19th SOLEIL USERS' MEETING

JANUARY 27th - 28th, 2025

Synchrotron SOLEIL (Saint-Aubin)

SCIENTIFIC COMMITTEE

Pierre ASSELIN (De la Molécule aux Nano-objets : Réactivité, Interactions et Spectroscopies - Paris)
Amélie BORDAGE (Institut de Chimie Moléculaire et des Matériaux d'Orsay - Orsay)
Florent CARN (Laboratoire de Matière et Systèmes Complexes - Paris)
Charlotte CATROUILLET (Institut de Physique du Globe de Paris - INSU - INEE – Paris)
Fabien CHEYNIS (Centre Interdisciplinaire de Nanoscience de Marseille - Marseille)
Héloïse DOSSMANN (Institut Parisien de Chimie Moléculaire - Paris)
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Simona RANERI (National Research Council - Institute of Chemistry of Organometallic Compounds – Sesto Fiorentino)
Asma TOUGERTI (Unité de Catalyse et de Chimie du Solide – Villeneuve d'Ascq)

Information and registration

SOLEIL SYNCHROTRON

SOLEIL Users' Meeting

January 27th - 28th, 2025

Synchrotron SOLEIL, Saint-Aubin - France

Summary

Welcome!

The 19th **SOLEIL Users' Meeting** takes place on **Monday January 27th and Tuesday 28th, 2025** at **SOLEIL**.
This invaluable forum for the synchrotron radiation users' community provides the opportunity to exchange and learn about the evolution of the machine and the beamlines.
It is also the occasion to share scientific, technical and practical issues about synchrotron radiation use.

This year, the Committee is offering a revised programme to address your feedback, including a reorganisation of the parallel sessions.

The **plenary talks** cover recent research highlights of SOLEIL in the following areas:

- *Cultural heritage*
- *Electronic and magnetic properties of matter*

Scientific communications are presented during **5 parallel sessions**, selected from the submitted abstracts:

- *Biology and health*
- *Diluted matter*
- *Geosciences / Environment / Cultural Heritage*
- *Chemistry & Soft Matter (Characterization of structure and properties of chemical materials, reactivity, catalysis, chemical surfaces and interfaces / Soft matter)*
- *Physics & Advanced Materials (Structural, electronic and magnetic properties of matter / Physical surfaces and interfaces)*

In the afternoon of January 27th, 2025 a **social time and scientific exchange are** held at SOLEIL, including the poster session, booths from commercial exhibitors and the dinner buffet.

Satellite and **tutorials** are also organised on site on Tuesday afternoon, the 28th of January, 2025:

- *Satellite: The New CryoEM – POLARIS*
- *Tutorial 1: The latest developments of FASTOSH*
- *Tutorial 2 : Surface diffraction - Data treatment and analysis*

Bienvenue !

Le **19ème Colloque des Utilisateurs de SOLEIL** se tient les **lundi 27 et mardi 28 janvier 2025**, à SOLEIL**.**

Ce rendez-vous incontournable pour la communauté des utilisateurs du rayonnement synchrotron est l'occasion d'échanges et d'informations sur l'évolution de la machine et des lignes de lumière.
Il est aussi le lieu pour échanger sur les aspects scientifiques, techniques et pratiques de l'utilisation du rayonnement synchrotron.

Cette année, le Comité propose un programme revisité en réponse à vos suggestions, incluant, notamment, une réorganisation des sessions parallèles.

Les **conférences plénières** couvriront les thèmes de recherche des communautés d'utilisateurs de SOLEIL suivants :

- *Patrimoine*
- *Propriétés électroniques et magnétiques de la matière*

Les **5 sessions parallèles** sont composées d'exposés scientifiques originaux, sélectionnées à partir des résumés soumis :

- *Biologie et santé*
- *Matière diluée*
- *Géosciences / Environnent / Patrimoine*
- *Chimie & Matière molle (Caractérisation de la structure and propriétés des matériaux chimiques, réactivité, catalyse / Matière molle)*
- *Physique & Matériaux avancés (Structure, Propriétés électroniques et magnétiques de la matière / surfaces et interfaces)*

Un **temps de convivialité et de discussion** est organisé à SOLEIL le lundi 27 janvier 2025 après-midi avec la session posters, les stands d'entreprises et le buffet dînatoire.

Un satellite et des tutoriels sont également organisés le mardi 28 janvier 2025 après-midi à SOLEIL :

- *Satellite : le nouveau CryoEM – POLARIS*
- *Tutoriel 1 : Les derniers développement de FASTOSH*
- *Tutoriel 2 : Diffraction de Surface – Traitement des données et analyses*

19th SOLEIL USERS' MEETING

27-28 January 2025

Programme

Monday, January 27th, 2025

SOLEIL Auditorium - Main Building

09:00 - 10:00	Registration & coffee
10:00 - 10:10	Welcome / Introduction, S2UO Chair – Amélie Bordage
10:10 - 10:50	The word of SOLEIL General Director – Jean Susini
10:50 - 11:35	**PLENARY TALK** – Cultural Heritage ***Jose Tapia*** *– Lab-BC UAR3506 CNRS-C2RMF-Chimie ParisTech PSL, Paris, France* Chemical characterization using a combination of in-situ, laboratory and Synchrotron-based technics of prehistoric coloring matter from the Franco-Cantabrian region. From Font-de-Gaume to Altamira
11:35 - 12:05	***The SOLEIL High Pressure Group*** *– SOLEIL Synchrotron, Saint-Aubin, France* High pressure in SOLEIL: Tools and science
12:05 - 12:30	*AFURS / S2UO / PRC*
12:30 - 13:45	*Lunch*
14:00 - 15:45	Parallel sessions – 5 different rooms • Biology and Health • Chemistry & Soft Matter (Characterization of structure and properties of chemical materials, reactivity, catalysis, chemical surfaces and interfaces / Soft matter) • Diluted matter • Geosciences / Environment / Cultural Heritage • Physics & Advanced Materials (Structural, electronic and magnetic properties of matter / Physical surfaces and interfaces)
15:45 - 16:15	*Coffee break*
16:15 - 18:00	Parallel sessions – 5 different rooms • Biology and Health • Chemistry & Soft Matter (Characterization of structure and properties of chemical materials, reactivity, catalysis, chemical surfaces and interfaces / Soft matter) • Diluted matter • Geosciences / Environment / Cultural Heritage • Physics & Advanced Materials (Structural, electronic and magnetic properties of matter / Physical surfaces and interfaces)
18:00 - 20:00	*Posters session / Commercial Exhibitors / Aperitif*
20:00 - 21:30	*Award of the best student poster / Dinner*

Tuesday, January 28th, 2025

SOLEIL Auditorium - Main Building

9:00 - 10:00	Parallel sessions on technic / methodology – 5 different rooms • X-ray Absorption Spectroscopy (soft / tender / Hard X-ray) • Imaging / Microscopy / Tomography • Photoelectron Spectroscopies • Diffraction & Scattering / Coherence Network for Students & Postdocs
10:00 - 10:30	*Coffee break – Cafeteria Space*
10:30 - 11:15	**PLENARY TALK** – Electronic and magnetic properties of matter ***Federico Mazzola****– University Ca'Foscari, Venice, Italy* Probing chiral phases in quantum matter.
11:15 - 12:30	**ROUND TABLE** – Experimental environments at SOLEIL: status, on-going projects, our dreams for SOLEIL 2 *S2UO and SOLEIL staff*
12:30 - 13:45	*Lunch*
14:00 - 15:00	Visit of 3 Beamlines (PLEIADES / ROCK / SWING)
14:00 - 17:00	**Tutorial 1** – The latest developments of FASTOSH *Gautier Landrot – SOLEIL Synchrotron (SAMBA beamline)* ***PHENIX Room*** **Tutorial 2** – Surface diffraction - Data treatment and analysis *Fabien Cheynis (S2UO), Alessandro Coati, Andrea Resta, Alina Vlad, Yves Garreau (SOLEIL)* ***LIBRA Room*** **Satellite** – The new CryoEM - POLARIS *Paloma Fernandez-Varela (S2UO), Pierre Legrand, Eric Larquet, Heddy Soufaril (SOLEIL-POLARIS)* ***SOLEIL Auditorium***

Parallel Session Schedule

Biology and Health

Chairpersons: B. Masquida, P. Fernandez Varela

SOLEIL - Reception Building Auditorium

Monday, January 27th, 2025

14:00 - 14:30 (25'+5')	Regauletion of toxSAS toxin-antitoxin systems: Phage sensors and defence effectors ***Abel Garcia Pino*** *– Univ. Libre de Bruxelles, Belgium*
14:30 - 14:55 (20'+5')	First detection of calcium oxalate in human hairs ***Christophe Sandt*** *- SOLEIL Synchrotron, St Aubin, France*
14:55 - 15:20 (20'+5')	Domain of 36 of tropoelastin interacts with model membranes ***Manon Ragouilliaux*** *- MEDyC, Reims, France*
15:20 - 15:45 *(20+5')*	Analysis of the organic-inorganic interface in pigmented biominerals: The case of octocoral sclerites and sea urchin spines ***Arturo J. Álvarez Valverde*** *- Laboratoire de Chimie de la Matière Condensée de Paris, France*
15:45 - 16:15	*Coffee break*
16:15 - 16:45 (25'+5')	Biophysical and structural study of La Crosse virus endonuclease inhibition for the development of new antiviral options ***Mikael Feracci*** *- Architecture et Fonction des Macromolécules Biologiques, Marseille, France*
16:45 - 17:10 (20+5')	Exploring cryo-correlative light, electron, and X-ray microscopy (cryo-CLEXM): Methods and insights for biological imaging ***Harisa Rista*** *- SOLEIL Synchrotron, St Aubin, France*
17:10 - 17:35 (20'+5')	Hijacking of host PDZ proteins by the hepatitis B virus during infection ***Lina Boumediene*** *- Institut Pasteur, Paris, France*
17:35 - 18:00 (20'+5')	Multiscale atlas of Xpa in vivo biomolecular condensates ***Pierre Montaville*** *- SOLEIL Synchrotron, St Aubin, France*

Parallel Session Schedule

Chemistry & Soft Matter
(Characterization of structure and properties of chemical materials, reactivity, catalysis, chemical surfaces and interfaces / Soft matter)

