EBS Workshop on
X-ray Spectroscopy of Magnetic Materials

ESRF Auditorium – Grenoble, France
7, 8 & 9 October 2019

• Programme
• Abstracts
• List of participants
# EBS workshop on X-ray Spectroscopy of Magnetic Materials

ESRF, Grenoble, France
ESRF auditorium

**Day 1 - Monday 7th October 2019**

<table>
<thead>
<tr>
<th>Time</th>
<th>Activity</th>
</tr>
</thead>
<tbody>
<tr>
<td>08:30 - 09:15</td>
<td>Registration in the ESRF Central Building entrance hall with welcome coffee</td>
</tr>
<tr>
<td>09:15 - 09:30</td>
<td>Welcome/Introduction</td>
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<tr>
<td></td>
<td>Harald Reichert</td>
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<td>Director of Research</td>
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<td></td>
<td>ESRF Grenoble, France</td>
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<tr>
<td>09:30 - 10:30</td>
<td>Session 1</td>
</tr>
<tr>
<td></td>
<td>Chair: Harald Reichert</td>
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<tr>
<td>09:30 - 10:00</td>
<td>Ultrafast X-ray Imaging of Magnetic Materials</td>
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<tr>
<td></td>
<td>Roopali Kukreja</td>
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<td></td>
<td>University of California Davis, USA</td>
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<tr>
<td>10:00 - 10:30</td>
<td>XMCD Studies of Actinide Compounds with Formally Nonmagnetic 5f-Electron Ground State</td>
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<tr>
<td></td>
<td>Roberto Caciuffo</td>
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<tr>
<td></td>
<td>European Commission, JRC-Karlsruhe, Germany</td>
</tr>
<tr>
<td>10:30 - 11:00</td>
<td>Coffee and poster session</td>
</tr>
<tr>
<td>11:00 - 12:30</td>
<td>Session 2</td>
</tr>
<tr>
<td></td>
<td>Chair: Marina Spasova</td>
</tr>
<tr>
<td>11:00 - 11:30</td>
<td>X-ray Spectroscopy on Molecular Magnets: Magnetometry, Electronic Structure, and more.</td>
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<tr>
<td></td>
<td>Fernando Bartolomé</td>
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<td>University of Zaragozé, Spain</td>
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<tr>
<td>11:30 - 12:00</td>
<td>Long-Lived Magnetic States in Single-Atom Magnets at Surfaces</td>
</tr>
<tr>
<td></td>
<td>Sebastien Stepanow</td>
</tr>
<tr>
<td></td>
<td>ETH Zürich, Switzerland</td>
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<tr>
<td>12:00 - 12:30</td>
<td>A New Twist to Interface Magnetism</td>
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<td>Sarnjeet Dhesi</td>
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<tr>
<td></td>
<td>Diamond Light Source Didcot, UK</td>
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<tr>
<td>12:30 - 14:00</td>
<td>Lunch at the EPN campus restaurant</td>
</tr>
<tr>
<td>14:00 - 16:00</td>
<td>Session 3</td>
</tr>
<tr>
<td></td>
<td>Chair: Philippe Saintavit</td>
</tr>
<tr>
<td>14:00 - 14:30</td>
<td>In-Situ Characterization of SPION Solutions by Means of 1s2p RIXS-MCD</td>
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<tr>
<td></td>
<td>Marcin Sikora</td>
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<td></td>
<td>AGH University of Science and Technology Krakow, Poland</td>
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<tr>
<td>14:30 - 15:00</td>
<td>Elucidating the Electronic Structure of Magnetite using X-ray Magnetic Dichroism</td>
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<td></td>
<td>Hebatalla Elnaggar</td>
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<tr>
<td></td>
<td>Utrecht University, The Netherlands</td>
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<tr>
<td>15:00 - 15:30</td>
<td>Magnetic- and Orbital-Excitations in 4d Transition Metals</td>
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<td></td>
<td>Hlynur Gretarsson</td>
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<td></td>
<td>Petra-III Hamburg, Germany</td>
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<tr>
<td>15:30 - 16:00</td>
<td>Impact of La Doping on the Local Electronic Properties of the Ru(^{4+}) Ion: A Ru L-Edge XMCD study on Ca(_2)-La(_x)RuO(_4)</td>
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<tr>
<td></td>
<td>Larissa Ishibe Veiga</td>
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<tr>
<td></td>
<td>University College London, UK</td>
</tr>
<tr>
<td>16:00 - 16:30</td>
<td>Coffee and poster session</td>
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<tr>
<td>Time</td>
<td>Session</td>
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<tr>
<td>16:30 - 18:30</td>
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<td>19:00 - 21:00</td>
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</tbody>
</table>

**Day 2 - Tuesday 8th October 2019**

<table>
<thead>
<tr>
<th>Time</th>
<th>Session 5</th>
<th>Chair: Alain Fontaine</th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>09:00 - 10:00</td>
<td></td>
<td>Magnetic Scattering and Spectroscopy at High Pressures at APS and APS-U</td>
<td>Daniel Haskel</td>
<td>APS Argonne National Laboratory, USA</td>
</tr>
<tr>
<td>09:30 - 10:00</td>
<td></td>
<td>X-ray Magnetic Circular Dichroism Spectroscopy and Magnetic Imaging at SPring-8</td>
<td>Motohiro Suzuki</td>
<td>Spring-8 Hyogo, Japan</td>
</tr>
<tr>
<td>10:00 - 10:30</td>
<td></td>
<td>Recent Developments at the Resonant Scattering and Diffraction Beamline P09 at PETRA III at DESY</td>
<td>Sonia Francoual</td>
<td>Petra-III Hamburg, Germany</td>
</tr>
<tr>
<td>10:30 - 11:00</td>
<td></td>
<td>Coffee and poster session</td>
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<tr>
<td>11:00 - 12:30</td>
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<td>Session 6 Chair: Pieter Glatzel</td>
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<tr>
<td>11:00 - 11:30</td>
<td></td>
<td>Soft X-ray XMCD at Ultra-Low Temperature</td>
<td>Philippe Ohresser</td>
<td>Synchrotron Soleil Gif-sur-Yvette, France</td>
</tr>
<tr>
<td>11:30 - 12:00</td>
<td></td>
<td>Hard XMCD under Extreme Conditions</td>
<td>Fabrice Wilhelm</td>
<td>ESRF Grenoble, France</td>
</tr>
<tr>
<td>12:00 - 12:30</td>
<td></td>
<td>XAS at Extremes: Magnetic Materials under Pressure and Pulsed Magnetic Fields</td>
<td>Olivier Mathon</td>
<td>ESRF Grenoble, France</td>
</tr>
<tr>
<td>12:30 - 14:00</td>
<td></td>
<td>Lunch at the EPN campus restaurant</td>
<td></td>
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<tr>
<td>14:00 - 16:00</td>
<td></td>
<td>Session 7 Chair: Cyrille Train</td>
<td></td>
<td></td>
</tr>
<tr>
<td>14:00 - 14:30</td>
<td></td>
<td>Modular Approaches to Magnetic Materials of 4d, 5d, and 5f Metal Ions</td>
<td>Kasper Pedersen</td>
<td>Petra-III Hamburg, Germany</td>
</tr>
<tr>
<td>14:30 - 15:00</td>
<td></td>
<td>Cyanido-Bridged Fe/Co Pairs : How to Make Them and How to Study the Switching Mechanism with XAS and XMCD Spectroscopies</td>
<td>Corine Mathonière</td>
<td>University of Bordeaux, France</td>
</tr>
<tr>
<td>15:00 - 15:30</td>
<td></td>
<td>Exploring the Magnetism of Molecules at the Nanoscale using Synchrotron and Scanning Probes-Based Techniques</td>
<td>Matteo Mannini</td>
<td>University of Florence, Italy</td>
</tr>
<tr>
<td>15:30 - 16:00</td>
<td></td>
<td>“Spin Injection” in Molecule-Based Materials toward Conducting Magnets</td>
<td>Rodolphe Clerac</td>
<td>CNRS Bordeaux, France</td>
</tr>
</tbody>
</table>
### Day 3 - Wednesday 9th October 2019

<table>
<thead>
<tr>
<th>Time</th>
<th>Session Description</th>
<th>Chair</th>
<th>Location</th>
</tr>
</thead>
<tbody>
<tr>
<td>09:00 - 09:30</td>
<td>Theoretical Description of X-ray Experiments on Magnetic Materials out of Equilibrium</td>
<td>Hubert Ebert</td>
<td>University of Munich, Germany</td>
</tr>
<tr>
<td>09:30 - 10:00</td>
<td>Band Structure Approach to RIXS</td>
<td>Alexander Yaresko</td>
<td>MPI für Festkörperforschung Stuttgart, Germany</td>
</tr>
<tr>
<td>10:00 - 10:30</td>
<td>Magnetic Surface Resonant X-ray Diffraction</td>
<td>Yves Joly</td>
<td>CNRS Grenoble, France</td>
</tr>
<tr>
<td>10:30 - 11:00</td>
<td>Electronic and Magnetic Character of UTe₂ Unconventional Superconductor</td>
<td>Alexander Shick</td>
<td>Institute of Physics Prague, Czech Republic</td>
</tr>
<tr>
<td>11:00 - 11:30</td>
<td>Coffee and poster session</td>
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<tr>
<td>11:30 - 12:30</td>
<td>X-ray Magneto-Chiral Dichroism</td>
<td>Andrei Rogalev</td>
<td>ESRF Grenoble, France</td>
</tr>
<tr>
<td>12:00 - 12:30</td>
<td>Recent and Future Developments at the Soft X-ray Spectroscopy Beamline ID32</td>
<td>Nicholas Brookes</td>
<td>ESRF Grenoble, France</td>
</tr>
<tr>
<td>12:30 - 12:45</td>
<td>Summary/Closure</td>
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<tr>
<td>12:45 - 14:00</td>
<td>Lunch at the EPN campus restaurant</td>
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</tbody>
</table>
Spin transport is the key for reading or writing bits in spintronic devices by utilizing the Giant Magnetoresistance effect or the spin transfer torque effect. Spin currents have also been shown to play important role in the ultrafast manipulation of magnetization via all optical switching. Hence, detailed understanding of spin currents is a crucial step in development of spintronic devices. In this talk, I will describe our recent experimental studies using emerging synchrotron and free electron laser techniques that can probe these materials with both high spatial and temporal resolution. I will discuss our work on imaging spin dynamics in nano-devices and probing spin transport across ferromagnet/copper interface. We have developed an extremely sensitive spectro-microscopy detection method based on element specific X-ray magnetic circular dichroism to probe spin transport in Co/Cu devices. The sensitivity of this new ‘lock-in’ technique has allowed us to detect the extremely small transient Cu magnetization with sub 100 nm spatial resolution. This technique has also enabled imaging of nanoscale motion of localized nonlinear spin waves in spin torque oscillator, allowing a detailed insight into p-like character of localized spin-wave excitation. I will also discuss our recent work on ultrafast imaging following optical pumping at free electron laser sources.
XMCD Studies of Actinide Compounds with Formally Nonmagnetic 5f-Electron Ground State

R. Caciuffo

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X-ray Magnetic Circular Dichroism (XMCD) is a very efficient tool to determine the orbital and spin components of the magnetic moment in actinide compounds. These quantities are easily obtained from two sum rules applied to the dichroic signal at the M$_4$ and M$_5$ spectral edges [1]. XMCD can be applied also to compounds where the actinide atoms have a formally nonmagnetic ground state, such as AmFe$_2$ [2] and PuCoGa$_5$ [3].

