (FEL) or upgraded synchrotrons (such as the Extremely Brilliant Source (EBS) upgrade at ESRF available since September 2020), opens a favorable window to develop new diagnostics or rethink the existing experimental programs. Indeed, the high brilliance of these new X-ray sources offers the possibility of unprecedented time-resolved in situ X-ray diagnostics, allowing to track high P-T processes at the nanosecond (ns) to microsecond ( $\mu$ s) timescales. Preventing chemical migration, contamination or grain growth, these diagnostics can now be coupled with dynamic or static compression, to unravel the structure and mechanical properties of geomaterials over a wide density range.

### References

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## **Neutron diffraction in MnSb<sub>2</sub>O<sub>6</sub>: coupled chiralities in a polar magnet**

Edmond Chan

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Multiferroic materials have been intensively studied these last decades for their interesting physics and their promising magnetoelectric applications [1]. Materials having a crystallographic chirality are particularly interesting in the sense that their structure couples to magnetism and can display novel magnetoelectric coupling. This is the case of MnSb<sub>2</sub>O<sub>6</sub> which crystallizes in P321 space-group.

Neutron diffraction techniques are powerful in determining both nuclear and magnetic structures. In addition, the use of polarized neutrons in our work allows to investigate on the structural chirality (using Schwinger scattering), and detailed magnetic ground state (using Spherical Neutron Polarimetry) [2]. By a combination of neutron experiments, we have determined a mixture of chiral structural and magnetic domains in MnSb<sub>2</sub>O<sub>6</sub>, highlighting the debated magnetic ground state [3]. We subsequently propose a mechanism leading to previously observed electric polarization based on coupled structural and magnetic chiralities.

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## **ID32, the soft X-ray spectroscopy beamline at ESRF**

Flora Yakhou

ESRF

ID32 is the only beamline at the European Synchrotron Radiation Facility that operates in the soft x-ray range with energies spanning from 400 to 1800 eV. It is making full use of all the unique characteristics of synchrotron radiation: high brilliance, energy tunability, polarisation and coherence of the beam to enable state-of-the-art studies of the magnetic and electronic properties of materials. Low temperature X-ray Magnetic Circular dichroism experiments are performed in a 9 Tesla high field magnet with a full surface science sample preparation facility attached to it. Very high resolution Resonant Inelastic X-ray Scattering measurements with a resolving power exceeding 30000 are routinely performed with our rotating arm spectrometer and various sample environments, with the added possibility of analysing the polarisation of the scattered photons. Transmission holography experiments exploiting the inherent coherence of the beam and the magnetic contrast arising from opposed polarisations enable the – possibly time-resolved - imaging of nanoscale magnetic domains with stunning resolution.

I will present all the capabilities presently offered by the beamline, future developments and the different access modes to beam time on ID32.

## Magneto-optical sensing of the pressure-driven magnetic ground states in bulk CrSBr

**Amit Pawbake**<sup>1,\*</sup> Thomas Pelini,<sup>1</sup> Ivan Mohelsky,<sup>1</sup> Dipankar Jana,<sup>1</sup> Ivan Breslavetz,<sup>1</sup> Chang-Woo Cho,<sup>1</sup> Milan Orlita,<sup>1</sup> Marek Potemski,<sup>1</sup> Marie-Aude Measson,<sup>2</sup> Nathan Wilson,<sup>3</sup> Kseniia Mosina,<sup>4</sup> Aljoscha Soll,<sup>4</sup> Zdenek Sofer,<sup>4</sup> Benjamin Piot,<sup>1</sup> M. E. Zhitomirsky,<sup>5,6</sup> and Clement Faugeras<sup>1,\*</sup>

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Competition between exchange interactions and magnetocrystalline anisotropy may bring new magnetic states that are of great current interest. An applied hydrostatic pressure can further be used to tune their balance. In this work, we investigate the magnetization process of a bi-axial antiferromagnet CrSBr in an external magnetic field applied along the easy axis. We find that the single metamagnetic transition of the Ising type observed in this material under ambient pressure transforms under hydrostatic pressure into two transitions, a first-order spin-flop transition followed by a second-order transition toward a polarized ferromagnetic state near saturation. This reversible tuning into a new magnetic phase is obtained in layered bulk CrSBr at low temperature by varying the interlayer distance using high hydrostatic pressure, which efficiently acts on the interlayer magnetic exchange and is probed by magneto-optical spectroscopy.