Chairpersons: Amélie Bordage, Asma Tougerti, Florent Carn

CEA BLOCH Auditorium

Monday, January 27th, 2025

14:00 – 14:30 (25'+5')	Suivi in situ des mécanismes de formation de nanocristaux semi-conducteurs ***Benjamin Abecassis*** *– ENS Lyon, France*
14:30 - 14:55 (20'+5')	Synergistic effect of oxygen and water on the environmental reactivity of 2D layered GeAs ***Luca Persichetti*** *– Università di Roma, Tor Vergata, Italy*
14:55 - 15:20 (20'+5')	Electronic structure of binary and ternary uranium sulphides probed by X-ray spectroscopies ***Thomas Stephant*** *- ISCR, Rennes, France*
15:20 - 15:45 (20+5')	Investigation of composite organic nanoparticles: A comparative study using soft X-ray STXM and Ptychography ***Corentin Rieb*** *- University of Strasbourg, France*
15:45 - 16:15	*Coffee break*
16:15 - 16:45 (25'+5')	Catalysts for dehydroaromatization of methane: insights from operando characterization ***Nikolay Kosinov*** *– Eindhoven University of Technology, The Netherlands*
16:45 - 17:10 (20+5')	Droplet levitation for in-situ SAXS studies ***Claire Hotton*** *- Laboratoire de Physique des Solides, Orsay, France*
17:10 - 17:35 (20'+5')	A real time kinetic study of hydrogen diffusion in a single crystal by in situ infrared absorption ***Kamil F. Dziubek -*** *Faculty of Chemistry, Warsaw University of Technology, Warszawa, Poland*
17:35 - 18:00 (20'+5')	Understanding CH4 steam reforming catalysts under dynamic reaction conditions by systematic in situ/operando XAS and XRD studies ***Andrea De Giacinto*** *- Karlsruhe Institute of Technology, Germany*

Parallel Session Schedule

Diluted matter

Chairpersons: Héloïse Dossmann, Pierre Asselin

LIBRA Room

Monday, January 27th, 2025

14:00 – 14:30 (25'+5')	VUV photoionization of molecules of astrophysical interest: applied molecular physics in space ***Helgi Rafn Hrodmarsson*** *– LISA UPEC, Créteil, France*
14:30 - 14:55 (20'+5')	Operando PEPICO spectroscopy to unveil plastic pyrolysis mechanisms ***Patrick Hemberger*** *– PSI, Villigen, Switzerland*
14:55 - 15:20 (20'+5')	Flux and fluence effects on the VUV photodesorption and photoprocessing of CO_2 ices ***Antoine Hacquard – MONARIS,*** *Sorbonne Université, Paris, France*
15:20 - 15:45 (20+5')	Can we perform on-the-fly X-ray photoelectron spectroscopy of mass/charge selected ions produced by electrospray ionization source? ***Aleksandar R. Milosavljević -*** *SOLEIL Synchrotron, Saint Aubin, France*
15:45 - 16:15	*Coffee break*
16:15 - 16:45 (25'+5')	The methyl cation CH_3^+ produced by Synchrotron and laser VUV photoionisation of the methyl radical CH_3: Spectroscopy and reactivity ***Christian Alcaraz*** *– ICP Université Paris-Saclay, France*
16:45 - 17:10 (20+5')	Design and preliminary tests of a new double imaging photoelectron photoion coincidence (i^{2}PEPICO) spectrometer for VUV gas phase applications ***Myriam Drissi*** - *SOLEIL Synchrotron, Saint Aubin, France*
17:10 - 17:35 (20'+5')	*High-resolution structure and energetics of the DABCO molecule attached to clusters for modelling energy transport in matter* ***Audrey Scognamiglio*** - *ISMO, Orsay, France*
17:35 - 18:00 (20'+5')	Angularly resolved post-collision interaction on core ionized neon and water molecules in gas phase ***Abhishek Verma*** - *LCPMR, Paris, France*

Parallel Session Schedule

Geosciences / Environment / Cultural Heritage

Chairpersons: Charlotte Catrouillet, Simona Raneri

PHENIX Room

Monday, January 27th, 2025

14:00 – 14:30 (25'+5')	Asteroid Ryugu Chemica characterization from Synchrotron spectroscopy in the mid to far infrared, and AFMIR, of Hayabusa 2 samples ***Emmanuel Dartois*** *– ISMO Université Paris-Saclay, France*
14:30 - 14:55 (20'+5')	A BAG for major-to-trace elemental mapping and speciation of flat fossils at the PUMA beamline ***Pierre Gueriau*** *- IPANEMA, Saint-Aubin, France and Institute of Earth Sciences, Lausanne, Switzerland*
14:55 - 15:20 (20'+5')	Cu Redox speciation at magnetite surface: A Cu $L_{3,2}$-Edge study ***Jaimy Scaria*** *- Géosciences Rennes, France*
15:20 - 15:45 (20+5')	Determination of optical parameters of water ice using infrared Synchrotron Radiation: Characterisation of ice clouds emission ***Cecilia Taverna*** *- SOLEIL Synchrotron, Saint Aubin, France*
15:45 - 16:15	*Coffee break*
16:15 - 16:45 (25'+5')	Operando VIS-NIR microscopic hyperspectral imaging to monitor possible SR X-ray damage on paintings ***Amelia Suzuki*** *– School of Science and Technology, Nottingham Trent University, U.K.*
16:45 - 17:10 (20+5')	SR-XRPD investigation of artificially and naturally aged weighted silk fibers ***Alina Krotova -*** *ARCHES group and AXIS group University of Antwerp, Belgium*
17:10 - 17:35 (20'+5')	The magic scroll of the future Ampus museum of writing. The story continues... ***Jean-Paul Itié*** *- SOLEIL Synchrotron, St Aubin, France*
17:35 - 18:00 (20'+5')	Counterfeiting silver coins, from Roman republic to middle age ***Vincenzo Palleschi*** *- Laboratory of Applied and Laser Spectroscopy, Pisa, Italy*

Parallel Session Schedule

Physics & Advanced Materials
(Structural, electronic and magnetic properties of matter / Physical surfaces and interfaces)

Chairpersons: Debora Pierucci, Fabien Cheynis, Sara Gonzalez

SOLEIL Auditorium - Main Building

Monday, January 27th, 2025

14:00 – 14:30 (25'+5')	Atomic structure of novel 2D materials unveiled by surface X-ray diffraction ***Geoffroy Prevot** – INSP CNRS – Sorbonne Université, Paris, France*
14:30 - 14:55 (20'+5')	Unveiling electric field distribution in quantum dotbased devices by operando scanning photoemission microscopy ***Dario Mastrippolito** - SOLEIL Synchrotron, Saint Aubin, France*
14:55 - 15:20 (20'+5')	Mapping of stress/strain distribution within chromium layers deposited on Zr substrates at the ESRF facility ***Clément Ribart** - CEA, Grenoble, France*
15:20 - 15:45 (20+5')	Development of a high-throughput XRD wide-surface furnace for In Situ hydration Study of Proton Conductor electrolytes ***Giulio Cordaro** - CentraleSupélec, Gif-sur-Yvette, France*
15:45 - 16:15	*Coffee break*
16:15 - 16:45 (25'+5')	Structure and magnetic properties of FeRh nanomagnets deposited on perovskite surfaces ***Florent Tournus** - ILM, Lyon, France*
16:45 - 17:10 (20+5')	On the cuprates' universal waterfall feature: Evidence of a momentum-driven crossover ***Benjamin Bacq-Labreuil** - IPCMS, Strasbourg, France*
17:10 - 17:35 (20'+5')	A combination of real-time diagnostics probing the impact of nitrogen on silver thin film growth ***Michał Kamiński** - Karlsruhe Institute of Technology, Germany*
17:35 - 18:00 (20'+5')	Inverse freezing and melting of FeGd ferrimagnetic domains at the spin-reorientation transition ***Matthias Riepp** - IPCMS, Strasbourg, France*

Parallel sessions on technique / methodology

X-ray Absorption Spectroscopy (soft / tender / Hard X-ray)

Chairpersons: Amélie Bordage (S2UO), Sara Gonzalez (S2UO), Delphine Vantelon (SOLEIL)

SOLEIL - Reception Building Auditorium

Tuesday, January 28th, 2025

Introduction
09:00 - 09:20 Beamlines presentation, overview and perspectives
First feedbacks from the survey for XAS

09:20 – 10:00 Round table discussion / questions from the users

Parallel sessions on technique / methodology

Imaging / Microscopy / Tomography

Chairpersons: Andrew King (SOLEIL), Simona Raneri (S2UO), Pierre Asselin (S2UO)

SOLEIL Auditorium - Main Building

Tuesday, January 28th, 2025

Introduction
09:00 - 09:10 ***Andrew King*** *– SOLEIL Synchrotron (PSICHE and SOLEIL referent for imaging techniques)*

09:10 - 09:30 Beamlines presentation, overview and perspectives

09:30 - 10:00 Round table discussion / questions from the users

Parallel sessions on technique / methodology

Photoelectron Spectroscopies

Chairpersons: Debora Pierucci (S2UO), Fabien Cheynis (S2UO)

CEA BLOCH Auditorium

Tuesday, January 28th, 2025

09:00 - 09:20 (15'+5')	Nano-ARPES facility at ANTARES beamline for direct electronic structure determination of 2D materials ***José Avila** - SOLEIL Synchrotron, St Aubin, France*
09:20 - 09:40 (15'+5')	TEMPO beamline: high-resolution and time-resolved photoemission spectroscopy from UHV to near ambient pressure ***Matthieu Silly** - SOLEIL Synchrotron, St Aubin, France*
09:40 - 10:00	Round table discussion

Parallel sessions on technique / methodology

Diffraction & Scattering / Coherence

Chairpersons: Florent Carn (S2UO), Benoît Masquida (S2UO)

PHENIX Room

Tuesday, January 28th, 2025

09:00 - 09:10 (6'+4')	***Philippe Fontaine** – SIRIUS beamline*
09:10 - 09:20 (6'+4')	***William Shephard** – Proxima-2A beamline*
09:20 - 09:30 (6'+4')	***Thomas Bizien** – SWING beamline*
09:30 - 09:40 (6'+4')	***Alessandro Coati** – SixS beamline*
09:40 - 09:50 (6'+4')	***Dominique Tiaudière** – DiffAbs beamline*
09:50 - 10:00 (6'+4')	***Pierre Legrand** – Proxima 1 and Polaris beamlines*

Networks for students & postdoc

Chairperson: Charlotte Catrouillet (S2UO), Asma Tougerti (S2UO)

LIBRA Room

Tuesday, January 28th, 2025

09:00 - 10:00

PLENARY SESSION

PLENARY TALKS

SOLEIL Auditorium - Main Building

Monday, January 27th, 2025

Cultural Heritage

10:50 – 11:35 Chemical characterization using a combination of in-situ, laboratory and Synchrotron-based technics of prehistoric coloring matter from the Franco-Cantabrian region. from Font-de-Gaume to Altamira.

Josè Tapia*– Lab-BC UAR3506 CNRS-C2RMF-Chimie ParisTech PSL, Paris, France*

11:35 - 12:05 High pressure in SOLEIL: Tools and science

The SOLEIL High Pressure Group *– Synchrotron SOLEIL – France*

Tuesday, January 28th, 2025

Electronic and magnetic properties of matter

10:50 – 11:35 Probing chiral phases in quantum matter.