In AmFe$_2$, trivalent americium has a J = 0 ground state arising from the cancellation of the orbital and spin moments, whereas the iron sublattice orders ferromagnetically at about 700 K. Despite the nonmagnetic nature of Am$^{3+}$, neutron diffraction measurements suggest the presence of a non-zero 5f magnetic moment [4]. Taking advantage of the shell- and element-specific characteristics of XMCD, we have been able to confirm that Am in AmFe$_2$ has indeed a non-zero magnetic moment. We have found that the spin component is exactly twice as large as the orbital one and that the total Am moment is opposite to that of Fe, as expected under the assumption that the former is induced by the large molecular field arising from the latter [2].

PuCoGa$_5$ is an unconventional superconductor with a critical temperature T$_c$ =18.7 K. SQUID measurements on single crystal samples in the normal state provide a temperature independent susceptibility, suggesting a non-magnetic character of Pu. We used XMCD to study the vortex phase below T$_c$ and found that an external magnetic field induces a Pu 5f magnetic moment at 2 K equal to the temperature-independent moment measured by SQUID in the normal phase up to 300 K. This observation is in agreement with theoretical models [5] claiming that the Pu atoms have a nonmagnetic singlet ground state resulting from the hybridization of the conduction electrons with the intermediate-valence 5f electronic shell. Unexpectedly, the orbital component of the 5f magnetic moment increases significantly between 30 and 2 K; the antiparallel spin component increases as well, leaving the total moment practically constant. We suggest that this indicates a low temperature breakdown of the complete Kondo-like screening of the local 5f moment.

References
X-ray Spectroscopy on Molecular Magnets:
Magnetometry, Electronic Structure, and more

F. Bartolomé

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CSIC – Universidad de Zaragoza, 50009 Zaragoza, Spain, bartolom@unizar.es

The last decade of the past century witnessed the advent of both single molecule magnets (SMMs) and third generation synchrotron sources. The observation of quantum phenomena and the potential of SMMs to store information at the molecular level, boosted the interest of the scientific community on them. In parallel, X-ray scattering techniques have evolved to be irreplaceable tools in the study of magnetism of materials.

Many potential applications of molecular magnets imply its deposition onto a surface, and the preparation of small structures. Cyclic complexes are of particular interest because of their planar high symmetry. I will present recent work on wheel molecules of formula \(\{\text{Cr}_{10}(\text{OMe})_{20}(\text{O}_2\text{CCMe}_3)_{10}\}\) (Me = methyl), \([\text{Cr}_{10}]\), both in bulk and evaporated by direct sublimation in UHV on metallic single-crystal surfaces. A 2D self-organized quasi-hexagonal network monolayer of \([\text{Cr}_{10}]\) molecules, is formed on Cu(111) or Au(111). The magnetic characterization of monolayer, multilayer and bulk (powder and singlecrystal) samples of \([\text{Cr}_{10}]\) has revealed that this cluster exhibits an unusually high Sz-ground state.

Molecular overlayers on substrates have also a broad field of application in catalysis, sensing, molecular electronics, light-to-energy conversion, etc. I will present our recent work on Iron-phtalocyanines (FePc), which have been proposed as substitutes for precious metals in catalysis of the Oxygen Reduction Reaction. Sub-monolayer phases of FePc on Ag(110) are catalitically active. Remarkably, in oxygen-dosed phases, \(\text{O}_2\) intercalates between the molecules and the surface, substantially changing the Fe magnetic moment [1]. XMCD allows to determine the distribution of non-oxygenated, and oxygenated species.

The path from the bulk to the surface in molecular magnetism implies a change in the available experimental techniques, in particular for the study of magnetic relaxation. In the bulk, relaxation is mostly studied by AC magnetic susceptibility, but when characterizing a thin-film sample, the signal from the substrate is orders of magnitude larger than the one from the sample itself. Due to its brilliance and chemical selectivity, XMCD has become the magnetometry of choice in surface-deposited molecular magnetism [2]. However, the only remaining witness of slow magnetic relaxation is bistability, which only appears in extremely slow relaxing systems, by opening the hysteresis loop. The information about the processes originating SMM behavior is mostly lost. The development of user-friendly X-ray Scattering techniques to study magnetic dynamics would be of great interest in the future. Hopefully, EBS may allow the inception of such experimental techniques at the ESRF.

References
Magnetic atoms on surfaces are emerging as a new class of systems with exceptionally long spin relaxation times, which allows for reading and writing magnetic bits on the atomic scale. [1-3] The magnetic properties of the single-ion magnets depend crucially on their atomic environment and enhancing their spin dynamics may lead to the development of single-atom qubits. Recent observations of magnetic remanence in individual Ho atoms adsorbed on ultrathin MgO(100) layers on Ag(100) provided the first evidence of a single atom magnet on a surface. [1] The opening of the hysteresis loop indicates that the lifetime of Ho atoms is on the order of hours at cryogenic temperatures. Meanwhile more rare-earth adatom systems have been identified having exceptionally long spin relaxation time $T_1$.

Despite the raising interest in these systems, it is still not clear which factors determine their very long relaxation time and if a long coherence time can be expected. The talk highlights our recent efforts in understanding the magnetic properties of single-atom magnets on surfaces using synchrotron light. We put particular emphasis on the different contributing factors to long-lived magnetic states including strong uniaxial magnetic anisotropy, symmetry protection of the ground state from quantum tunneling and other first order scattering processes as well as the peculiarities of the spin-phonon coupling with the supporting substrate.

References
A New Twist to Interface Magnetism

S. S. Dhesi

Diamond Light Source, UK dhesi@diamond.ac.uk

On beamline I06 at Diamond Light Source (UK) electron microscopy combined with polarised X-ray spectroscopies allows high-resolution imaging of strain and electrical effects on nanomagnetism. In this talk, recent results using PhotoEmission Electron Microscopy (PEEM) combined with X-Ray Magnetic Linear Dichroism (XMLD) and X-Ray Magnetic Circular Dichroism (XMCD) will be presented.

Antiferromagnetic spintronics aims to exploit zero net magnetic moment materials as efficient generators, detectors and transmitters of spin current. PEEM, with magnetic contrast arising from XMLD, has been employed to directly image changes in the antiferromagnetic domain structure of CuMnAs [1,2] after electrical rotation of the magnetic moments. The XMLD-PEEM images are correlated with in situ Anisotropic Magnetoresistance transport measurements [3].

XMLD-PEEM imaging has also been used to directly visualise the antiferromagnetic domain structure of epitaxial (111)-oriented BiFeO₃ (BFO) films. Angle-dependent XMLD-PEEM images are combined to reveal a sub-micron network of antiferromagnetic domains (see Figure 1) that are coherently coupled to monoclinic domains. The magnetoelastic coupling is found to stabilise the antiferromagnetic domain structure, providing a possible pathway towards strain-engineering multiferroic domains in (111)-oriented BFO films [4].

Finally, I will explore electrical switching of Ni films grown on PMN-PT substrates. XMCD-PEEM maps of the in-plane magnetization reveal a shear-strain mediated magnetoelectric effect [5].

![Figure 1: A full spatial threshold map of cycloidal domains in BiFeO₃ (right image) constructed by appropriately combining XMLD-PEEM images collected at multiple angles (left 3 images). The contrast is consistent with the sample surface comprising an equal number of the 3 k-domains.](image)

References
In-Situ Characterization of SPION Solutions by Means of 1s2p RIXS-MCD

J. Kuciakowski¹, A. Kmita¹, D. Lachowicz¹, K. Pitala¹, M. Wytrwał¹, J. Ablett³, A. Juhin⁴, D. Kozię⁵, M. Sikora¹

¹AGH University of Science and Technology, Kraków, Poland, ²European Synchrotron Radiation Facility, Grenoble, France, ³Synchrotron SOLEIL, Gif-sur-Yvette, France, ⁴IMPMC, Sorbonne Universités, UMR CNRS 7590, Paris, France, ⁵CHyN, Hamburg University, Hamburg, Germany, marcin.sikora@agh.edu.pl

Magnetic properties of superparamagnetic iron oxide nanoparticles (SPION) in solutions are routinely determined using volume magnetometry. Quantitative analysis of such measurements is often questioned by uncertainty in their size, concentration and chemical composition. In this talk we will discuss the possibility of application of 1s2p RIXS-MCD [1,2] (magnetic circular dichroism in resonant inelastic x-ray scattering) for in-situ characterization of magnetic properties of SPION solutions (Figure 1).

Figure 1: Principle of 1s2p RIXS-MCD on SPION solutions. (A) Definition of transitions involved, namely 1s→3d excitation and 2p→1s emission, and the energy transfer scale. (B) Sketch of the experimental setup. (C) Typical 1s2p HERFD-XAS (high energy resolution fluorescence detected x-ray absorption spectrum) of spinel iron oxide probed at varying incident energy and constant emission energy corresponding to the maximum of Ka1 fluorescence line. (D) RIXS plane, two dimensional map of emission intensity probed in the incident energy range marked in (C) and emission energy (transfer) spanning Ka1 resonance. (E) RIXS-MCD plane being the difference of RIXS probed with opposite helicity of incident x rays. Solid and dashed lines in (D) and (E) correspond to HERFD scans probed at emission energy corresponding to maximum of 1s2p RIXS and MCD features within Fe K pre-edge.