### References:

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## Réacteur à cellule haute-pressure/température pour l'étude des catalyseurs par XAS operando au Synchrotron

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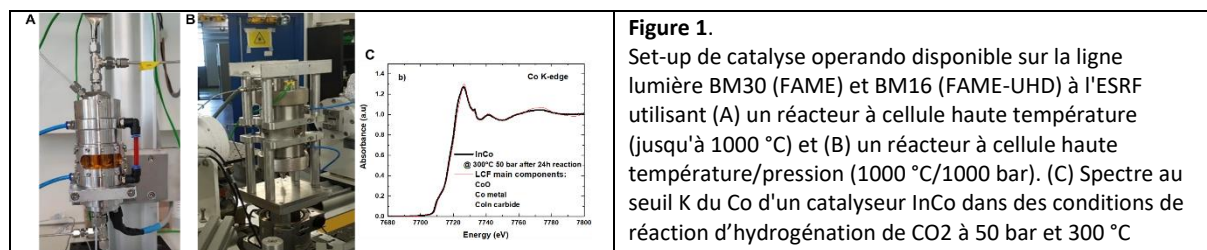
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La catalyse hétérogène joue un rôle essentiel dans de nombreux processus industriels et offre un grand potentiel pour relever les défis mondiaux actuels [1]. Cependant, bien que le potentiel des catalyseurs hétérogènes soit indéniable, notre capacité à concevoir des catalyseurs plus actifs est entravée par le manque de compréhension détaillée du catalyseur pendant la réaction. Ce manque d'informations est préjudiciable à la détermination des relations structure-réactivité catalytique des catalyseurs et à l'identification de leurs sites actifs, ce qui est nécessaire pour une meilleure compréhension des mécanismes catalytiques. Il est donc essentiel d'étudier les catalyseurs hétérogènes dans des conditions "operando", par des techniques expérimentales permettant, en parallèle, le suivi de l'évolution éventuelle de la structure du catalyseur sous gaz, et l'évaluation de son activité catalytique. La spectroscopie d'absorption des rayons X (XAS) utilisant le rayonnement synchrotron est bien adaptée à l'étude des matériaux catalytiques dans des conditions de fonctionnement. Cette technique permet une analyse adéquate de la structure électronique et géométrique d'un élément, en particulier la phase active d'un catalyseur [2]. Dans cette contribution, nous présentons plusieurs exemples d'études operando XAS de catalyseurs au cours de différentes réactions, en utilisant différents dispositifs expérimentaux. Notre premier réacteur (Fig.1.A) peut fonctionner à des températures élevées (jusqu'à 1000 °C) et à la pression atmosphérique [3], c'est-à-dire dans des conditions couramment appliquées dans la catalyse hétérogène d'intérêt industriel et environnemental. La conception offre la possibilité d'utiliser les modes de détection par fluorescence et par transmission et comprend un réacteur à flux continu en carbone vitreux, qui permet à la majorité des rayons X d'être transmis à l'échantillon. En revanche, notre second réacteur (Fig. 1.B) peut fonctionner à des pressions élevées (jusqu'à 1000 bar) [4], ce qui le rend idéal pour la caractérisation des catalyseurs dans des réactions nécessitant des conditions de haute pression. Les cellules de ces différents réacteurs ont été utilisées avec succès dans différentes expériences (Fig. 1.C) et sont à la disposition des utilisateurs des lignes de lumière FAME (BM30) et FAME-UHD (BM16) à l'ESRF.