Federico Mazzola *– University Ca’Foscari, Venice, Italy*

Chemical Characterization using a Combination of *in situ*, Laboratory and Synchrotron-based Techniques of Prehistoric Coloring Matter from the Franco-Cantabrian Region. From Font-de-Gaume to Altamira

J. Tapia[1,2], M. Eveno[2,3] and I. Reiche[1,2]

1 Laboratoire de développement instrumental et de méthodologies innovantes pour les Biens Culturels (Lab-BC), UAR3506 CNRS – C2RMF – Chimie ParisTech PSL University, Paris, France
2 Institut de recherche de Chimie Paris (IRCP), UMR8247 CNRS – Chimie ParisTech PSL University, Paris, France
3 Centre de Recherche et de Restauration des Musées de France, C2RMF, Paris, France

ABSTRACT

Paleolithic cave art stands as one of the earliest forms of art from modern humans, and consequently it is important to analyze and reach a better comprehension of it. The coloring matter found in these representations is typically black, red and yellow. The study of black Paleolithic coloring matter is well-developed, with established analytical procedures [1],[2]. However, the study of reds is more complicated and remains limited, as the coloring matter and the wall support present a similar chemical composition [3].

Our ongoing research has two main aims. First and most importantly, to improve the characterization of red Paleolithic pigments compared to the *in situ* analyses that can be undertaken with a portable instrument in caves. Second, to find differentiation criteria between the composition of the calcitic walls and the coloring matter, which would help to improve the performances of *in situ* measurements [4].

This presentation highlights analytical strategies for the study of complex prehistoric coloring matter. We will present non-invasive analyses performed *in situ,* such as those undertaken with portable XRF (pXRF) at the cave of Font-de-Gaume. These results will be combined with analyses performed on microsamples of red coloring matter from the caves of Altamira (including fourteen samples from the *Techo de los Polícromos*), La Garma, and several other key Paleolithic cave sites in Northern Spain, using micro-X-ray fluorescence (SR-µXRF) at the PUMA beamline, SOLEIL synchrotron. Applying this sensitive non-destructive technique allowed us to study trace elements and chemical markers, and identify the coloring matter in a way that is not possible *in situ* as of today. New distinction criteria between Paleolithic figures that were made with different red coloring matter were determined, alongside with a comparative analysis of the representations between them. New insights on the creation steps of these prehistoric figures were gained. Finally, by combining the results from *in situ* and synchrotron sensitive analyses, we further improved the identification of the elements to be searched for during future *in situ* analyses.

Figure 1. Techo de Los Polícromos, Altamira, with one of the analyzed locations marked with a white cross. © CNRS J. Tapia

REFERENCES

[1] Trosseau et al. JAAS 2021
[2] Reiche et al Archaeometry 2024
[3] Reiche, Tapia et al Archéosciences 2023
[4] Tapia et al Journal of Cultural Heritage 2024

High Pressure in SOLEIL: Tools and Science

The SOLEIL High Pressure Group

SOLEIL Synchrotron, L'Orme des Merisiers, 91190 Saint Aubin, France

ABSTRACT

Pressure, like temperature, is an intensive parameter used to explore the phase diagram of materials. By modifying interatomic distances, it allows access to exotic phases of matter by possibly inducing various types of phase transitions: structural, electronic, and magnetic, along with changes in other physicochemical properties. High-pressure science has broad applications, from synthesizing novel materials to replicating the extreme conditions of planetary cores, spanning disciplines such as physics, chemistry, materials science, biology, and geoscience.

At SOLEIL, high-pressure science is distributed across 7 beamlines, each offering unique, world-class capabilities, alongside a dedicated high-pressure laboratory (Materials Lab). Various high-pressure apparatuses are available to generate extreme conditions, reaching up to megabar pressures, including Diamond Anvil Cells (DAC) and large volume presses (LVP), accommodating samples sizes ranging from micrometers to millimeters. This diverse range of techniques and setups enables users at SOLEIL to perform complementary experiments, often utilizing multiple beamlines.

This presentation provides an overview of the high-pressure community at SOLEIL, along with a selection of the available techniques and high-pressure setups. Finally, it will highlight some key scientific results achieved using high-pressure techniques.

Probing Chiral Phases in Quantum Matter

F. Mazzola

University Ca'Foscari - Venice, Italy

ABSTRACT

For many years, since the beginning of the 19th century, the existence of magnetism in low dimensions has been both desired and controversial. It was long thought, that magnetic orders in low dimensional systems could not be realized at temperatures different from zero. At least, this was what the Mermin-Wagner theorem stated for isolated Heisenberg spins. The scarcity of low-dimensional materials with magnetic properties, and the partial understanding of the role of spin-anisotropy have supported this picture for several decades. In three-dimensions, magnetism has revolutionized our everyday life, enabling familiar technologies which are of common use. A few examples include computers' memories, RAM, hard-disks, key cards, credit cards, electric batteries, light, and distance sensors. This relentless pace of development has motivated the search for magnetism in systems with increasingly smaller sizes. With cooperation of experimental and theoretical physics, researchers discovered that spin-anisotropy can stabilize low-dimensional magnetism. In this, spin-orbit coupling plays an important role. Surface experimental probes, such as angle-resolved photoelectron spectroscopy provide researchers access to the electronic structure of solids. Despite the advances in the field, recently, new forms of surface local magnetism completely different from standard descriptions have appeared. Here, I aim to give an overview of a new powerful methodology to uncover hidden phases of electrons, including spins, and magnetism which was so fare elusive.

PARALLEL SESSIONS

Parallel Session Schedule

Biology and Health

SOLEIL - Reception Building Auditorium

Monday, January 27th

Chairpersons: B. Masquida, P. Fernandez Varela

IT-01	Regauletion of toxSAS toxin-antitoxin systems: Phage sensors and defence effectors ***Abel Garcia Pino***
OC-01	First detection of calcium oxalate in human hairs ***Christophe Sandt***
OC-02	Domain of 36 of tropoelastin interacts with model membranes ***Manon Ragouilliaux***
OC-03	Analysis of the organic-inorganic interface in pigmented biominerals: The case of octocoral sclerites and sea urchin spines ***Arturo J. Álvarez Valverde***
IT-02	Biophysical and structural study of La Crosse virus endonuclease inhibition for the development of new antiviral options ***Mikael Feracci***
OC-04	Exploring cryo-correlative light, electron, and X-ray microscopy (cryo-CLEXM): Methods and insights for biological imaging ***Harisa Rista***
OC-05	Hijacking of host PDZ proteins by the hepatitis B virus during infection ***Lina Boumediene***
OC-06	Multiscale atlas of Xpa in vivo biomolecular condensates ***Pierre Montaville***

Regaulation of toxSAS Toxin-antitoxin Systems: Phage Sensors and Defence Effectors

A. Garcia-Pino

Université libre de Bruxelles, Brussels, Belgium

ABSTRACT

Bacteria have evolved diverse immunity mechanisms to protect themselves against the constant onslaught of bacteriophages. They use pathogen-associated molecular patterns (PAMPs) that require phage-specific triggers to be activated to sense foreign invaders. Major breakthroughs in the last decade have helped to establish firmly the Pan-immune concept, unravelling a myriad of different bacterial antiphage systems. Despite this, detailed mechanistic characterization of the triggers for their activation remains largely obscure. We used toxSASs as model to study the molecular mechanism by which bacterial immune systems senses conserved phages. ToxSAS are the toxic effectors of toxin-antitoxin modules, small operons encoding a pair of proteins of which one, the toxin, is a potent inhibitor of bacterial growth, while the second, the antitoxin, inhibits the toxin's activity during normal growth. They inhibit cell growth by pyrophosphorylating nucleotides or nucleic acids. The neutralization of toxSAS by their antitoxin counterparts, is strongly coupled to the enzymatic activity of the toxin: toxSAS that modify nucleotides are neutralized via the occlusion of the pyrophosphate acceptor-binding site whereas those that target tRNAs block the access of ATP to the pyrophosphate donor site of the toxSAS, without affecting recruitment of the tRNA pyrophosphate acceptor. Our structural, microbiological and biochemical evidence suggests that bipartite toxSAS TA operons use stable multimerization as an alternative to the colocalization of toxin and antitoxin domains of monomeric fused TAs, such as CapRel^{SJ46}, to switch between neutralized and active state. The remarked modularity and structural plasticity of toxSASs suggest that these evolutionary dynamics underpinning host-pathogen relationships may be more complex than the typical sequential coevolutionary interplay between sensors and triggers. Indeed, the structural plasticity and broad specificity of toxSAS suggest they have evolved to directly and simultaneously detect different proteins from the phages providing stronger defense, which limits phage evasion and may enable broader protection range.

First Detection of Calcium Oxalate in Human Hairs

C. Sandt

SMIS Beamline, Synchrotron SOLEIL, L'orme des Merisiers, Départementale 128,
91190 Saint-Aubin, France

ABSTRACT

Oxalate metabolism is an important process for multiple health conditions such as primary and secondary hyperoxaluria, urolithiases, and crystalline nephropathies. We report for the first time the detection of calcium oxalate in the medulla of human hairs from the general population. We used synchrotron radiation infrared microspectroscopy (SR-µFTIR) to measure the chemical composition of the human hair medulla. We measured over 200 human hair medullas from the general population, which is probably the largest set of infrared spectral data from the human hair medulla ever measured(1). Calcium oxalate was found in 11% of hairs. The frequency of calcium oxalate in hair of the general population is not compatible with the prevalence of primary hyperoxaluria, a rare genetic disease, but could be compatible with several origins, secondary hyperoxaluria, nephrolithiasis, enteric hyperoxaluria... Calcium oxalate can exist in multiple phases and was identified as calcium oxalate monohydrate (COM) by its infrared spectrum. It was observed in or contiguous to the hair medulla and never seen in the cortex. The origin of COM in human hair is currently unknown but our results rule out the possibility of external contamination through the hair shaft and point to a metabolic origin. Our discovery of calcium oxalate in hair comes at the same time as the discovery of the implication of glyoxylic acid, a precursor of calcium oxalate used in cosmetic products, in several reported cases of acute kidney injuries after cosmetic treatments(2-6). Calcium oxalate in hair could be used as a non-invasive biomarker in clinical applications such as the management and monitoring of conditions related to oxalate metabolism such as kidney stone formation, and primary hyperoxaluria.

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Domain 36 of Tropoelastin Interacts with Model Membranes

M. Ragouilliaux[1], J-M. Crowet[1], N. Belloy[1], S. Baud[1],
M. Deleu[2], F. Wien[3], L. Debelle[1]

[1]*UMR URCA/CNRS 7369 MEDyC, University of Reims Champagne-Ardenne, Reims, France*
[2]*LBMI, TERRA Teaching and Research Centre, Joint Research Unit BioEcoAgro, UMRt 1158, Gembloux Agro-Bio Tech, University of Liège, Gembloux, Belgium*
[3]*Synchrotron SOLEIL, L'Orme des Merisiers, Saint Aubin BP48, 91192 Gif-sur-Yvette, France*

ABSTRACT

In vertebrates, elastin is a crucial extracellular matrix protein. It can be found in the elastic fibers that endow tissues with elasticity and resilience [1].

Domain 36 of tropoelastin is unique and highly conserved. It is neither a hydrophobic nor a cross-linking domain but harbors a cluster of positively charged amino acids (-RKRK) at its end (C-terminus of tropoelastin) and a hydrophobic cluster at its beginning. This domain also contains the only two cysteyl residues of tropoelastin. They are disulfide-bonded to form an intrachain loop structure [2].