Details of the experimental procedure and results of the preliminary measurements performed on dispersions of pristine, zinc and cobalt doped spinel iron oxide nanoparticles will be presented. The feasibility of probing site selectively magnetic properties of 3d ions simultaneously with their site distribution in spinel ferrite lattice will be discussed. Finally, the solution-free magnetization profiles of SPION derived from the field dependence of RIXS-MCD intensity will be compared to volume magnetometry data. In this way the distribution function of magnetic diameters of SPION can be estimated without the uncertainty related to diamagnetic contribution of the solvent.

References
Elucidating the Electronic Structure of Magnetite using X-ray Magnetic Dichroism


Debye Institute for Nanomaterials Science, Utrecht University, Institut de Minéralogie, de Physique des Matériaux et de Cosmochimie, CNRS, Sorbonne Université, European Synchrotron Radiation Facility, Faculty of Physics and Applied Computer Science, AGH University of Science and Technology, Institut für Theoretische Physik, Universität Heidelberg, Photon Science Division, Paul Scherrer Institut.

H.M.E.A.Elnaggar@uu.nl

Transition metal oxides have gained a lot of interest due to their unique properties most famous of which are: superconductivity, multiferroicity, magnetism, catalysis et cetera. The rich physics offered by these systems arises from the intricate interplay of the various degrees of freedom (e.g. spin, orbital, charge and lattice). X-ray spectroscopy has become an established tool that can uncover element- and site-specific information offering an ideal toolbox to study strongly correlated systems. In particular, carefully designed experiments with optimized scattering geometries can reveal subtle spectral features that are typically indistinguishable. However, the challenge to optimize and interpret data from such experiments persists and has hindered the full exploitation of dichroic techniques.

In this talk, I will discuss a series of X-ray magnetic dichroism measurements on the prototypical Mott insulator, magnetite (Fe₃O₄). This illusive system remains a widely debatable subject ever since the phenomenal work of Verwey in 1939 [1] due to its complexity: Fe₃O₄ is a mixed valence, strongly correlated system where many interactions such as Jahn-Teller (dynamical and static), spin-orbit, Kugel-Khomskii and phonons are very close in energetics. As a matter of fact, the origin of the metal to insulator transition and the magnitude of the orbital magnetic moment in Fe₃O₄ are contentious [2-5]. I will show here that X-ray magnetic dichroism measurements in combination with theoretical simulations can offer a unique perspective on these open questions. I will focus on the following two aspects:

(a) Investigation of non-collinear ordering of the orbital magnetic moments in the high temperature phase of Fe₃O₄ using a combination of X-ray linear and circular magnetic dichroism measurements in the hard X-ray regime [6].
(b) Investigation of trimeron correlations in the high temperature phase of Fe₃O₄ using resonant inelastic X-ray scattering in the soft X-ray regime [7].

References

* Co-authors are presented in alphabetical order
In this talk I will address our interest in the 4d transition metals. In particular focus will be given to the Ru-based materials, where the presence of comparable energy scales, namely spin-orbit coupling (SOC), on-site Coulomb repulsion (U), and the crystal electric field (CEF) can give rise to non-trivial ground states [1,2]. I will demonstrate that by utilizing the well-known technique Resonant Inelastic X-Ray Scattering (RIXS) in the tender X-ray range [3] one can probe directly the key parameters and their interplay in these materials. Two examples of magnetic compounds will be given, the honeycomb-lattice SrRuO$_6$ [4] and the square-lattice Ca$_2$RuO$_4$ [5]. In the former the relatively strong nearest-neighbour exchange allows us to observe a magnon with a bandwidth of close to 200 meV, while in the latter the orbital excitations reflect its unusual magnetism. These two examples demonstrate the importance of Ru L$_3$-edge RIXS in exploring magnetic- and orbital-exitations in ruthenates and encourage further improvements in instrumentation.

References
Ca$_2$RuO$_4$ has attracted considerable attention as the Mott insulating analogue of the unconventional superconductor Sr$_2$RuO$_4$ [1]. It displays a metal to insulator transition (MIT) at $T_{\text{MIT}} \approx 357$ K, concomitant with a first-order structural phase transition and an antiferromagnetic (AFM) ordering below $T_N \approx 110$ K [2]. Contrary to many Mott insulators, the nature of the insulating ground state of Ca$_2$RuO$_4$ arises from the combination of Hund’s and spin-orbit couplings (SOC) as well as crystal fields, which makes the low-energy electronic structure very sensitive to structural distortions acting on the local Ru$^{4+}$ environment [3].

In this context, dramatic changes in the insulating state of Ca$_2$RuO$_4$ have been achieved by internal chemical pressure, where the substitution of Ca by La [4] suppress the MIT and drive the system to a metallic state. The different radii of the La$^{3+}$ and Ca$^{2+}$ ions cause the compressed RuO$_6$ octahedra of pure Ca$_2$RuO$_4$ to be progressively stretched along the c axis for increasing doping levels. This, in turn, is expected to significantly change the local physics of the Ru$^{4+}$.

Using a variety of X-ray scattering (REXS) [4] and absorption techniques (O K-edge XANES [5] and Ru L-edge XMCD [6]) we address in detail the impact of the structural changes on the electronic and magnetic properties of Ca$_{2-x}$La$_x$RuO$_4$ with $x = 0, 0.05(1), 0.07(1)$ and $0.12(1)$. The results show that La doping induces significant changes in the local crystalline environment at the Ru sites. The AFM ordering observed in Ca$_2$RuO$_4$ parent compound persists upon La substitution [4], thus excluding the presence of a predicted ferromagnetic phase [7]. The crystal field tuning caused by the structural distortion has a visible effect on the hole population of the Ru t$_{2g}$ orbitals [5]. Finally, both the non-statistical absorption branching ratio BR and the sizeable Ru orbital moment revealed by XANES and XMCD measurements confirm a significant impact of SOC in the low-energy physics of the insulating phase [6].

References
Spin Excitations Studied by Resonant Inelastic X-ray Scattering

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Magnetic order results from the exchange interaction among neighbouring moments at the interatomic scale. A microscopic description of magnetism requires thus the determination of both the atomic moments and their interaction. Both quantities concur to give the energy elementary spin excitations, i.e. magnons or spin waves, and of their dispersion. And, ultimately, to the dynamical susceptibility. This class of experiments has been the exclusive realm of inelastic neutron scattering for about half century. But since a few years, resonant inelastic X-ray scattering (RIXS) has been demonstrated to be effective for the determination of magnon dispersion in selected materials, mostly based on transition metals. I will explain why the choice of the absorption resonance is the key for these experiments and I will present some important examples concerning mostly antiferromagnetic materials, such as cuprates [1,2,3], NiO [4], iridates [5], ruthenates [6].

References
Multiferroic materials that present magnetoelectric (ME) coupling are possible candidates for low energy consumption magnetic storage media [1], since the magnetic information can be switched by an electric field instead of an electric current. There are only a few materials that present ME coupling in one single system, so called intrinsic multiferroics [2]. The electric polarization in these systems is often very small and in many cases the ordering temperature is much below room temperature. That has led to the development of artificial systems in the attempt to achieve room temperature operation. In such systems, a heterostructure of, for example, ferroelectric and ferromagnet are combined and the magnetoelectric coupling occurs at the interface [3].

In this talk I will present our work on heterostructures exhibiting ME coupling and how X-ray dichroism can help in the characterization and understanding of these systems. In La$_{0.7}$Sr$_{0.3}$MnO$_3$/[Pb(Mg$_{1/3}$Nb$_{2/3}$)O$_3$]$_{0.68}$-[PbTiO$_3$]$_{0.32}$ (011) (LSMO/PMN-PT) we have observed a shift on LSMO Curie temperature (Tc) of 10K close to room temperature by switching the ferroelectric polarization [4]. The shift in Tc is remanent in electric field. Simulations of the X-ray linear dichroism (XLD) spectra show good agreement with a structural modification in LSMO driven by the change in strain coming from the piezoelectric substrate. X-ray magnetic circular dichroism (XMCD) measurements along different directions point to an overall decrease in moment. Therefore, the shift in Tc likely originates from an increase (decrease) of electron hoping which benefits (hinders) the double exchange mechanism. In another system, the co-existence of two types of magnetoelectric coupling mechanisms at room temperature were unraveled by XMCD [5]. In a Co wedge deposited on PMN-PT (011) our XMCD measurements as a function of Co thickness point to the coexistence of a charge screening effect restricted to Co/PMN-PT interface together with a strain driven mechanism. Density Functional Theory calculates the contribution of each effect proposing a spiral state in the Co thin film created by the added effect of both coupling mechanisms.

References
Magnetism Investigations by XMCD & XRMS at ALBA BL29: Recent Highlights and On-Going Developments

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This talk will provide an overview of the Beamline for X-ray Resonant Absorption and Scattering at ALBA, BOREAS BL29, highlighting some experiments using spectroscopic or scattering approaches. At BOREAS, experiments use either the beamline XMCD vector-cryomagnet or a multipurpose UHV reflectometer in combination with x-ray photons of energy on the range of 100 eV to 4000 eV (VLS-grating monochromator) and full-polarization control (Apple II EPU). Example of studies range from in-situ investigation of the magnetism of isolated atoms or single molecules, to the properties of oxide materials in thin-film or bulk crystal form, topological insulators, or 2D materials such as Graphene. A brief account on main experiment topics, statistics and productivity will be provided. The second part of the talk will have an emphasis on scattering, and will present details on the instrument developed, remarking challenges addressed or remaining. Present limitations and directions for on-going or future development will be briefly discussed.

References
Chirality in Thin Films and Multilayers probed by Soft X-ray (Coherent) Scattering

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Many current forthcoming applications of magnetic materials involve heterostructures or alloys containing magnetic and non-magnetic elements. X-ray Resonant (Coherent) Scattering is the technique of choice to probe such phenomena thanks to its element-selectivity and spatial sensitivity.

In this presentation I will introduce the experimental set-up that we developed at SOLEIL and illustrate their capabilities. SEXTANTS [1] is a beamline of the SOLEIL synchrotron, covering the 50-1700eV energy range dedicated to soft x-ray scattering. The resolving power exceeds 10^4 and maximum flux on the sample ranges from 1×10^{14} (100 eV) to 2×10^{13} (1000 eV) ph./s/0.1% bw. The beamline main objective is the investigation of the electronic and magnetic properties of solids using three scattering techniques: resonant inelastic x-ray scattering (RIXS), x-ray resonant magnetic scattering (XRMS) and coherent x-ray scattering (CXS), the last one including also imaging via Fourier transform holography (FTH) [2].