**Figure 1.**

Set-up de catalyse operando disponible sur la ligne lumière BM30 (FAME) et BM16 (FAME-UHD) à l'ESRF utilisant (A) un réacteur à cellule haute température (jusqu'à 1000 °C) et (B) un réacteur à cellule haute température/pression (1000 °C/1000 bar). (C) Spectre au seuil K du Co d'un catalyseur InCo dans des conditions de réaction d'hydrogénation de CO<sub>2</sub> à 50 bar et 300 °C

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## IN20 upgrade and polarization analysis with PASTIS3

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IN20 is a thermal triple-axis instrument at the Institut Laue Langevin optimised for excitations in single crystals in the energy transfer range from 1 to 100 meV. The spectrometer can be equipped with various sample environments but is primarily used for polarised experiments. Its non-magnetic construction allows to use of all types§ and field strengths of ILL's cryomagnets (horizontal, vertical) in the full spectral range, as well as zero-field polarimetry (spherical polarization analysis). For the endurance program [1], IN20 has been upgraded with a new neutron velocity selector, as well as a double-focusing pyrolytic graphite monochromator and -analyser, improving both the neutron flux at the sample position and the background suppression.

In addition to the endurance upgrade program, a new setup for wide-angle XYZ polarization analysis, PASTIS3, has been commissioned on IN20. XYZ polarization analysis for inelastic neutron scattering is a powerful method for the separation of magnetic, nuclear coherent and incoherent scattering. However, in the thermal neutron range, these experiments have typically been carried out using the conventional triple-axis technique with a single analyser and detector, where polarized cross-sections are measured point by point. The PASTIS3 device uses two independent polarized <sup>3</sup>He neutron spin filters for polarizing and analysing the neutron spins. Here, the <sup>3</sup>He analyser cell provides a continuous 102-degree coverage of scattering angles. The neutron polarization direction is controlled by a magnetic field and a coil has been designed to deliver any field direction with high spatial homogeneity. The setup has been tested on IN20 together with the multi-angle analyser Flatcone [3]. I will here discuss the design of the setup and present our first results, as well as planned developments.

### References

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## Unraveling the atomic dance in metallic glass-forming liquids and glasses in the multi-GPa range

Antoine Cornet

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Glasses are dynamically defined materials: if crystallization can be bypassed, the viscosity of the liquid drastically rise by up to 10 orders of magnitude upon cooling, eventually leading to a “frozen” liquid which has lost its ability to flow [1]. In addition to this drastic dynamical arrest, the structure of the overall amorphous material does not suffer significant changes: the atomic structure of a glass is virtually undistinguishable from its liquid’s parent [2]. Therefore, a comprehensive microscopic theory of the glass forming liquids and glasses, a long-sought quest in condensed matter physics, requires an accurate description of the system dynamics i.e. its internal motion, from the inter-constituent’s length scale up to the macroscopic regime, and over the complete timescale of the ongoing relaxation processes.

Down to the atomic scale, X-Ray Photon Correlation Spectroscopy (XPCS) takes advantage of the impinging of coherent x-rays to reveal such dynamics at the atomic scale in amorphous materials: the diffracted intensity consists of interference patterns which reflect the exact, non-averaged, location of the scattering units, and the timescale over which the diffraction pattern changes corresponds strictly to the timescale over which the scattering units move [3].

So far, the limited flux of coherent x-rays at high energy (>20keV) restricted the possibility of heavy sample environments, limiting the application of potential external parameters and the study of the subsequent response of the materials. The advance of the 4th generation of synchrotron sources such as the EBS-ESRF lifts this obstacle as x-ray with coherent flux of 10<sup>12</sup> ph/s at high energy become available [4]. In this talk, I will present our work toward the development of XPCS in-situ under high pressure in the multi-GPa regime on samples compressed within a Diamond Anvil Cell (DAC). This opens the way to density dependent studies of the internal dynamics of glass and supercooled liquids in hard systems, characterized by a bulk modulus in the GPa range. As such I will present the technical developments needed to perform High Pressure XPCS, the current possibilities at the id10 beamline of the ESRF, and case studies in metallic systems.

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