As domain 36 seems to be important to orchestrate elastogenesis and because tropoelastin molecules reside at the membrane periphery during this process, we hypothesized that domain 36 could possibly interact with the membrane. To test this possibility, molecular dynamics simulations of 1000 ns with 3 replicas were carried out and suggested that domain 36 could bind to the outer layer of a model membrane. This possibility was further confirmed by experimental data from synchrotron radiation circular dichroism (SRCD). Specifically, orientated circular dichroism (OCD) spectra demonstrated the interaction of domain 36 with a membrane.

Keywords: elastin, lipids, molecular dynamics, circular dichroism, synchrotron radiation

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Analysis of the Organic-inorganic Interface in Pigmented Biominerals: The Case of Octocoral Sclerites and Sea Urchin Spines

A.J. Álvarez Valverde[1,2], J. Perrin[3], F. Jamme[3], O. Breedy[2],
M.L. Montero[2], N. Nassif[1], M. Albéric[1]

[1] Laboratoire de Chimie de la Matière Condensée de Paris (LCMCP),
Sorbonne Université, 4 Place Jussieu, 75005, Paris
[2] Materials Science and Engineering Research Center (CICIMA),
University of Costa Rica, San José, Costa Rica
[3]SOLEIL Synchrotron, L'Orme des Merisiers, 91190 Saint-Aubin

ABSTRACT

Marine organisms such as mollusks, echinoderms, and corals display striking colors due to the presence of small organic pigment molecules occluded, together with other macromolecules, within their $CaCO_3$-based biominerals.[1-4] Current understanding suggests that these organic molecules are incorporated during the biomineralization processes. However, the biological processes underlying the coloration of these complex and colorful hybrid materials still remain unknown. To address this question, we here studied the interplay between the organic and inorganic phases in two pigmented biominerals: the reddish sclerites from *Muricea fruticosa* octocorals and the green/purple spines from *Paracentrotus lividus* sea urchins. The reddish coloration arises from polyenals[5] while the green and purple hues come from polyhydroxylated naphthoquinones (PHNQ)[2].

In *M. fruticosa* sclerite cross-sections, we revealed using Raman imaging, a heterogeneous microscale spatial distribution of the polyenals and evidenced a higher pigment concentration towards the biomineral surface describing an arched and layered wavy pattern. This pigmented layer correlates with high fluorescence signals measured at the DISCO beamline and Second Harmonic Generation (SHG) microscopy. SHG also reveals that polyenals are occluded within the calcite phase with high intermolecular ordering. These results suggest that the pigment distribution within calcite follows the sclerite growth lines during the later stage of mineralization.

In contrast to *M. fruticosa* sclerites, *P. lividus* spines cross-sections show a continuous coloration from the inner to the outer part of the biominerals but with different hues (purple *versus* green) suggesting various pigment compositions during mineral growth. Strong fluorescence signals due to the organic molecules were also measured by Raman spectroscopy, which revealed their spatial arrangement as wavy arched lines similar to those found in sclerites. DISCO imaging showed additional radial fluorescent signals suggesting the presence of other molecules, which also exhibit intermolecular ordering evidenced by SHG.

Overall, our results suggest that pigment molecules are incorporated differently according to the stage of mineralization, indicating that biomineralization and pigmentation processes are two metabolic processes linked during the lifetime of these two model animals.

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Biophysical and Structural Study of La Crosse Virus Endonuclease Inhibition for the Development of New Antiviral Options

M. Feracci[a], S. Hernandez[a,b], L. Garlatti[a,c], C. Mondielli[a,d], R. Vincentelli[a], B. Canard[a,e], J. Reguera[a], F. Ferron[a,e], and K. Alvarez[a*]

[a]Université Aix-Marseille, Architecture et Fonction des Macromolécules Biologiques (AFMB)–UMR7257 CNRS– Case 932, 163 Avenue de Luminy, 13288 Marseille CEDEX 09, France.
[b]Université Lille; INSERM, UMR-S 1172, Lille Neuroscience and Cognition Research Centre, 59000 Lille, France.
[c]OmegaChem, Lévis, 480 Rue Perreault, Québec G6W 7V6, Canada.
[d]Evotec (France) SAS, Campus Curie, 195 Route d'Espagne, 31036 Toulouse, France
[e]European Virus Bioinformatics Center, Leutragraben 1, 07743 Jena, Germany.
**Correspondence e-mail: karine.alvarez@univ-amu.fr*

ABSTRACT

The large Bunyavirales order includes several families of viruses with a segmented ambisense (-) RNA genome and a cytoplasmic life cycle that starts by synthesizing viral mRNA. The initiation of transcription, which is common to all members, relies on an endonuclease activity that is responsible for cap-snatching. In La Crosse virus, an orthobunyavirus, it has previously been shown that the cap-snatching endonuclease resides in the N-terminal domain of the L protein. Orthobunyaviruses are transmitted by arthropods and cause diseases in cattle. However, California encephalitis virus, La Crosse virus and Jamestown Canyon virus are North American species that can cause encephalitis in humans. No vaccines or antiviral drugs are available. In this study, three known Influenza virus endonuclease inhibitors (DPBA, L-742,001 and baloxavir) were repurposed on the La Crosse virus endonuclease. Their inhibition was evaluated by fluorescence resonance energy transfer and their mode of binding was then assessed by differential scanning fluorimetry and microscale thermophoresis. Finally, two crystallographic structures were obtained in complex with L-742,001 and baloxavir, providing access to the structural determinants of inhibition and offering key information for the further development of Bunyavirales endonuclease inhibitors.

Exploring Cryo-Correlative Light, Electron, and X-ray Microscopy (Cryo-CLEXM): Methods and Insights for Biological Imaging

H. Rista, C. Lumy, S. Kubsky, P. Legrand, P. Montaville and F. Jamme

Synchrotron SOLEIL, Experience division DISCO and PROXIMA1 beamlines

ABSTRACT

Cells and other biological samples are complex and heterogeneous systems. In order to fully comprehend such systems, it is necessary to explore them using both chemical and high-resolution ultrastructural information. To get this information, we use Cryo Correlative Light, Electron, and X-ray Microscopy cryo-workflow (Cryo-CLEXM). Each one of the methods involved in CLEXM or in one of its variations (CLEM, CLXM) spans a specific resolution range, and correlation becomes possible when these ranges overlap. The correlation allows for a deeper understanding of how biological function influences, and is influenced by, cellular structure and dynamics.

We used Cryo-CLEXM to study Liquid-Liquid Phase Separation (LLPS) in HEK293fs cells transfected with the XPA fluorescent protein expression vector and to investigate morphological changes in replication organelles in Huh7 cells infected with GLT1cc HCV.

The preservation of samples in their near-native state is the first bottleneck researchers encounter in cryo-imaging. Preservation is achieved by means of fast plunge freezing (vitrification) of the sample itself. However, the success rate is highly sample-dependent. For this reason, any multimodal bioimaging workflow would benefit from dedicated cryo-sample qualification protocols. The validation relies on specific information on the molecular as well as the macroscopic properties. X-Ray Diffraction (XRD) analysis reveals the molecular arrangement of water in the sample through the interpretation of its diffraction patterns, while quantitative information can be obtained using interferometry. This technique provides the topography of the sample’s surface, therefore allowing a correlation between its thickness and its diffraction pattern.

With this poster, I would like to highlight how well-designed sample preparation protocols and correlative multimodal imaging can adapt to answer biological questions across diverse sample types and functions.

Hijacking of Host PDZ Proteins by the Hepatitis B Virus during Infection

L. Boumediene[1], R. Veloso[2], A. Thureau[3], B. Raynal[4],
C. Venien-Bryan[2], and C. Caillet-Saguy[1]

[1]*Humoral Immunology Unit, Institut Pasteur, Paris, France*
[2]*Physique des Matériaux et de Cosmochimie, IMPMC, Sorbonne Université, Paris, France*
[3]*SWING beamline, Synchrotron SOLEIL, Gif-sur-Yvette, France*
[4]*Molecular Biophysics Platform-C2RT, Institut Pasteur, Paris, France*

ABSTRACT

Chronic infection to hepatitis B virus (HBV) constitutes a major public health concern, despite the broad availability and potency of the vaccine. Our project focuses on the HBV capsid protein (Hbc) and its interactions with PDZ-containing proteins both in vitro and within infected cells. The HBV capsid is an icosahedral structure with a 40nm diameter and is assembled by 240 monomers of a single 183-residue protein chain. At the C-terminal end of this sequence lies a short PDZ-binding motif (PBM), which interacts with the small globular PDZ domains of host cells proteins (1), by competing with the endogenous PBMs (2). This competition is a well-documented viral strategy that disrupts host cell homeostasis. Our goal is to elucidate the interactions between the HBV capsid and host PDZ domains using a combined structural approach, including cryo-electron microscopy (cryoEM) and small-angle X-ray scattering (SAXS) experiments. SAXS experiments, conducted on the SWING beamline at SOLEIL, revealed the impact of PDZ binding on the global structure of the capsid. The scattering profiles of the Hbc:PDZ complex were compared to those of the capsid alone, uncovering a notable change in the overall size of the complex, a result further validated by cryo-EM. Ongoing cryo-EM experiments aim to provide atomic-level insights into the HBc-PDZ interaction. These structural studies will be complemented by in vivo experiments to deepen our understanding of the biological significance of these interactions.

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Multiscale Atlas of Xpa *in vivo* Biomolecular Condensates

O. Leymarie[1*], A. Skobelkina[1*], S. Jeannin[2], C. Brewee[1], T. Isabet[1],
P. Montaville[1#], E. Pereiro[4], H. Chauvet[1], L. Chavas[3], F. Jamme[1#]

[1] Synchrotron SOLEIL, L'Orme des Merisiers - Départementale 128 - 91190 Saint Aubin – France
[2] Institute for Integrative Biology of the Cell (I2BC), CEA, CNRS, Uni Paris-Sud, Uni Paris-Saclay, Gif-sur-Yvette, France
[3] Nagoya University, Nagoya 464-8603, Japan
[4] ALBA Synchrotron Light Source, Cerdanyola del Valle`s, Barcelona 08290, Spain,

** Co-first authors*
 # Co corresponding authors

Keywords : in vivo crystallography, multimodal multiscale imaging, cryoSXT, multiphoton imaging

ABSTRACT

A model system studied at SOLEIL as an in-house project allows us to highlight the contribution of synchrotron radiation for studying interfaces in biology. XPA is a fluorescent protein that spontaneously crystallizes in-vivo[1]. Expression of XPA in HEK293 cells is an artificial model system suitable for biomolecular condensate studies and allows modulations of intracellular properties[2] that affect in vivo phase transitions of XPA to decipher mechanistic clues of such processes. The understanding of the transition of XPA from the intracellular liquid dilute phase to the crystalline solid dense phase required to combine live fluorescence imaging and correlative cryo fluorescence and cryo soft X-ray[3] microscopy and X-ray diffraction analysis. This multiscale approach revealed that XPA forms liquid condensates that are closely associated to biological membranes[4] of intracytoplasmic organelles. It also provided a description of the dynamics of these XPA liquid condensates, a spatial description at the cellular and meso scale of the phase transition from the liquid to the crystalline state in the cellular environment, and the autophagic cellular response to the liquid liquid phase separation of XPA.