In the second part, several recent results obtained at SEXTANTS beamline will be presented, and in particular I will show that XRMS is the tool to study magnetic chirality as illustrated with two systems: on one hand, I will briefly introduce the approach using prototype Pt/Co multilayers in which Dzyaloshinskii-Morya interaction (DMI) is induced by the inversion symmetry breaking at the interfaces [3,4] and on the other hand, an investigation we have done of BiFeO₃ thin epitaxial layers in which the “bulk” DMI plays a major role in its magnetic configuration and in particular in the stabilization of the antiferromagnetic cycloid. Combining real space [5] and reciprocal space [6] approach we reveal the presence of periodic chiral AFM objects along the ferroelectric (FE) domain walls.

In the last part I will present the latest development of resonant scattering and in particular how the use of the x-ray coherence available at modern light source allow nowadays to image the sample with a spatial resolution of few tens of nanometers and a time resolution ranging from ns down to few ps timescale.

References
We describe current capabilities at the Advanced Photon Source for probing the evolution of magnetic order at high pressures using resonant X-ray absorption and scattering techniques in the diamond anvil cell. The complementarity of resonant magnetic scattering and x-ray magnetic circular dichroism for these studies is demonstrated with recent work on compressed beta-Li$_2$IrO$_3$, a hyperhoneycomb iridate where spin liquid and dimerized phases appear to compete for the ground state [1-4]. Opportunities in this area presented by the upcoming upgrade of the APS source (APS-U) will be discussed.

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References
X-ray Magnetic Circular Dichroism Spectroscopy and Magnetic Imaging at SPring-8

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X-ray magnetic circular dichroism (XMCD) spectroscopy is a key technique to study the magnetic materials, offering the element specificity and the high detection sensitivity of magnetic moments. The XMCD technique can be combined with the state-of-the-art X-ray focusing optics such that scanning magnetic microscopy using a circularly-polarized X-ray nanoprobe will become possible. At SPring-8, we have developed XMCD spectroscopy and magnetic microscopy/imaging techniques in the soft and hard X-ray regions [1–4]. In this talk, we describe the current status of the instrumentation developments at the soft X-ray (BL25SU) and hard X-ray (BL39XU) beamlines dedicated to the XMCD study. These beamlines offer the capability of fast helicity switching of the circularly polarized X-ray beam with the high-sensitive lock-in detection of dichroic signals. For magnetic imaging, BL25SU is equipped with a Fresnel zone plate for a scanning soft-XMCD microscopy under strong magnetic fields by a unique combination with an 8-T-superconducting magnet [2, 3]. BL39XU is equipped with a Kirkpatrick-Baez mirror setup for scanning hard-XMCD microscopy with the spatial resolution of 100 nm [4]. We present some recent results including XMCD study of voltage-induced magnetic anisotropy in transition metal/MgO junctions [5, 6], time-resolved XMCD microscopy in a Co/Pt multilayer micro-disk to study the spin precession dynamics in the non-linear regime of ferromagnetic resonance [7], and three-dimensional observation of internal magnetic domains using scanning hard-X-ray magnetic microtomography [8].

References
Recent Developments at the Resonant Scattering and Diffraction
Beamline P09 at PETRA III at DESY

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Beamline P09 at PETRA III at DESY allows for state of the art resonant X-ray elastic scattering (REXS) experiments on strongly correlated and magnetic materials in high magnetic fields and at low temperatures in a wide range of X-ray energies covering the L_{III},L_{III} absorption edges of the 3-5d transition metals and 4f lanthanides, and the M_{IV} and M_{V} absorption edges of the 5f actinides [1,2]. Since 2016, the beamline is equipped with new phase retarder plates allowing for full polarization control between 2.7 and 13.2 keV. We have further worked at extending the experimental capabilities so as to enable: (a) magnetic X-ray scattering at low temperatures in the tender X-ray range (b) magnetic X-ray scattering at high pressure and low temperatures and (c) X-ray magnetic circular dichroic experiments on powders and on thin films [3]. As well, the development of a crystallographic computing software analysis tool MagStREXS has been initiated to help the REXS user community with magnetic structure determinations from resonant X-ray scattering data. Hereby I will present the instrument, a snapshot of the recent developments and related future plans and some of the scientific results obtained so far on selected compounds.

References
Ultra-Low Temperature (ULT: bellow 1 Kelvin) is of primary importance when dealing with new states of matter and the studies of magnetic phenomena. On the DEIMOS beamline at SOLEIL synchrotron a new ULT refrigerator dedicated to soft X-ray Magnetic Circular Dichroism (XMCD) allows now users to perform experiments down to 220mK.

Preliminary experiments with an ErPd alloys and Fe₄ molecules demonstrate the outstanding performances of this new set-up, in terms of lowest achievable temperature under x-rays, the precision in temperature control over 4 orders of magnitude, the speed of the cooling down/warming up procedures and the strongly reduced eddy current power allowing fast scanning of the magnetic field during XMCD measurements.

In this presentation I will describe the modifications made to the 7 teslas cryo-magnet used on the DEIMOS beamline, to make it compatible with ULT. I will also present the thermometric measurements made to calibrate the temperature at the sample place.

Figure 1: CAD of the cryostat. In the insert XMCD detected magnetization curves for of a monolayer of Fe₄ molecules in the 350-900 mK range.
Hard XMCD under Extreme Conditions

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ESRF ID12 is a beamline dedicated to polarization dependent X-ray absorption spectroscopy in the photon energy range from 2 to 15 keV. A large part of scientific program at ID12 concerns use of X-ray Magnetic Circular Dichroism to unravel the microscopic origin of magnetism in a large variety of materials: ferro- ferri- and paramagnets. Over the years many efforts have been devoted to develop specific instrumentation to perform XMCD under multiple extreme conditions of magnetic field, temperature and pressure. Nowadays, users can routinely perform XMCD studies on different samples like ultrathin films, nanoparticles or bulk crystals under magnetic field as high as 17T magnetic field and temperatures down to 2.1 K [1]. In the last 5 years, 8 Tesla solenoid with RT bore was equipped with a specially designed anvil cell allowing XMCD measurements under pressures up to 60 GPa and temperatures down to 2.7 K [2]. More recently, we have demonstrated that hard XMCD measurements on paramagnetic molecular systems can be performed at temperatures as low as 500mK [3]. The performance of the beamline will be illustrated with a selection of some prominent achievements in the field of hard XMCD under multiple extreme conditions. Finally, challenges and scientific opportunities open with the new EBS source will be discussed.

References
BM23 and ID24 are two ESRF beamlines dedicated to X-ray Absorption Spectroscopy. BM23 is a bending magnet energy-scanning beamline dedicated to high quality EXAFS and XANES. Magnetic materials are being routinely investigated, with focus on superconductivity, nanomaterials or magnetoelectricity for example. XAS sheds light on the evolution of the electronic state and local structure with parameters like doping, temperature, pressure or magnetic field. ID24 is an energy-dispersive XAS beamline installed on a high β straight section equipped with four undulators. Using Quarter Wave Plates, polarization dependent studies such as X-ray Magnetic Circular Dichroism (XMCD) can be performed. A large part of ID24’s scientific activity is dedicated to study materials under extreme conditions.

Pressure directly acts on interatomic distances, allowing to tune both bandwidths and bandgaps, thus possibly inducing correlated magnetic and structural instabilities. In Fe, Co and Ni magnetism arises from the partially filled spin-polarized 3d band, whose properties are strongly determined by the crystal structure and by external factors such as temperature, magnetic field, and pressure. Therefore, exploring the stability limits of ferromagnetism as a function of these thermodynamical variables is an essential issue to get a deeper insight on its appearance [1].

As applying pressure, coupling XAS to a high magnetic field is a powerful tool to investigate the correlation between structural, electronic and magnetic degrees of freedom. On ID24, since 2005, we are developing a setup to measure XAS and XMCD under high pulsed magnetic fields. Recently, we investigated sublattice magnetization processes in ferrimagnetic Er₃Fe₅O₁₂ garnet [2] and in the rare earth transition metal intermetallics HoFe₅Al₇ [3] at the Fe K-edge and at the rare earth L-edges in pulsed fields up to 30 T.

For ID beamlines, the EBS will provide a factor 30 reduction in emittance. The EBS project offers opportunities (brilliance increase) that are difficult to exploit with the ED-XAS spectrometer. We thus propose an evolution of ID24 with the following aims: I) Convert one ED-XAS branch into a branch equipped with a scanning spectrometer dedicated to high brilliance XAS. II) Optimize EDXAS-L for dedicated applications where the ED spectrometer demonstrates a clear advantage, like pulsed magnetic field, applications.

References
Modular Approaches to Magnetic Materials of 4d, 5d, and 5f Metal Ions

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Diffuse orbitals and large magnetic anisotropy, resulting from strong spin-orbit coupling, make complexes with central ions from the heavier transition element periods interesting modules for magnetic materials. In this talk, the use of [MF₆]²⁻ complexes of the 4d, 5d and 5f series as modules for molecular magnetic systems of various dimensionality will be presented [1,2,3,4]. It will further be shown how X-ray spectroscopy, and in particular, X-ray magnetic circular dichroism may provide novel insight into the magnetic properties of such molecule-based materials.

References
Cyanido-Bridged Fe/Co Pairs: How to Make Them and How to Study the Switching Mechanism with XAS and XMCD Spectroscopies

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The rational design of molecular systems, which exhibit switchable physical properties as a function of external stimuli ($T$, $E$ or $H$, $hv$, $P$, etc.) is a subject of the intense research activity to conceive high-performance molecule-based electronic devices [1-2]. Over the past decade, chemists have investigated the synthesis of magnetic and photoresponsive complexes through rational choices of cyanido-based building blocks. This approach has been extremely successful, and various molecular architectures have been obtained with remarkable properties such as spin crossover, [3] electron-transfer process [4-5], and photoinduced magnetism [6].

In our quest to obtain new molecular systems and concomitantly to reduce the size of these functional molecules, we designed dinuclear complexes using a building block strategy. Some of them are exhibiting switchable optical and magnetic properties in solution and/or in solid state [7-8].