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Parallel Session Schedule

Chemistry & Soft Matter
(Characterization of structure and properties of chemical materials, reactivity, catalysis, chemical surfaces and interfaces / Soft matter)

CEA BLOCH Auditorium

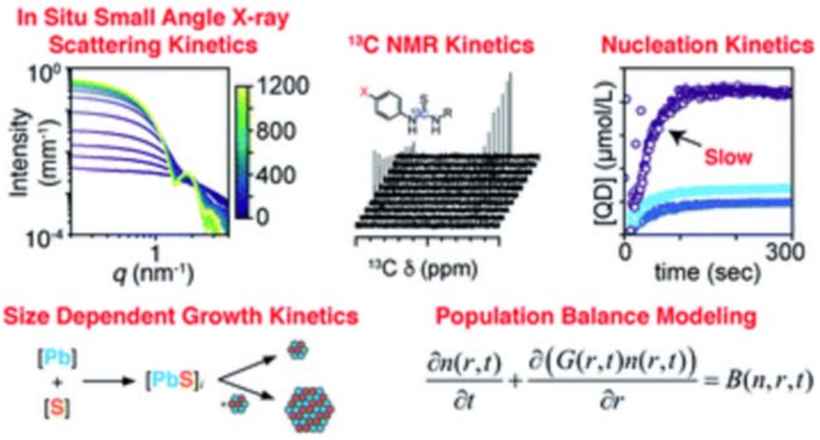
Chairpersons: Amélie Bordage, Asma Tougerti, Florent Carn

IT-03	Suivi in situ des mécanismes de formation de nanocristaux semi-conducteurs ***Benjamin Abecassis***
OC-07	Synergistic effect of oxygen and water on the environmental reactivity of 2D layered GeAs ***Luca Persichetti***
OC-08	Electronic structure of binary and ternary uranium sulphides probed by X-ray spectroscopies ***Thomas Stephant***
OC-09	Investigation of composite organic nanoparticles: A comparative study using soft X-ray STXM and ptychography ***Corentin Rieb***
IT-04	Catalysts for dehydroaromatization of methane: insights from operando characterization ***Nikolay Kosinov***
OC-10	Droplet levitation for in-situ SAXS studies ***Claire Hotton***
OC-11	A real time kinetic study of hydrogen diffusion in a single crystal by in situ infrared absorption ***Kamil F. Dziubek***
OC-12	Understanding CH4 steam reforming catalysts under dynamic reaction conditions by systematic in situ/operando XAS and XRD studies ***Andrea De Giacinto***

Suivi in situ des Mécanismes de Formation de Nanocristaux Semi-conducteurs

B. Abecassis

Laboratoire de Chimie, Ecole Normale Supérieure de Lyon, 46 allée d'Italie, 69008 Lyon



ABSTRACT

La synthèse de nanoparticules colloidales est devenue depuis 30 ans un champ très actif de la chimie synthétique. L'enjeu principal est de controler la taille et la forme des nanoparticules dont les porpriétés physiques exploitées dans les applications sont fortement dépendante.
Si les synthèses modernes produisent des distributions de taille extraordinairement étroites, les mécanismes à l'oeuvre pendant la formation des nanoparticules sont encore mal connus. La monodipsersité resulterait d'une « explosion de nucléation » rapide [1] suivie d'une croissance limitée par la diffusion et d'une focalisation de la distribution de taille [2]. En utilisant une combinaison de diffusion de rayons X in situ, d'absorption optique et de spectroscopie de résonance magnétique nucléaire (RMN) 13C, nous surveillons la cinétique de la génération de solutés de PbS, la nucléation et la croissance cristalline à partir de trois précurseurs de thiourée dont la réactivité de conversion s'étend sur 2 ordres de grandeur. Dans les trois cas, la nucléation est lente et se poursuit pendant plus de 50 % de la précipitation. Un modèle d'équilibre de la population basé sur une loi de croissance dépendant de la taille (1/r) s'adapte aux données avec une constante de taux de croissance unique (kG) pour les trois précurseurs. Cependant, l'ampleur de la kG et l'absence de dépendance à la viscosité du solvant indiquent que l'étape limitant la vitesse n'est pas la diffusion de la solution vers la surface des nanoparticules. Plusieurs mécanismes limités à la réaction de surface et un modèle de pénétration du ligand qui s'adapte aux données de nos expériences en utilisant un seul paramètre d'ajustement sont proposés pour expliquer les résultats. [3,4]

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Synergistic Effect of Oxygen and Water on the Environmental Reactivity of 2D Layered GeAs

L. Persichetti[1,±], G. Giorgi[2], L. Lozzi[3], M. Passacantando[3],
F. Bournel[8,9], J-J. Gallet[8,9], L. Camilli[1,*]

[1]*Dipartimento di Fisica, Università di Roma "Tor Vergata", Via Della Ricerca Scientifica,*
1- 00133 Rome, Italy
[2]*Department of Civil and Environmental Engineering (DICA), University of Perugia,*
Via G. Duranti 93, 06125, Perugia, Italy;
[6]*Department of Physical and Chemical Science, University of L'Aquila, via Vetoio, Coppito, 67100*
[8]*Sorbonne Université, CNRS, Laboratoire de Chimie Physique-Matière et Rayonnement,*
Campus Curie, UMR 7614, 4 place Jussieu, 75005 Paris, France
[9]*Synchrotron SOLEIL, L'orme des Merisiers, B.P. 48, Saint Aubin,*
Gif-sur-Yvette Cedex 91192, France

ABSTRACT

Among the 2D materials beyond graphene, ultra-thin oxides have recently attracted increasing attention due to their wide range of potential applications, spanning from catalysis to energy generation [1-2]. One of the main issues limiting their technological impact is the challenging preparation which is typically not scalable [2]. Aiming at finding alternative pathways to the formation of ultra-thin 2D oxide films, we recently investigated the oxidation of a parent layered material as a potential approach [3]. As a model system, we chose GeAs which, per se, is a fascinating 2D material exhibiting compelling properties related to its intrinsic anisotropy [4] but also showing high reactivity to air with the formation of a top Ge-oxide passivating layer. This affects quite substantially GeAs implementation in optoelectronics, producing relevant changes in important parameters such as the contact resistance or the refractive index. Thus, it is of outmost importance to investigate the reactivity of GeAs with O_2 but also in conditions closer to realistic working environments of a device, i.e. the behavior when exposed to humid O_2 and water. At the TEMPO beamline of Soleil, by exploiting the NAP-XPS endstation of Sorbonne Université, we explored the reactivity of layered GeAs in the presence of oxygen and/or water [5]. By conducting X-ray photoelectron spectroscopy (XPS) experiments under near-ambient pressures (up to 20 mbar), we obtained valuable insights into the material's stability under realistic conditions. GeAs displayed minimal reactivity with dry O_2 and de-aerated H_2O. However, the introduction of slight humidity (Rw = 0.5% at T = 20°C) in an O_2 atmosphere notably enhanced its reactivity. This synergistic effect was well supported by density functional theory calculations, which revealed a strongly exothermic formation energy for the simultaneous chemisorption of O_2 and H_2O, compared to the adsorption of each molecule individually.

We acknowledge the support of the European Union-Next Generation EU through the MUR PRIN2022 ATYPICAL (2022JW8LHZ) and MUR PRIN 2022-PNRR SPIGA (P2022LXNYN).

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Electronic Structure of Binary and Ternary Uranium Sulphides Probed by X-ray Spectroscopies

T. Stephant[1,2], M. O. J. Y. Hunault[2], P. L. Solari[2],
M. Pasturel[1], C. Prestipino[3]

[1] *Univ Rennes, CNRS, ISCR-UMR6226, F-35000, Rennes, France*
e-mail : thomas.stephant@univ-rennes.fr
[2] *SOLEIL Synchrotron, L'Orme des merisiers, 91190 Saint-Aubin, France*
[3] *Laboratoire CRISMAT, UMR 6508, CNRS, ENSICAEN, 14050 Caen, France*

ABSTRACT

Thanks to its 5*f* orbitals, uranium benefits from several valence states, from U^{2+} to U^{6+}, in inorganic compounds and possesses a wide crystal-chemistry. The radial expansion of these orbitals leads to energetically close crystal field (dominating in the case of 3*d* elements) and spin-orbit coupling (dominating in the case of 4*f* rare earths) interactions, and subsequent rich and exotic physical properties [1] (*e.g*. coexistence of superconductivity and ferromagnetism). Associated to a chalcogen element (Q = S, Se, Te), uranium forms inorganic compounds characterized by various crystallographic structures leading to unique uranium polyhedral structures [2] with *e.g*. the presence of $(S_2)^{2-}$ dimers.

We present the investigation of binary and ternary uranium sulphides (U-*T*-S systems, *T* = 3*d* transition metal) [3], that have been characterized by HERFD-XANES and RIXS spectroscopies at the U M_4 and L_3 edges and XANES at *T* K edge. These measurements enabled us (i) to determine the oxidation states of uranium and transition metal *T* for these compounds, and (ii) to get some hints about the most influent parameters on the position and shape of the white line.

The obtained information on the crystallo-chemistry of selected examples of compounds will be discussed in parallel to their physical properties.

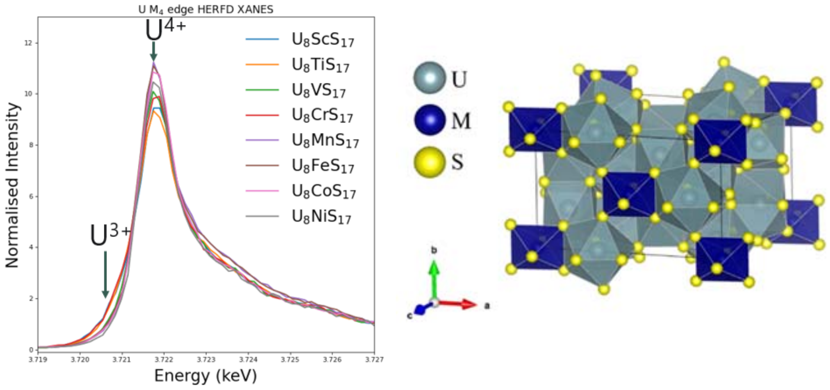

Figure 1: Variation of the HERFD XANES spectra at U M_4-edge for the U_8TS_{17} series and the related crystal structure.

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Investigation of composite organic nanoparticles: A comparative study using soft X-ray STXM and Ptychography

C. RIEB [1, 2], A. HEBRAUD [1], S. SWARAJ [2]

[1]University of Strasbourg, Strasbourg, France
[2] SOLEIL Synchrotron, Saint-Aubin, France
Email: corentin.rieb@etu.unistra.fr

Organic semiconducting (OSC) nanoparticles (NPs) were developed for applications in photovoltaics and photocatalysis.[1,2] Two different preparation processes were implemented that result in NPs with different internal morphology: core/shell or intermixed. We present here the characterization of these materials using STXM (Scanning Transmission X-ray Microscopy) and Ptychography[3] with soft X ray. Certain aspects, such as comparative spatial resolution, carbon deposition and spectro-ptychography are exemplified.

While STXM C and N K-edge analysis allowed for straightforward characterization of core/shell morphology and its purity, ptychography reconstruction provided much higher spatial resolution along with, arguably, interesting insight into the intermixed morphology with low damage.

The dose rate calculation gives a value of 1187MGy for Figure 1A and 0.148MGy for Figure 1B. Moreover, the spatial resolution of STXM high-resolution image is worth (18.4 ±1.2nm) that Ptychography (16.9±1.9nm). These values were determine by using Fourier Ring Correlation[4–6] (Figure 1E and 1F).

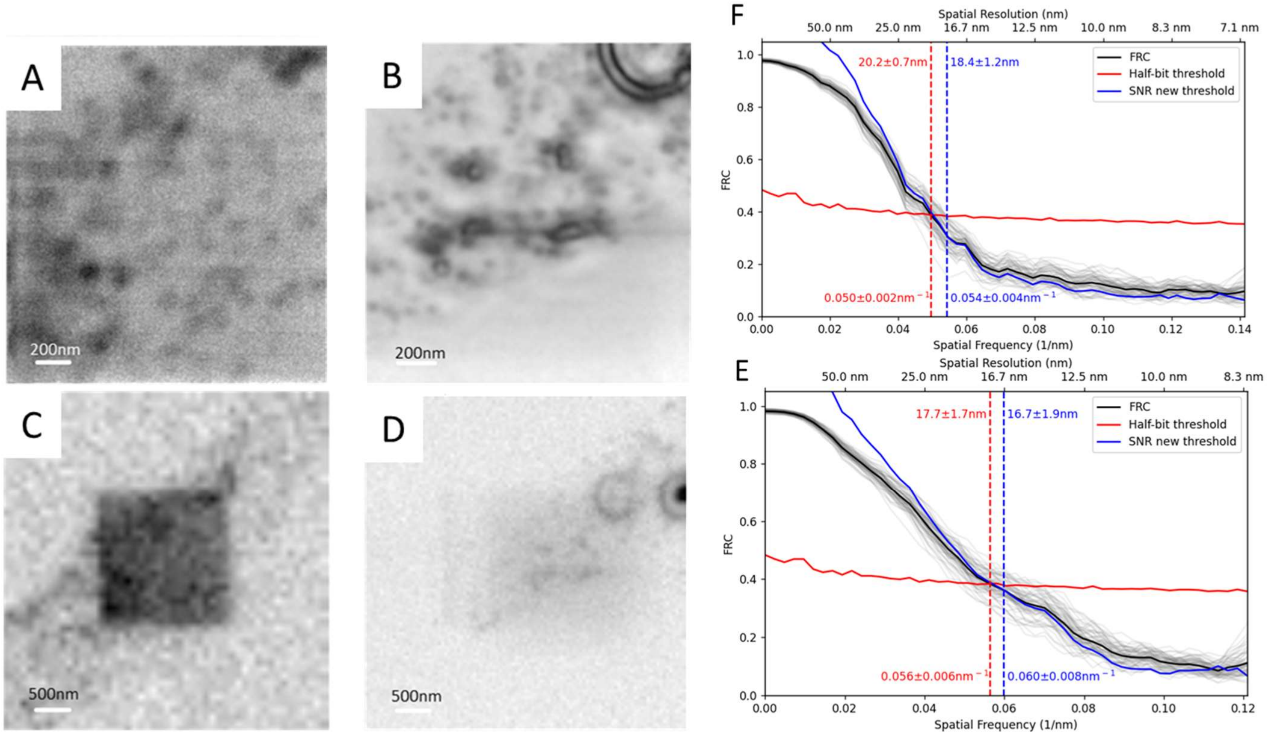

Figure 1: Intermixed NPs of PTQ10/ITIC-4F observed by STXM (A) and Ptychography (B). Images STXM (C) and (D) showed carbon deposition due to the precedent observation, STXM for (A) and Ptychography for (B) and graphics (E) and (F) correspond to FRC of image (A) and (B).

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Catalysts for Dehydroaromatization of Methane: Insights from Operando Characterization

N. Kosinov

Eindhoven University of Technology, The Netherlands n.a.kosinov@tue.nl

ABSTRACT

Non-oxidative dehydroaromatization of methane (MDA) to hydrogen gas and a mixture of light aromatics (benzene, toluene, xylenes and naphthalene) is a promising process for the direct valorization of natural gas to liquid hydrocarbons. The application of this reaction in practical technology is hindered by the lack of understanding about the mechanism and nature of the active sites in benchmark zeolite-based catalysts. Main challenges in gaining insight into the mechanistic aspects of MDA are the high reaction temperature at which the reaction takes place and its transient nature, which involves rapid activation and deactivation stages when the fresh catalyst is exposed to the methane feed.[1] Here, we used a combination of pulse reaction technique, isotope labelling, operando XRD, operando XANES, operando TGA and several other characterization tools to elucidate the active sites and complex MDA mechanism over various Mo/ZSM-5, Re/ZSM-5, Fe/ZSM-5 and other catalysts. First, using operando XANES, we show that the active sites in Mo/ZSM-5 and Fe/ZSM-5 are partially reduced and highly dispersed species stabilized by the zeolite framework; in Re/ZSM-5 – small metallic rhenium nanoparticles.[2,3] Further, we demonstrate that MDA is governed by a hydrocarbon pool mechanism, in which benzene is derived from secondary reactions of confined polyaromatic carbon species with the initial products of methane activation.[4,5] Once these confined species grow further, they block the pores and lead to the complete catalyst deactivation. Importantly, using isotope labelling and 13C NMR, we found that formation of deactivating species (coke) inside the pores is a highly reversible process.[6] Based on these insights we propose several strategies to improve activity and stability of MDA catalysts. First, we demonstrate that high-pressure operation helps mitigating the formation of coke and significantly improves catalyst productivity.[6] Second, we synthesized a novel Fe@ZSM-5 catalyst with highly-dispersed Fe-sites stabilized within the zeolite framework. This catalyst demonstrates significantly higher activity and selectivity as compared to conventional Fe/ZSM-5 materials [7, 8]

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Droplet Levitation for In-situ SAXS Studies

C. Hotton[1], T. Bizien[2], E. Paineau[1] and C. Hamon[1]

[1]*Université Paris-Saclay, CNRS, Laboratoire de Physique des Solides, Orsay, 91405, France*
[2]*Synchrotron SOLEIL, L'Orme des Merisiers, 91192 Gif-sur-Yvette Cedex, France*

ABSTRACT

In soft matter science, conventional ex-situ studies often fail to capture dynamic processes, rapid phase transitions and transient states, highlighting the need for in-situ approaches to fully understand such complex phenomena. Among recent developments, acoustic levitation[1] renews interest for conducting in-situ studies on self-standing droplets, enabling real-time kinetic analysis. We successfully implemented a compact acoustic levitator setup on the SWING beamline. The combination of small-angle X-ray scattering (SAXS) with acoustic levitation can address a variety of scientific challenges, including the investigation of out-of-equilibrium phenomena, triggering the self-assembly of colloids into smart materials, and revisiting the underlying mechanisms during drying processes.

We have chosen three examples to illustrate in-situ SAXS studies of these phenomena. Imogolite double-walled nanotubes (Ge-DWINTs) are ideal for exploring liquid-crystal phase transitions in out-of-equilibrium conditions, as their aspect ratio can be easily controlled through synthesis[2,3], forming stable colloidal dispersions that display rich liquid-crystalline phases at low concentrations before transitioning to a gel state[2,4]. In-situ SAXS experiments during evaporation of the droplet in levitation revealed the nature of liquid-crystal phases and provide a complete exploration of the phase diagram for Ge-DWINTs[5]. Using only one microliter of sample, we could analyze the system from dilute to highly concentrated states in less than one hour. Both the aspect ratio of the nanotubes and the initial volume fraction were key factors affecting phase transitions. Alternatively, bimetallic gold and silver nanorods[6] offer an excellent platform for investigating the formation of supercrystals, revealing critical insights into the structural compaction process during evaporation induced self-assembly (EISA)[7,8]. For this system, in-situ kinetic studies showed a phase transition from an isotropic to a hexagonal structure, followed by a progressive lattice compaction due to increased depletion forces during EISA. Finally, coupling acoustic levitation with time-resolved SAXS experiments enabled us to probe for the first time the continuous evolution from osmotic swelling to crystalline swelling during the drying process of a smectite suspension.

Overall, these results establish acoustic levitation as a powerful, emerging tool for rapid phase diagram determination and real-time observation of superstructure formation and rapid transitions.

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A Real Time Kinetic Study of Hydrogen Diffusion in a Single Crystal by *in situ* Infrared Absorption

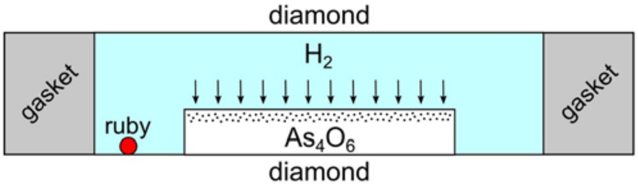
K.F. Dziubek,[1] P.A. Guńka,[2] and F. Capitani[3]

[1]Faculty of Chemistry, Warsaw University of Technology, Noakowskiego 3, 00-664 Warszawa, Poland
[2]Institut für Mineralogie und Kristallographie, Univ. Wien, Josef-Holaubek-Platz 2, 1090 Wien, Austria
[3]Synchrotron SOLEIL, L'Orme des Merisiers, 91192 Gif sur Yvette Cedex, France

ABSTRACT

Hydrogen migration in the Earth's depths is an important phenomenon that may be more widespread than previously thought [1] and, therefore, it requires further investigation. Hydrogen has enormous potential as an environmentally friendly alternative to fossil fuels, and identifying the previously unexplored deep reservoirs of H_2 may help to tackle climate change and improve energy security [2,3].

We have experimentally determined pressure-induced hydrogen incorporation into the single crystal of arsenolite, As_4O_6, employing synchrotron Fourier transform infrared spectroscopy [4]. The process leading ultimately to the formation of the inclusion compound $As_4O_6{\cdot}2H_2$ at 1.47(5) and 1.37(5) GPa was studied in a diamond anvil cell. The complete permeation lasted 1 and 11 h, respectively. $As_4O_6{\cdot}2H_2$ growth rate is linear with a slope of 7.7(4) μm/h at 1.47(5) GPa, and the rate-limiting step is the reaction As4O6 + 2H2 → $As_4O_6{\cdot}2H_2$ taking place at the $As_4O_6/As_4O_6{\cdot}2H_2$ phase boundary. Interestingly, at 1.37 (5) GPa the kinetics of $As_4O_6{\cdot}2H_2$ formation has two domains of linear growth with slopes of 0.25(1) and 1.2(1) μm/h.



In addition to the reaction kinetics data, a method of growing thin plates of As_4O_6 single crystals via spatial confinement will be presented. Using micro-sized thin-layered platelets (as shown in the Figure) proved to be a compelling way of studying the kinetics of diffusion in crystals. The measurements carried out at the SMIS infrared spectromicroscopy beamline in the SOLEIL Synchrotron clearly attest to the effectiveness of this new approach.