In the second part of this presentation, the thermally and light induced electron transfer of the dinuclear complex $[(\text{T}p)\text{Fe}^\text{III}(\text{CN})_3\text{Co}^\text{II}(\text{PY}5\text{Me}_2)]^+$ ($\text{PY}5\text{Me}_2 = 2,6\text{-bis}(1,1\text{-bis}(2\text{-pyridyl})\text{ethyl})\text{pyridine}$; $\text{T}p = \text{hydridotris}(\text{pyrazol-1-yl})\text{borate}$), has been studied using a combination of bulk structural, spectroscopic, magnetic and photomagnetic studies. Moreover, the switching mechanism followed at the local level by X-ray Absorption Spectroscopy (XAS) and X-ray Magnetic Circular Dichroism (XMCD) measurements at Fe and Co L$_2$,3 edges will be presented [9].

References

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Exploring the Magnetism of Molecules at the Nanoscale using Synchrotron and Scanning Probes-Based Techniques

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The next generations of devices for spintronics[1,2] and quantum computing[3,4] will be based on molecular and inorganic nanostructures with peculiar magnetic properties. The development of these devices requires the know-how to properly assemble each building blocks and, primarily, to control the magnetism of these objects at the nanoscale. It is thus fundamental to adopt specific characterization tools featuring the required sensitivity to study these materials at the nanoscale and to make correlations between the observed behavior and the chemical nature and structural arrangement of each component in the hybrid architectures.

An overview of this approach and recent results obtained on molecular systems will be provided evidencing how X-ray circular magnetic dichroism (XMCD) experiments and other spectroscopic tools based on large scale facilities may lead to fundamental steps forward in this demanding exploration by directly accessing to static and dynamic magnetic properties ultra-thin deposits of molecular layers[5] as well as to carefully evaluate the role of different substrates that may alter also the behavior of the molecular species. In parallel scanning probe microscopies operated analogously at cryogenic temperatures may allow to explore the individual nanostructures constituted by single molecules and arrays of molecules. The comparison and the combination of XMCD results achieved on a large area with the local properties extracted from the analysis of the data achieved using Inelastic Energy Tunneling Spectroscopy (IETS)-STM[6,7] and Magnetic Force Microscopy (MFM)[8] can now pave the way for prototyping hybrid nanodevices embedding these objects with an increased knowledge of their magnetism.

References

“Spin Injection” in Molecule-Based Materials toward Conducting Magnets

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The magnetic properties of a complex or a material usually result from cooperative effects between the magnetic spins. The choice of the linker between the spin carriers is therefore a crucial element to control, as it mediates the communication and interactions between them. The use of a redox-active bridging ligand as a linker is a particularly attractive strategy. By oxidation(s) or reduction(s), it can act as a control switch of the magnetic interactions. While in its diamagnetic state, it mediates usually weak magnetic interactions, in its radical form, it can promote a better spin delocalization inducing large magnetic interactions and in the same time, a good electronic conductivity which could lead to new high $T_C$ conductive magnets. In this presentation, the design of new magnetic systems based on redox-active ligands will be discussed and illustrated by (i) dinuclear M(II) complexes, $[\text{M}_2(\text{tphz})(\text{tpy})_2](\text{PF}_6)_4$ (Left part of the Figure below; M = Co or Ni; tpy = terpyridine; tphz = tetrapyridophenazine) and (ii) a two-dimensional coordination network, $\text{Cr(}\text{pyrazine})_2\text{Cl}_2$ (Right part of the Figure below).[1,2] The electronic and magnetic properties of these systems were described using different physical characterization techniques including X-ray spectroscopy (XAS and XMCD) in the hard X-ray range.

![Figure](image.png)

Figure Left: view of the molecular structure of the $[\text{Co}_2(\text{tphz})(\text{tpy})_2]^{4+}$ complex. Right: view of the 2D structure of $\text{Cr(}\text{pyrazine})_2\text{Cl}_2$

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References:
Magnetism with Nuclear Resonance Scattering

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With the Extremely Brilliant Source Upgrade Programme, Nuclear Resonance beamline [1] ID18 of the European Synchrotron Radiation Facility (ESRF, Grenoble, France) will allow users to study magnetism with a spatial resolution of ~100 nm.

Figure 1. The double-stage diamond anvil cell for ultra-high pressure generation [2]. Image courtesy Elena Bykova, University of Bayreuth.

Figure 2. Diamond with a ferropericlase inclusion (left). Mössbauer spectrum (right) suggesting the presence of magnetic splitting (from [3]).

In this talk, we will consider applications of nuclear resonance scattering for studies of magnetism in nano-scale systems. This includes ultra-high, in TPa range, pressures using novel high-pressure instruments as double-stage diamond anvil cell (DAC, Fig.1), studies of magnetism with tiny samples as sub-micron iron-bearing inclusions in diamonds (Fig. 2), the nano-scale landscape of magnetic avalanches in superconducting samples in experiments similar to Ref. 4 (Fig. 3), and some other challenging applications enabled by coming development of the ESRF.

Figure 3. Tin foil, surrounded by compressed H$_2$S, is located in a DAC at a pressure of about 153 GPa. Synchrotron radiation excites the nuclei of the Mössbauer isotope $^{119}$Sn (left). At high temperature, nuclear forward scattering shows quantum beats (right) due to magnetic splitting of the nuclear levels (from [4]).

References
Magnetic Transitions in $\text{Fe}_2\text{O}_3$ at High Pressures: Magnetism in the Earth’s Mantle

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The Earth’s crust-mantle boundary (Moho discontinuity) has been traditionally considered as a fundamental boundary between the magnetic crust and the nonmagnetic mantle. Nevertheless, this assumption has been questioned recently by geophysical studies and by the identification of magnetic remanence in mantle xenoliths [1,2], which suggest deep magnetic sources. Owing to their high critical temperatures, iron oxides are the only potential sources of magnetic anomalies at mantle depths [3]. However, the lack of data on their magnetic properties at relevant pressure-temperature conditions hampers any conclusive result about the Curie depth, below which mantle temperature is too high to sustain any kind of long-range magnetic order.

Here I report investigations of the magnetic transitions and critical temperatures in $\text{Fe}_2\text{O}_3$ polymorphs [4] to 95 GPa and 1200 K by means of Synchrotron Mössbauer Source spectroscopy [5] in laser-heated diamond anvil cells. The experiments were conducted at ID18 beamline of the ESRF. Our results demonstrate [6] that hematite, $\alpha$-$\text{Fe}_2\text{O}_3$, remains magnetic at the depth of the Earth’s transition zones along cold or very cold subduction paths in the West Pacific region. The geophysical implications of these findings will be discussed.

References
Who cares about Magnetism at Extreme Conditions?

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Synchrotron radiation and the ESRF-EBS project in particular is highly suited to exploring the magnetic properties of materials at extreme conditions, for example at high pressure. Samples are generally small, only a few tens of microns or less in diameter, and may contain heterogeneities that provide additional clues to their history or behaviour. Temperature can also be varied, either raised or lowered, to further explore the extreme conditions realm. Nuclear resonance offers unique possibilities to probe magnetism at extreme conditions through its high sensitivity to the alignment of electron spins. The nuclear resonance beamline ID18 at ESRF offers a number of techniques that can be applied at extreme conditions, including nuclear forward scattering and its energy domain equivalent, synchrotron Mössbauer source spectroscopy. So who cares about such possibilities? The presentation will explore many of the answers to this question that span research fields from theoretical physics to astrophysics to geophysics as well as practical aspects to consider when planning experiments.
Study of Spin Reorientations by Nuclear Forward Scattering 
in Fe and Ir Containing Compounds

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One of the interesting properties of the Mössbauer spectroscopy (MS), as well as its time analogue Nuclear Forward Scattering (NFS) of Synchrotron Radiation (SR), is the dependence of the cross-section on the nuclear spins orientation relative to the X-ray beam direction and polarization. This feature allows for measurements of the type and direction of the magnetic ordering in the single crystals or multilayer structures. However, such studies are rare with MS due to the typically small size of the single crystals. On the other hand, due to the high brilliance of the SR, NFS is appropriate method for such investigations as shown in the talk with several examples.

The first example is the study of the spin reorientations occurred in the Fe-superconductors, like TlFe\textsubscript{1.6}Se\textsubscript{2} [1]. Here, the orientation of the Fe magnetic moments shows temperature driven transitions between $c$-axis and $ab$-plane directions as revealed by NFS and by the magnetic neutron scattering.

Another example is the coupling of the Eu and Fe magnetic moments orientations in the compounds EuFe\textsubscript{2-x}Ni\textsubscript{x}As\textsubscript{2} as seen by NFS on Fe and Eu. Here, the orientation of the Eu magnetic sublattice jumps from $ab$-plane to the $c$-axis when the ordering of the Fe moments disappears.

The last example is the study of the metamagnetic transition in Sr\textsubscript{2}IrO\textsubscript{4} via NFS on Ir [2]. Here, using unusual properties of the nuclear transition on $^{193}$Ir, it becomes possible to obtain not only average direction of the spins orientation, but also characteristic dispersion for the spins distributions, that help to investigate the nature of the transition.

References
Theoretical Description of X-ray Experiments on Magnetic Materials out of Equilibrium

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Representing the electronic structure of an investigated material in terms of its electronic Green function is a well established and flexible starting point to calculate its X-ray absorption and optical spectra [1]. With very few exceptions, this scheme was used so far only to deal with materials in their equilibrium ground state. We present extensions of this scheme in two directions by dealing with a steady-state out-of-equilibrium situation as well as explicitly time dependent pump-probe experiments.

In the first part of the contribution we consider the case of XAS/XMCD investigations in multilayer systems subjected to a finite static electric field [2]. Our computational framework is based on the spin-polarized relativistic multiple scattering or SPR-KKR (Korringa Kohn Rostoker) Green function scheme. This approach has been extended by various authors to the steady-state out-of-equilibrium situation described by a corresponding energy dependent non-equilibrium Green function (NEGF) [3]. The NEGF has been used so far primarily to deal with transport properties. Here we demonstrate its use for the investigation of spectroscopic properties by calculating the electric field induced changes in the XAS- and XMCD-spectra of the Pd L_{2,3}-edges in a Co/Pd bilayer system. A corresponding extension of the XMCD sum rules allows a comparison of field induced moments as seen by XAS-spectroscopy and calculated directly on the basis of the NEGF. An extension of the scheme to deal with the spin-pumping experiments of Van der Laan and coworkers [4] is briefly discussed.