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Understanding CH_4 Steam Reforming Catalysts under Dynamic Reaction Conditions by Systematic *In situ/operando* XAS and XRD Studies

E. Tusini[1], A. De Giacinto[1], A. Zimina[2], D. Doronkin[2],
M. Casapu[1], J.-D. Grunwaldt[1,2]

[1] Institute for Chemical Technology and Polymer Chemistry, Karlsruhe Institute of Technology (KIT),
Engesserstr. 20, 76131 Karlsruhe, Germany
[2] Institute of Catalysis Research and Technology, Karlsruhe Institute of Technology (KIT),
Herrmann-von-Helmholtz-Platz 1, 76344 Eggenstein-Leopoldshafen, Germany
Email: grunwaldt@kit.edu

ABSTRACT

Catalytic methane steam reforming is a well-established commercial process for production of hydrogen and synthesis gas, contributing to approximatively 90% of the world-wide hydrogen production [1]. During recent years, small-scale applications for H_2-production such as fuel-cells have attracted increasing interest. However, due to their daily start-up and shut-down operation mode, the typical Ni-based catalysts are exposed to dynamic conditions, which leads to Ni oxidation and catalyst deactivation. To improve the performance of the Ni-based catalysts, the alloying of Ni with a noble metal is a favored approach [2,3]. To elucidate the interplay between Ni and Pt in bimetallic NiPt/$MgAlO_x$ catalysts, we conducted systematic *in situ/operando* XAS and XRD studies at the ROCK and DIFFABS beamlines of SOLEIL synchrotron during catalyst pre-reduction and methane steam reforming under transient reaction conditions [4]. The results obtained show that during catalyst activation by H_2-reduction, metallic Ni or Ni-Pt alloys are formed in the mono- and bimetallic samples, respectively, only above 800°C due to the high stability of the Ni-Mg mixed oxides. Under transient reaction conditions, pronounced oxidation and reincorporation of Ni into the support was observed for the monometallic sample, while catalyst deactivation is diminished in the bimetallic Ni-Pt catalyst. Herein, the catalyst composition plays as well an important role. Overall, the outcome of our studies helps at understanding the catalyst activation/deactivation process and provides the basis for a rational development of improved methane steam reforming catalysts.

ACKNOWLEDGMENTS

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Parallel Session Schedule

Diluted matter

LIBRA Room

Chairpersons: Héloïse Dossmann, Pierre Asselin

IT-05	VUV photoionization of molecules of astrophysical interest: applied molecular physics in space ***Helgi Rafn Hrodmarsson***
OC-13	Operando PEPICO spectroscopy to unveil plastic pyrolysis mechanisms ***Patrick Hemberger***
OC-14	Flux and fluence effects on the VUV photodesorption and photoprocessing of CO_2 ices ***Antoine Hacquard***
OC-15	Can we perform on-the-fly X-ray photoelectron spectroscopy of mass/charge selected ions produced by electrospray ionization source? ***Aleksandar R. Milosavljević***
IT-06	The methyl cation CH_3^+ produced by Synchrotron and laser VUV photoionisation of the methyl radical CH_3: Spectroscopy and reactivity ***Christian Alcaraz***
OC-16	Design and preliminary tests of a new double imaging photoelectron photoion coincidence (i^2PEPICO) spectrometer for VUV gas phase applications ***Myriam Drissi***
OC-17	High-resolution structure and energetics of the DABCO molecule attached to clusters for modelling energy transport in matter ***Audrey Scognamiglio***
OC-18	Angularly resolved post-collision interaction on core ionized neon and water molecules in gas phase ***Abhishek Verma***

VUV Photoionization of Molecules of Astrochemical interest: From Diatomics to Complex Organics Molecules (COMs) to Polycyclic Aromatic Hydrocarbons (PAHs)

H. R. Hrodmarsson

LISA UMR 7583 Université Paris-Est Créteil and Université Paris Cité, Institut Pierre et Simon Laplace, 61 Avenue du Général de Gaulle, 94010, Créteil, France

ABSTRACT

VUV photons are important drivers of multiple chemical and physical processes in space. As different molecules can be used as tracers for different types of cosmic environments, it is important to characterize both qualitatively and quantitatively the processes induced by VUV photons in different radiation fields.[1] This contribution will focus on VUV photoionization of various molecules of astronomical interest, going from diatomics upward to fullerenes, using the light from the DESIRS beamline at the Synchrotron SOLEIL facility.[2] The talk will cover the importance of absolute photoionization cross sections of small molecules such as OH,[3] and SH,[4] the importance of dissociative photoionization of astrobiologically relevant molecules (Derbali *et al.* 2019, 2020; Hrodmarsson *et al.* 2024a),[5-7] and the contribution of photoelectric heating by polycyclic aromatic hydrocarbons (PAHs) (Hrodmarsson *et al. in prep*).[8]

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Operando PEPICO Spectroscopy to Unveil Plastic Pyrolysis Mechanisms

J. Cabana[ab*], Z. Zhang[a], Z. Pan[ab], P. Kumar[a], A. Bodi[a], G.A. Garcia[c], L. Nahon[c], J.A. van Bokhoven[ab], and P. Hemberger[a]

[a] *Reaction Dynamics Group, Laboratory for Synchrotron Radiation and Femtochemisty, Paul Scherrer Institut, Villigen, Switzerland*
[b] *Eidgenössische Technische Hochschule Zürich, Zurich, Switzerland*
[c] *Synchrotron SOLEIL, L'Orme des Merisiers, St. Aubin, BP 48, 91192 Gif-sur-Yvette, France*

ABSTRACT

In recent years, plastic recycling has gained significant attention, as currently, only 10% of globally produced plastics are recycled, while the annual growth rate of plastic production is projected to reach 884 Mt by 2050.[1,2] Chemical recycling methods, such as pyrolysis and catalytic pyrolysis, are becoming increasingly popular, aiming to degrade polymers into their monomers or valuable chemicals and fuels, such as aromatics and olefins. However, these processes are far from economically viable, and a comprehensive mechanistic understanding is required for targeted optimization.

In this talk, we will discuss how plastic pyrolysis mechanisms over zeolites can be elucidated by detecting reactive intermediates using state-of-the-art operando photoelectron photoion coincidence (PEPICO) spectroscopy, as implemented at the Swiss Light Source (SLS) and Soleil synchrotron.[3]

1,3-dichlorobutane is utilized as a model compound for polyvinyl chloride (PVC). Pyrolysis experiments reveal radicals such as methyl, chloromethyl, and propargyl, along with 1,3-butadiene, which is produced via dehydrochlorination. While this diene favors di- and trimerization to yield aromatics such as benzene, toluene, and xylenes over HZSM-5, it remains less reactive over magnesia (MgO) or faujasites (HFAU). These results are discussed in conjunction with literature mechanisms and suggest novel avenues to increase the conversion and selectivity of this process.

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Flux and Fluence Effects on the VUV Photodesorption and Photoprocessing of CO_2 Ices

A. Hacquard[1],*, D. Torres-Díaz[1,2], R. Basalgète[1], D. Toulouse[1],
G. Féraud[1], S. Del Fré[3], J.A. Noble[4], L. Philippe[1], X. Michaut[1],
J-H. Fillion[1], A. Lafosse[2], L. Amiaud[2], M. Bertin[1]

1 - Sorbonne Université CNRS, MONARIS, UMR8233, F-75005 Paris, France.
2 - Université Paris-Saclay, CNRS, Institut des Sciences Moléculaires d'Orsay, 91405 Orsay, France.
3 - Univ. Lille, CNRS, UMR 8523 – PhLAM – Physique des Lasers Atomes et Molécules,
F-59000 Lille, France.
4 - Physique des Interactions Ioniques et Moléculaires, CNRS, Aix Marseille Univ., 13397 Marseille, France.
** also at Sorbonne Université, Observatoire de Paris, PSL university, CNRS, LERMA,*
F-75005, Paris, France

ABSTRACT

CO_2 is a major component of the icy mantles surrounding dust grains in planet and star formation regions [1]. Therefore, understanding its photodesorption is crucial for explaining gas phase abundances in the coldest environments of the interstellar medium that are irradiated by vacuum-UV (VUV) photons. In the last decade, photodesorption yields determined experimentally from CO_2 samples grown at low temperatures (T=15 K) have been found very sensitive to experimental methods and conditions. This has led to numerous and different values for the yields in the literacy [2-5]. Several mechanisms have been suggested to explain the desorption of CO_2, as well as its photoproducts O_2 and CO [3-7]. In this study, we explore the different mechanisms being photochemical process or physical process in order to explain these discrepancies. One of our main concern is the role played by the employed photon flux – an experimental parameter largely overlooked in previous studies, although it may significantly impact the measured yields, and thus has to be taken into account when it comes to applying the experimental values to conditions of the interstellar medium.

This presentation reports on the dynamics of photodesorption yields and sample composition as a function of photon fluence. Experiments were performed under different photon flux conditions (ranging from 7.3×10^{12} photon/s to 2.2×10^{14} photon/s) using monochromatic synchrotron radiation in the VUV (DESIRS beamline at synchrotron SOLEIL). Evolution cross sections are presented here to characterize the dynamics of ice yields and composition. Our work shows that CO2 photodesorption proceeds mostly via a DIET mechanism, as it was already hinted in some previous experimental studies, and that the role of photochemistry is comparatively negligible, contrary to what was previously assumed. This study also demonstrates that, depending on the mechanisms responsible for the desorption, different photon flux conditions will lead to different measured desorption yields, a conclusion of high importance for the extrapolation of the laboratory data to astrophysical conditions.

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Can we Perform on-the-fly X-ray Photoelectron Spectroscopy of Mass/Charge Selected Ions Produced by Electrospray Ionization Source?

A.R. Milosavljević, C. Nicolas, E. Robert and J. Bozek

Synchrotron SOLEIL, Saint Aubin, BP48, 91192 Gif-sur-Yvette Cedex, France

ABSTRACT

There has been a long-standing effort to perform X-ray photoelectron spectroscopy (XPS) of mass over charge (m/z) selected large/complex molecular ions [1-3]. Modern soft ionization techniques, such as electrospray ionization (ESI) [4] allow to bring in the gas phase very large and fragile molecules, for example, peptides, proteins, oligomers, clusters, micro-solvated species etc. About a decade ago, coupling of radio frequency (RF) ion traps with synchrotron radiation made possible to investigate electronic properties of such species by performing absorption spectroscopy [5,6]. In such an experiment, m/z resolved ion yields are measured as a function of the photon energy [5,6]. However, the later technique does not allow extraction of photoelectrons, therefore powerful synchrotron-based techniques, such as photoelectron spectroscopy and X-ray photoelectron spectroscopy, cannot be performed.

In the last several years, we have developed at PLEIADES a system that can produce and deliver in ultra-high vacuum a focused beam of m/z selected ions produced by an electrospray source. We have performed several experiments attempting to measure kinetic energy distribution of photoelectrons from the m/z targets. In this presentation, we will show preliminary results and discuss fundamental and experimental challenges to perform such an investigation.