Recent developments in time-dependent density functional theory (TD-DFT) paved the way towards investigating and quantitatively interpreting, on an ab initio level, the ultrafast demagnetization processes in ferromagnetic systems caused by a strong laser pulse [5]. These time-dependent phenomena can be monitored by time-resolved spectroscopic techniques such as for example angle-resolved photo emission (ARPES) or magnetic circular X-ray dichroism (XMCD), with the latter having the additional advantage of being element-specific. A theoretical description for a time-dependent ARPES experiment has been recently worked out and implemented by the authors on the basis of the one-step model of photo emission [6]. The corresponding time evolution of the investigated system due to a pump pulse is described by means of the two-time Keldysh NEGF that is evaluated by means of the SPR-KKR Green function method. In this contribution a corresponding description for pump-probe XMCD experiments will be presented that makes use of the same theoretical framework. In addition, when compared to the previous ARPES investigations [6], the time dependence of the single-particle potentials is taken care by means of self-consistent TD-DFT calculations [5]. It should be stressed that the Keldysh NEGF formalism allows in addition to account for the impact of various relaxation mechanisms represented by a corresponding complex and energy-dependent self-energy. First numerical results for XMCD spectra at the L_{2,3}-edge of ferromagnetic Co exposed to a strong pump laser pulse that are based on a time dependent potential and using so far a quasi-static standard description of the XMCD experiment [1] will be presented.

References
We present formalism for calculating resonant inelastic x-ray spectra (RIXS) within the density functional band structure approach implemented recently in the PY LMTO computer code. The implementation allows studying photon momentum and polarization dependence of RIXS spectra of magnetic as well as non-magnetic compounds.

Ir $L_3$ RIXS spectra calculated for Na$_3$Ir$_3$O$_8$ and Li$_3$Ir$_3$O$_8$ show good agreements with experimental data and allow to identify transitions from occupied Ir $d$ states to unoccupied $j_{eff}=1/2$ and $e_g$ states.

Band structure RIXS calculations for SrRu$_2$O$_6$ cannot describe contributions to experimental Ru $L_3$ spectra coming from collective magnon excitations or localized intra-ionic spin-orbital ones. On the other hand, the calculations allow to distinguish these contributions from electron-hole continuum which is reasonably well described using the band structure approach [1].

References
Magnetic Surface Resonant X-ray Diffraction

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Bulk magnetic resonant diffraction is known and has been used for many years. It has been applied to study spin and orbital moments in archetypical systems. Using multipolar expansion, it has also allowed the probing of higher order terms such as the toroidal or anapole moment present in specific classes of materials.

On the other side, surface non-resonant diffraction is extensively used to probe the structure of surfaces and ultra-thin films. Experiments for surface resonant diffraction are not yet very common, but they have already given very interesting results, at electro-chemical interfaces or for ultra-thin metal-oxide films [1]. It is then its sensitivity to the projected density of state of the resonant atoms which is used to get information nearly unreachable by any other techniques. In this context we have recently developed the ab initio simulation of this spectroscopy and are able to calculate both the crystal truncation rods and the spectra taken on different points of the truncation rods [2].

We propose now a new extension of our procedure to magnetic systems. This work is prospective because data for magnetic surface resonant diffraction are quite rare and not compared with simulations. Nevertheless, the new capacities at the ESRF must make this techniques reachable.

We go to present in different examples, how the simulations are down and what sensitivity can be expected at the surface of magnetic systems.

References
Electronic and Magnetic Character of UTe$_2$ Unconventional Superconductor

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The interplay between ferromagnetism and superconductivity is a challenging problem in the coupling between the two major states of condensed matter. Recently discovered UTe$_2$ superconductor with $T_c = 1.7$ K [1,2] had originally been suggested to provide a new phase of superconducting matter in which half of the electrons become superconducting and half remain normal (thus with Fermi surfaces). In more recent studies down to 50 mK [3] strong evidence was presented for $p$-wave triplet pairing with point nodes. This behavior brings to mind the U-based ferromagnetic superconductors, with the difference that no long-range magnetic order has been observed in UTe$_2$ down to 25 mK.

We report density functional theory plus Hubbard U calculations for UTe$_2$ superconductor. We make a comparison between the electronic structure and spin and orbital magnetic properties of UTe$_2$ and UGe$_2$. We show that the Fermi surfaces display low dimensional features, reminiscent of the UGe$_2$ [4].

References
X-ray Magneto-Chiral Dichroism

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Systems, in which fundamental symmetries of nature are broken, play a fascinating role not only in physics but also in chemistry and in life sciences. A symmetry breaking manifests itself in various optical phenomena and therefore interaction of light with matter is the most powerful tool to deepen our understanding of complex matter. Two most vivid examples are magneto-optical and natural optical activity. Although the underlying physics is fundamentally different, the two phenomena manifest themselves quite similarly in homogeneous media: a rotation of the polarization plane of light or a difference in absorption of circularly polarized light (circular dichroism). This resemblance has motivated many eminent scientists, starting from Pasteur himself, to look for a link between magnetism and optical activity, but in vain. The corresponding optical manifestation is magneto-chiral effect which was first predicted in 1962 [1]. It may only occur in systems where both inversion and time-reversal symmetries are simultaneously broken. These symmetry conditions are satisfied in magneto-electric media and chiral molecular magnets. The striking feature of this effect can be derived from the symmetry considerations that it is a property of unpolarized light. The first experimental evidence of the magneto-chiral effect has been given only in 1997 by G. Rikken and E. Raupach [2] using visible light. In the X-ray range, the effect was discovered soon after in magnetoelectric Cr$_2$O$_3$ single crystal at ID12 beamline following the discovery of natural optical activity in the X-ray range [3].

A fascinating interplay of magnetism and structural chirality is reported here with two illustrative examples. The first one deals with the archetypical Single Chain Magnets M(hfac)$_2$NITPhOMe where M is 3d transition metal ions (Mn$^{2+}$ or Co$^{2+}$) [4]. For the first time, all the three dichroism spectra (X-ray magnetic circular dichroism (XMCD), X-ray natural circular dichroism (XNCD) and X-ray magnetochiral dichroism (XM$\chi$D)) have been measured at the K-edges of transition metals. It was found that the amplitude of magnetochiral effect in the X-ray range is linked to an orbital magnetic moment carried by absorbing atom.

The second example concerns more recent detection of XM$\chi$D signal in a paramagnetic lanthanide coordination complex, namely, Na$_5$[Ho(ODA)$_3$](BF$_4$)$_2$(H$_2$O)$_6$ at low temperatures and under high magnetic field [5]. The magnetochiral dichroism signal was found to be surprisingly weak despite a large orbital moment carried by Ho$^{3+}$ ion. This is due to a very weak hybridization of the strongly localized 4f states of the lanthanides which are responsible for magnetism in these ions.

References

Recent and Future Developments at the Soft X-ray Spectroscopy Beamline ID32

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ID32 was a phase 1 upgrade beamline with a scientific focus on magnetism and electronic structure using soft X-ray spectroscopy [1]. It started operation at the end of 2014 on the XMCD (X-ray magnetic circular dichroism) branch and on the RIXS (resonant inelastic X-ray scattering) branch in mid-2015.

The XMCD branch offers sophisticated sample preparation facilities attached to a UHV 9T superconducting fast sweeping (up to 8T/minute) magnet [2]. There is also a 4T perpendicular field, which allows XMLD (X-ray magnetic linear dichroism) experiments to be carried out - figure 1a.

The RIXS branch has very high energy resolution (~30meV at 930 eV), a 4-circle goniometer and a continuously variable scattering arm (50-150 degrees) under UHV vacuum - figure 1b. There is also the possibility of measuring the polarisation of the scattered X-rays [2].

Figure 1:  a. The left hand panel shows the XMCD experimental area with the 9T magnet.
            b. The Right hand panel shows the RIXS end-station and scattering arm.

Recent results illustrating some of the new capabilities of the beamline will be presented. In addition, the improvements planned during the EBS shutdown will be described.

References
List of Posters

- **I. de Moraes**: Towards Model Hard-Soft Magnetic Nanocomposites Produced by Nano-Fabrication

- **K.V. Frolov**: Some Problems for X-ray Spectroscopy of Iron-Containing Magnetic Materials

- **O.A. Golovnya**: Peculiarities of Development of High-Coercivity State of Sm-Co-Fe-Cu-Zr Magnets upon Step Cooling

- **V. Kolesnikova**: Magnetic Properties of Amorphous and Multiphase Microscale Wires

- **J. Mardegan**: Pressure Effects on the Magnetic Superconductor Eu(Fe_{0.88}Ir_{0.12})_2As_2

- **C. Paleo**: Co Nano-Cluster Inclusions in L10-FePt Matrix as a Model System of Nanocomposite Magnets

- **S. Shevyrtalov**: Martensitic Transformation Features of Ni-Fe-Ga-Ge Ferromagnetic Microwires

- **N.F. Shkodich**: High-Entropy CuCrFeTiNi Alloy Produced by High-Energy Ball Milling and Spark Plasma Sintering: Structural and Magnetic Characterization

- **K. Sobolev**: Magnetic Properties of Highly Pure (Cr_{1-x}Mn_x)_{2}AlC MAX-Phase

- **M. Spasova**: Magnetic Properties of High Entropy CoCrFeMnNi Alloy Prepared by High Energy Ball Milling

- **B. Tomasello**: Correlated Quantum Tunnelling of Monopoles in Spin Ice
Towards Model Hard-Soft Magnetic Nanocomposites Produced by Nano-Fabrication

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Permanent magnets (PM) are key for the development of green energy technologies and robotics. The ideal PM, with a high energy product, \((BH)_{\text{max}}\) combines high coercivity \(H_c\) and a high remanence \(M_r\). Following the discovery of hard magnetic properties in Nd-poor alloys of approximate composition \(\text{Nd}_{4}\text{Fe}_{78}\text{B}_{18}\) [1], the concept of hard-soft exchange-spring nanocomposites was developed [2], in which a magnetic phase, known to have soft magnetic properties in the bulk, reveals hard magnetic behaviour as a result of interfacial coupling at the nanoscale with a hard magnetic phase. While several fabrication methods have been used to produce hard-soft nanocomposites [3], the magnetic properties achieved so far, do not match expectations, as the size of the soft features is not sufficiently controlled. In this work, we report on the use of nanofabrication to produce model hard-soft nanocomposites with ultimate control of the nanoscaled soft phase.

Elongated Co and FeCo nano-rods (thickness = 10 nm, width = 20-100 nm, length = 200-400 nm and inter-rod distances of the order of the width), capped with a 3nm layer of Au, were produced by e-beam lithography and evaporation. The Au layer was then etched, and a hard magnetic layer (FePt) was subsequently deposited on top of the nano-rods. The hard layer was patterned so as to minimize the surface of the hard phase not covering nano-rods. Samples of large surface area (6 mm²) of arrays of soft nano-rods alone as well as such arrays embedded in a hard matrix have been fabricated for VSM-SQUID and XMCD measurements. The magnetic properties will be related to characteristics of the nanostructures, derived from SEM and TEM imaging.

Figure 1: (a) Schematic diagram of soft nanorods (thickness t=10 nm) covered by a hard magnetic layer (t=50 nm) and TEM cross-section image. (b) SEM images of an array of soft nano-rods (main image) and such an array covered with a hard-magnetic patch (zoom). (c) Nano-rod arrays with different feature size.

References
Some Problems for X-ray Spectroscopy of Iron-Containing Magnetic Materials

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Iron is one of the most common elements. The constantly high interest of researchers in iron compounds is observed in various fields of physics, chemistry, biology and medicine. And primarily this is due to the variety of magnetic properties observed in these compounds, for example, in a new multiferoics, in particular, iron-containing langasites of the $A_3M\text{Fe}_3\text{X}_2\text{O}_{14}$ (\(A = \text{Ba, Sr}; M = \text{Sb, Nb, Ta}; X = \text{Si, Ge}\)) family and rare-earth ferroborates $R\text{Fe}_3(\text{BO}_3)_4$ (\(R = \text{Y, La - Lu}\)), as well as magnetic nanowires (diameter 30 - 500 nm) of solid solutions of iron and other 3d metals.

Multiferroics showing simultaneously ferroelectric and magnetic ordering, are of especial interest from the point of view of application in prospective spintronic and magnetooptical devices, and also in devices for recovery of the electric energy and the systems of alternative energetic. Nanowires showing strong magnetic anisotropy and complex magnetic dynamics may find application for magnetic sensors, spintronic devices, hydrogen fuel cell electrodes, and for biomedical technologies, including antitumor therapy.

We assume that the use of the new capabilities of the upgraded ESRF beamlines will allow obtaining new unique experimental data in our studies of iron-containing langasites, rare-earth ferroborates and magnetic nanowires presented in this report.

This work is supported by the Ministry of Science and Higher Education within the State assignment FSRC «Crystallography and Photonics» RAS and Russian Foundation for Basic Research (Project No. 17-02-00766).
The Sm-Co-Fe-Cu-Zr magnets have the highest known maximum energy product \((BH)_{m}\) together with high thermal stability of magnetic hysteresis properties. The coercivity \(H_c\) originates from the pinning of domain walls at interfaces of the \(\text{Sm}_2(\text{Co,Fe})_{17} (R3m - 2:17)\) and \(\text{Sm}(\text{Co,Cu})_{5} (P6/mmm - 1:5)\) phases of the nanocrystalline cellular structure, which is formed via the precipitation of the high-temperature-solid solution at 800-850°C [1]. The subsequent re-distribution of the alloy components between the 2:17 and 1:5 phases in the course of step cooling from 800 to 400°C increases coercivity \(H_c\) of the magnets. This work studies with XRD analysis of peculiarities of high-coercivity state development in the high temperature (HTPM - \(\text{Sm}(\text{Co}_{0.76}\text{Fe}_{0.12}\text{Cu}_{0.09}\text{Zr}_{0.03})_{7.0}\)) and high energy (HEPM - \(\text{Sm}(\text{Co}_{0.64}\text{Fe}_{0.28}\text{Cu}_{0.06}\text{Zr}_{0.02})_{7.57}\)) permanent magnets in the course of the step annealing. The main increase of \(H_c\) in the HEPM (which has about 15 vol. % of the 1:5 phase) occurs after annealing at \(T = 700°C\) (Fig. 1a). The quenching of the magnet from 830°C results in a large misfit of the \(c\) lattice parameters of the coherent 2:17 and 1:5 phases, as well as increased stresses at the interface. The Cu diffusion from the 2:17 into the 1:5 phase upon annealing at \(T = 700°C\) leads to the relaxation of the stresses (Fig. 1d). At \(T < 700°C\), \(H_c\) grows negligibly (Fig. 1a). The volume fraction of the 1:5 phase in the HTPM quenched from 830°C reaches 50%; and the difference in the \(c\) lattice parameters of the 2:17 and 1:5 phases is negligible (Fig. 1e). Therefore, the Cu redistribution at \(T = 700\) and 600°C is slow and insignificant. The annealing at 500°C considerably accelerates the Cu diffusion and increase of \(H_c\), especially, this is true for the initial step of annealing \((t < 0.2 \text{ h})\). As is shown in [1], interface stresses grow upon annealing at the temperature lower than the Curie temperature of the 1:5 phase (~ 550°C). The lattice extension of the 1:5 along the \(c\) axis originating from the magnetic elasticity increases the misfits (Fig.1 f) and stresses. The Cu-enrichment of the interface of the 2:17 and 1:5 phases relaxes the stresses and increases the boundary-energy gradient, as well as \(H_c\).

![Figure 1: Coercivity kinetics (a,b) and fragments of x-ray diffraction patterns taken from the planes perpendicular to the texture direction of the HEPM (c, d) and the HTPM (e, f).](image)

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References
Magnetic Properties of Amorphous and Multiphase Microscale Wires

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Microscale magnetic wires with cylindrical symmetry possess unique magnetic properties due to the manufacturing process [1]. Among the advantages, the microwires have tunable magnetic properties both through varying the initial technical parameters and through varying magnetoelastic and magnetostatic anisotropy after the production process. As a result, ferromagnetic microwires have applications in security control and coding systems, sensitive sensors of magnetic fields, mechanical stresses, temperatures, deformation, as well as in microelectronics and medical applications [2, 3]. The applications mostly based on materials with an amorphous structure. These materials are difficult to be studied with X-ray techniques. At the same time, it has been shown recently that presence of several magnetic phases also has a potential application interest [4, 5]. The understanding of magnetic interactions in the amorphous and multiphase magnetic wires will help to increase efficiency of related devices based on such samples. In this work, a fundamental question on magnetic interactions in the amorphous and multiphase microwire is considered.

The Fe-, FeCo- and Co-based microwires were studied. The structural properties were investigated by the XRD and HRTEM. The phase transformations were studied and analyzed by DSC. Two stages of the crystallization processes were detected for as-cast amorphous microwire and the three-step crystallization processes were detected for the wire with a partially crystalline structure in the as-cast state. Presence of different components in metallic nucleus poses the presence of different magnetic phases. The FORC-analysis (First Order Reversal Curve) was applied to detect the interaction between different magnetic phases and to trace its influence on the magnetization reversal process. This offered an opportunity to discuss the relationship between magnetoelastic and magnetostatic interactions, micromagnetic structure formation. For partially crystalline wires a positive magnetic interaction was detected due to the magnetostatic interaction between the clusters of crystallites. More complex interaction in systems appears with presence in structure additional clusters of crystalline inclusions which provides interesting multiphase behavior.

References
The observation of high-\( T_C \) superconductivity in FeAs based 122-type compounds (AFe\(_2\)As\(_2\)) has triggered an enormous scientific interest. Here, we investigate the rich playground among electronic, magnetic and structural properties of the Eu(Fe\(_{0.88}\)Ir\(_{0.12}\))\(_2\)As\(_2\) compound using x-ray absorption spectroscopy and single crystal diffraction techniques under high pressure and at room/low temperatures. The aforementioned Eu-based material orders ferromagnetic (FM) below \( T_{FM} \sim 17 \) K at ambient pressure, where small or complete absences of magnetic moments are observed at the Fe/Ir sites. Therefore the magnetism is dominated by the Eu\(^{2+}\) ions. In addition, macroscopic measurements surprisingly display a bulk superconductivity emerging below 22 K in which magnetism and superconductivity coexist at low temperatures.

Our X-ray absorption spectroscopy (XAS) measurements reveal that the Eu\(^{2+}\) ions have the magnetic state diminished followed by an enhancement of the amount of the non-magnetic Eu\(^{3+}\) ions. The collapse of the magnetic state starts arising around 3-5 GPa in which it can be seen as a delocalization of the 4f\(^7\) shell electrons. Consequently a charge transfer between the Eu magnetic ions and the dense orbital environment can be expected. In addition, we observe that around 15 GPa the x-ray magnetic circular dichroism (XMCD) is reduced drastically to 20 % in which the average oxidation state for the Eu ions is approximately +2.3.

To probe the lattice, high-pressure single crystal x-ray diffraction measurements were performed at room and at low temperatures. As observed in the XAS measurements, around 3-5 GPa a first transition from tetragonal (T) to collapsed-tetragonal (cT) is observed. This isostructural transition is mainly stimulated by the compression of the Eu ions. However, the cT phase is rapidly suppressed in which above 8-12 GPa the system transits to an orthorhombic (Or) phase with a strong temperature dependence. In this range of pressure and above, the Eu oxidation seems to be very close to the saturation value which makes the As-Fe/Ir-As and As-Eu-As layer distances play important role in the properties of the system. Consequently, the layer distance might directly affect the superconductivity and the magnetism in the system.

These results open up new possibilities for exploring how the structural, electronic and magnetic properties can be coupled to superconductivity in the 122 family.
Co Nano-Cluster Inclusions in L10-FePt Matrix as a Model System of Nanocomposite Magnets

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Nanocomposite magnets, consisting of a fine mixture between a large magnetocrystalline anisotropy phase and a large magnetization phase, is a promising route to develop new permanent magnets with unprecedented performances. Theoretical calculations predicted a potential energy product of 1 MJ/m\(^3\), which is twice as large as the one of best Nd\(_2\)Fe\(_{14}\)B magnets produced today [1]. But these calculations also pointed out the absolute necessity to confine the softer phase in nano-sized grains, typically smaller than 10 nm, which cannot be obtained with conventional process for mass scale material fabrication. So far only very few experimental works with encouraging results have been reported on such exchange-spring magnet [2]. Further experimental investigations are needed to assess the full potential of nanocomposite for designing high energy product magnetic materials.

We recently prepared Co@FePt transition metal (TM)-based nanocomposite films from low energy cluster beam deposition technique (LECBD) of Co clusters, \textit{in situ} embedded in FePt matrix independently produced by alternative electron gun evaporation on the same substrate [3,4]. This technique gives a fine control over the microstructure. The Co cluster inclusions can be selected in size prior their deposition, between 1 and 10 nm. The cluster to matrix volume ratio is adjusted controlling the thickness of each Co, Fe, Pt individual layers. Annealing is a crucial step to drive the initial Fe and Pt multilayers to the high-magnetic anisotropy L1\(_0\) phase with a limited diffusion of the clusters in the matrix. Specular X-rays Diffraction revealed a thermal transition to a chemically ordered L1\(_0\)-FePt alloyed matrix with a partial texture on Si substrate while environmental-TEM and EDX analyses allowed us to observe the partial diffusion of the Co clusters in the hard matrix.

Recently, X-ray absorption spectroscopy measurements (EXAFS, XLD and XMCD) were performed on annealed Co@FePt nanocomposite with various clusters concentrations and compared to equivalent multilayer samples at Fe and Co K-edges at BM30 and ID12 beamlines at the ESRF. The structural analysis proved that the Co clusters don’t entirely mix with the matrix during annealing contrary to multilayers. The study of such model system could give insights about the role of the nanostructure on magnetic hardness and could thus guide the development of mass scale synthesis processes.

This work is supported by the ANR collaborative project “SHAMAN”: Soft in HArMd MAgnetic Nanocomposites (2017-2020).

References
Here we report on the internal structure and magnetic properties in Ge-doped Ni-Fe-Ga rapidly quenched glass-coated microwires with the chemical composition of Ni$_{51.69\pm0.28}$Fe$_{26.97\pm1.09}$Ga$_{21.34\pm0.82}$ (SX0) and Ni$_{51.96\pm0.03}$Fe$_{23.75\pm0.14}$Ga$_{21.54\pm0.06}$Ge$_{2.75\pm0.11}$ (SX3). The diameter of an inner core of SX0 microwire has been determined as $d = (16.2\pm0.4)$ µm and SX3 microwire as $d = (12.0\pm0.2)$ µm. Glass coating thickness varied from 14 to 16 microns for both type of microwires.

The samples were cut by focused ion beam to perform cross-section measurements on SEM. Vertical lines on the image are the result of ion beam cutting. Bright areas show number of parallel lines from border to border of the microwire. Dark areas are situated close to the center of the sample and can be characterized by parallel lines but of different angle. The first type of lines are martensitic variants with the distance between the lines around 750-800 nm. The second type can also be classified as martensitic variant but of another phase. The cross-section of the second wire have the same features with additional bright areas (Fig. 1).

![Figure 1: SEM images of the cross-section of the SX0 (left) and SX3 (right) microwires](image)

X-ray diffraction measurements at room temperature showed that SX0 wires are in martensitic state and the gamma phase is absent. SX3 microwires demonstrate intermediate state with austenite and martensite phases, which means that substitution of iron on germanium leads to the shift of MT towards low temperatures. The measurements of magnetic properties confirmed the assumption and revealed the influence of germanium.
High-Entropy CuCrFeTiNi Alloy Produced by High-Energy Ball Milling and Spark Plasma Sintering: Structural and Magnetic Characterization

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Newly developed high-entropy alloys (HEAs) are receiving much attention for their unique structures and excellent properties [1-3]. Two groups independently proposed the study of HEAs containing multiple elements (at least 5) in equiatomic or nearly equiatomic concentrations (ranging between 5 and 35 at. %). These alloys are stabilized by the increase of the mixing entropy which is thought to suppress the formation of metallic phases and thus favors the formation of simple solid solutions with a fcc or bcc or bcc+fcc structures.

HEAs have been fabricated by several methods, including arc melting and casting, mechanical alloying, and laser cladding. Among these, especially promising seems to be high-energy ball milling (HEBM) in planetary ball mills that can yield stable microstructures and nanocrystalline alloys of better homogeneity compared to other non-equilibrium processes. In this communication, we report the first preparation of equiatomic CuCrFeTiNi HEA particles by short-term (30 min) HEBM and spark plasma sintering (SPS) and provide a structural and magnetic characterization.

Our structural and chemical analysis showed that micron sized particles of bcc CuCrFeTiNi consisting of nanosized crystalline grains (~6 nm) could be obtained after 30 min of HEBM. The HEA powders were thermally stable up to 500°C by DSC. The HEA powder was subsequently consolidated by SPS at 700°C resulting in a consolidated bulk HEA with co-existing bcc and fcc phases. The as-milled CuCrFeTiNi powder blend contained a solid solution with bcc (Im³m) structure. Annealing at 600°C (t=180 min) increased the crystallinity of the α-phase (bcc) and gave rise to formation of the γ-phase (fcc, Fm³m) whose amount grew with increasing dwell time. Between 800–1000°C, a tetragonal intermetallic σ-phase – most likely FeCr - appeared and subsequently vanished. At 1000°C, the final product was found to contain two solid solutions based on the γ-phase (fcc). The Vickers hardness $H_v$^{HEBM} = 7.7 GPa of the SPS consolidated CuCrFeTiNi alloy (milled for $t$ = 180 min) was markedly higher than the one of SPS-produced ones without HEBM ($H_v$ = 2.1 GPa). Paramagnetic behavior at room temperature with a small ferromagnetic contribution at low fields was observed for as-milled powder after 180 min of HEBM. A small magnetic hysteresis was observed at 5K and 300K with a coercive field of around 16 kA/m. Above 100K, the inverse susceptibility of a HEA powder ball-milled for $t$=240 min showed a clear paramagnetic response. The Curie temperature $T_C$ ~ 50 K was found.

References
Magnetic Properties of Highly Pure (Cr$_{1-x}$Mn$_x$)$_2$AlC MAX-Phase

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MAX-phases are the family of layered ternary compounds, with the common M$_{n+1}$AX$_n$ chemistry, where M is an early transition metal, A is an A-group element (mostly IIIA and IVA) and X is either C or N. MAX-phases possess the unique set of physical characteristics, combining metallic and ceramic ones which are high values of thermal and electrical conductivity, great oxidation resistance, easy machinability, tolerance to thermal shock and mechanical damages [1]. The major part of MAX-phases is paramagnetic, although the Cr$_2$AlC compound was anticipated to be an antiferromagnet [2]. Doping of Cr$_2$AlC MAX-phase with manganese on Cr-site was proposed to enhance the net magnetic moment of the compound [3]. However, due to the closeness of ferromagnetic (FM) and antiferromagnetic (AFM) states on an energy scale [4] and the poor quality of samples [5] it is still an actual goal to analyze the magnetic nature of (Cr$_{1-x}$Mn$_x$)$_2$AlC MAX-phase.

Samples of (Cr$_{1-x}$Mn$_x$)$_2$AlC MAX-phase with x = 0, 0.025, 0.05 and 0.1 were synthesized using the arc melting technique and characterized by means of XRD and SEM-EDX analysis. Magnetometry measurements were performed using SQUID and the vibrating sample magnetometer. Magnetization (M) versus temperature (T) and magnetic field (H) dependences were obtained in the range of T from 2 K to 350 K and H from zero to 7 T. When the fascinating feature of the canted AFM behavior was revealed by SQUID-magnetometry, the complex optimization procedure of the arc melting technique was performed to produce the highly pure phase of (Cr$_{1-x}$Mn$_x$)$_2$AlC. XRD and SEM-EDX analysis revealed the high quality of samples and the uniform distribution of Mn. Magnetic properties measurements showed the designated competition of FM and AFM interactions. These results provided deeper insight into the understanding of the magnetic nature of Cr-based MAX-phases and may be used for the further application-oriented investigations.

References
A novel approach to the fabrication of a new class of alloys - also known as high-entropy alloys (HEAs) was developed by Yeh et al. [1]. The HEAs containing at least 5 components in equiatomic or nearly equiatomic amounts (ranging between 5 and 35 at. %) are attractive in terms of mechanical, thermal, electrical and magnetic properties [2]. These HEAs are stabilized by the increased mixing entropy which suppresses the formation of binary and ternary metallic phases and thus favors the formation of solid solutions of many elements. HEAs have been fabricated by several methods, including arc melting and casting, mechanical alloying, and laser cladding. Among these, especially promising seems to be high-energy ball milling (HEBM) in planetary ball mills that can yield stable microstructures and nanocrystalline alloys of better homogeneity compared to other non-equilibrium processes [3].

We report the fabrication of CoCrFeMnNi HEA 80-120 µm particles by high-energy ball milling (HEBM) and provide their structural and magnetic characterization. Our XRD, SEM, and EDX results showed that a fcc CoCrFeMnNi solid solution with uniform distribution of the elements and refined microstructure of nanosized grains (~10 nm) could be obtained after 60 min HEBM. Magnetic studies reveal very complex behavior: spin glass magnetic response below 50 K; intrinsic exchange bias and vertical shift of the hysteresis loop after field cooling. Isothermal aging after quench down to the low temperature shows a typical features of the out-of-equilibrium dynamics of spin glass with slow relaxation processes. This presentation intents to instigate a discussion on X-Ray spectroscopy techniques capable to elucidate the intriguing magnetic phenomena of HEAs.

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References
Correlated Quantum Tunnelling of Monopoles in Spin Ice

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The spin ice materials Ho2Ti2O7 and Dy2Ti2O7 are by now perhaps the best-studied classical frustrated magnets. A crucial step towards the understanding of their low temperature behaviour – both regarding their unusual dynamical properties and the possibility of observing their quantum coherent time evolution – is a quantitative understanding of the spin-flip processes which under-pin the hopping of magnetic monopoles. We attack this problem in the framework of a quantum treatment of a single-ion subject to the crystal, exchange and dipolar fields from neighbouring ions. By studying the fundamental quantum mechanical mechanisms, we discover a bimodal distribution of hopping rates which depends on the local spin configuration, in broad agreement with rates extracted from experiment. Applying the same analysis to Pr2Sn2O7 and Pr2Zr2O7, we find an even more pronounced separation of timescales signalling the likelihood of coherent many-body dynamics.
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