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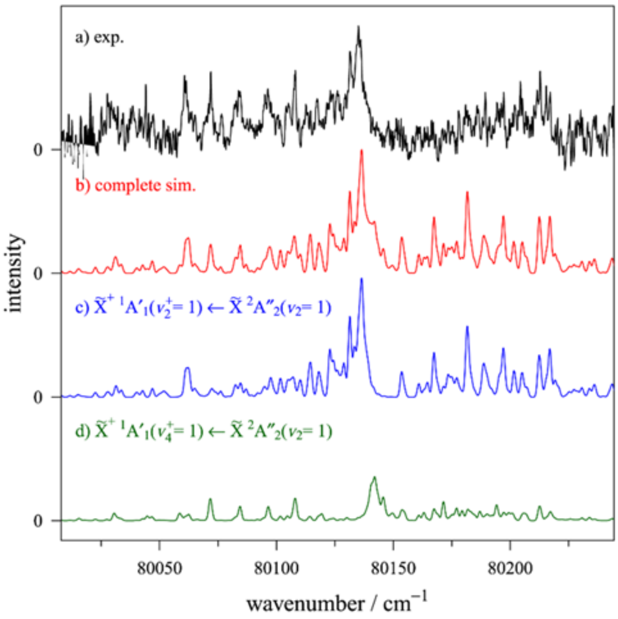
The Methyl Cation CH_3^+ Produced by Synchrotron and Laser VUV Photoionisation of the Methyl Radical CH_3: Spectroscopy and Reactivity

N.L. Chen[1], H.L. Le[1], B. Gans[1], S. Boyé-Péronne[1],
P.B. Changala[2], C. Alcaraz[3]

(1) ISMO, Univ. Paris-Saclay - CNRS, 91405 Orsay (France); (2) Center for Astrophysics, Cambridge, MA 02138 (USA); (3) ICP, Univ. Paris-Saclay - CNRS, 91405 Orsay (France)

ABSTRACT

A lot of experimental efforts have been put in the last decades at UPSaclay on the study of the spectroscopy and reactivity of CH_3^+ cations [1] which started with the development of sources of CH_3 radicals and their adaptation to synchrotron or laser sources. The recent rotationally resolved PFI-ZEKE study with a high resolution VUV laser presented here is a striking result of such a continuous work as it helps to confirm [2] the first direct observation of the CH_3^+ cation (by emission at 7 µm) in a protoplanetary disk by the JWST telescope [3]. High-level quantum-chemical calculations that accurately account for the strong Coriolis interactions beetween the out-of-plane v_2^+ and in-plane v_4^+ bending modes have been crucial for the understanding of the rovibronic structure of CH_3^+ and thus to model the spectrum.



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Design and Preliminary Tests of a New Double Imaging Photoelectron Photoion Coincidence (i^{2}PEPICO) Spectrometer for VUV Gas Phase Applications

M. Drissi, G.A. Garcia and L. Nahon

Synchrotron SOLEIL, L'Orme des Merisiers, Saint-Aubin, BP 48, Gif sur Yvette, France

ABSTRACT

Over the past decade, the main challenge in developing analytical experiments has been to develop an in situ, universal, selective, sensitive and multiplexed detection technique. These requirements have become essential to advance research in diverse fields such as astrochemistry, combustion, and atmospheric chemistry. Among the various molecular probing techniques, synchrotron-based photoelectron spectroscopy coupled to mass spectrometry has become particularly popular as it meets almost all of the above criteria. In particular, for complex mixtures, the PEPICO (photoelectron photoion coincidence) acquisition scheme has become the tool of choice as it allows the extraction of individual photoelectron and photoion spectra for each molecule present. Such spectral fingerprints can be used for the characterization of molecular structures (isomers, conformers and even enantiomers), the identification products and intermediates in complex chemical reactions/environments, measuring absolute cross sections, etc. [1-3]

The PEPICO technique is based on the sorting of electrons and ions into correlated pairs, each resulting from the ionization of a single neutral molecule, so-called true coincidence. In contrast, a false coincidence does not originate from the same initial neutral molecule. These false coincidences contribute to the background signal and limit the sensitivity of the instrument. In an ideal situation, the signal could be average as long as necessary, however, in most cases, time and resources are limited and make this solution non-viable. Another way around is to reduce the background signal by discriminating false from true coincidences. [4]

Here, I will present the latest development of our i^{2}PEPICO (double imaging photoelectron photoion coincidence) spectrometer installed at the DESIRS VUV beamline[5] and used to measure mass-selected photoelectron spectra in the gas phase with vibrational resolution. I will present the objectives and the electrostatic and mechanical design of our new spectrometer DELICIOUS IV which caters both to chemistry and physics communities by either lowering the detection limit and improving ultimate mass resolution or by decreasing the ultimate particle energy resolution. Then I will present the first tests performed with our current spectrometer[6] DELICIOUS III in preparation for commissioning. This includes the development of a code to determine the arrival position on the detector to discriminate true and false ions, as well as the design and testing of Helmholtz coils to replace the current µ-metal shielding used to cancel stray magnetic fields including the Earth's.

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High-resolution Structure and Energetics of the DABCO Molecule Attached to Clusters for Modelling Energy Transport in Matter

A. Scognamiglio[1], O. Shviro[1], B. Zarié[1], L. Barreau[1], D. Cubaynes[1],
B. Gans[1], E. Gloaguen[1], C. Schouder[1], G. A. Garcia[2], L. Nahon[2],
A. de la Lande[3] and L. Poisson[1]

[1] *Institut des Sciences Moléculaires d'Orsay, Université Paris-Saclay, CNRS, 91405 Orsay, France*
[2] *Synchrotron SOLEIL, L'Orme des Merisiers, St. Aubin BP 48, 91192 Gif sur Yvette, France*
[3] *Institut de Chimie Physique, Université de Paris-Saclay, CNRS, 91400 Orsay, France*

ABSTRACT

The diffusion of electrons in matter generates secondary species, such as secondary electrons and radicals, that can interact with nearby species. In cells, these interactions may cause DNA damage and increase cancer risk [1]. Recently, studies have shown that data on both elastic and inelastic scattering in small water clusters can support the development of theoretical models to predict energy transport at the macroscopic scale [2]. Clusters, in particular, provide effective models for describing organized condensed matter [3]. Our approach uses a highly symmetrical molecule, DABCO, known for its atomic-like orbitals [4], deposited on argon and water clusters and ionized via a 1-photon transition with synchrotron light (DESIRS beamline). Coincident ion and electron detection allows precise identification of the products formed and their photoelectron distributions. We have gathered detailed information on the electronic and vibrational structure of the isolated molecule and small clusters – examined here with unprecedented precision. We have as well retrieved information on the photoelectron angular distribution in both cases, from which we aim to gain insights on electron scattering processes.

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Angularly Resolved Post-collision Interaction of Core-ionized Neon and Water Molecule in the Gas Phase

A. Verma[1]*, R. Guillemin[1], R. Puttner[2], D.Peng[1], T. Marchenko[1],
O. Travnikova[1], M.N. Piancastelli[1], I. Ismail[1], F. Trinter[4], S. Thuermer[3],
U. Hergenhahn[3], B. Winter[3], J. Bozek[5] and M. Simon[1]

[1] *Sorbonne Université, CNRS, Laboratoire de Chimie Physique Matière et Rayonnement, F-75005, Paris, France*
[2] *Fachbereich Physik, Freie Universitat Berlin, Arnimallee 14, D-14195 Berlin, Germany*
[3] *Fritz-Haber-Institutder Max-Planck-Gesellschaft, Faradayweg 4-6,14195 Berlin, Germany*
[4] *Goethe Universitat Frankfurt Institut f ¨ ur Kernphysik Max-von-Laue-Strasse 1, 60438 Frankfurt am Main, Germany*
[5] *Synchrotron SOLEIL, l'Orme des Merisiers,Saint-Aubin, F-91192 Gif-sur-Yvette, Cedex, France*
∗Contact: abhishek.verma@sorbonne-universite.fr *Keywords: Photoelectron, post-collision*

ABSTRACT

interaction, polarization, recapture of photoelectron The absorption of a high-energy photon predominantly induces core ionization, which subsequently relaxes via Auger decay, resulting in a doubly charged atomic ion. For photon energies just above the ionization threshold of the coreshell, the ejected photoelectron is slow. It remains close to the atom when the Auger electron is emitted with a higher kinetic energy. This proximity fosters a significant Coulomb interaction among the three charged entities: the ion, the slow-moving photoelectron, and the high-energy Auger electron. Moreover, the ionic field varies during the course of the Auger decay. This process involves a three-body interaction between the emitted electrons and the ion, encompassing both energy and angular momentum exchange [1, 2]. This phenomenon is known as post-collision interaction (PCI). Despite decades of research [3], a complete understanding of PCI remains challenging. To improve our understanding of PCI, we conducted an experimental investigation focusing on the exchange of energy and angular momentum. The study was carried out on neon atoms and water molecules, two isoelectronic species, at the 1s edge of neon and the O 1s edge of the water. The experiments were conducted at the PLEIADES beamline at SOLEIL, which provides access to the soft X-ray energy range with high energy resolution and variable polarization. A high-resolution hemispherical electron analyzer was used to record the spectra. Slow photoelectron spectra were recorded using polarized light at selected energies above the ionization thresholds. Our observations reveal a significant PCI shift for both neon and water, which varies with photon energy. Additionally, photoelectron spectra exhibit a profile distortion that depends on the polarization of the incoming x-rays. PCI extends beyond energy exchange between the photoelectron and the Auger electron in the residual ionic field, also involving angular momentum exchange [2]. PCI significantly affects the angular asymmetry parameter, β, of the photoelectron at energies near the ionization threshold. Theoretical models [4] developed for angular momentum transfer showed significant discrepancies with our experimental findings. Notably, PCI effects are stronger in water molecules compared to neon atoms. This difference is tentatively attributed to the larger charge distribution in molecules after decay. Interestingly, in the case of neon 1s ionization, we observed the recapture of photoelectrons into higher Rydberg states due to considerable energy exchange. This highlights the complexity of PCI, which is influenced by both photon energy and polarization.

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Parallel Session Schedule

Geosciences / Environment / Cultural Heritage

PHENIX Room

Chairpersons: Charlotte Catrouillet, Simona Raneri

IT-07	Asteroid Ryugu Chemica characterization from Synchrotron spectroscopy in the mid to far infrared, and AFMIR, of Hayabusa 2 samples ***Emmanuel Dartois***
OC-19	A BAG for major-to-trace elemental mapping and speciation of flat fossils at the PUMA beamline ***Pierre Gueriau***
OC-20	Cu Redox speciation at magnetite surface: A Cu $L_{3,2}$-Edge study ***Jaimy Scaria***
OC-21	Determination of optical parameters of water ice using infrared Synchrotron Radiation: Characterisation of ice clouds emission ***Cecilia Taverna***
IT-08	Operando VIS-NIR microscopic hyperspectral imaging to monitor possible SR X-ray damage on paintings ***Amelia Suzuki***
OC-22	SR-XRPD investigation of artificially and naturally aged weighted silk fibers ***Alina Krotova***
OC-23	The magic scroll of the future Ampus museum of writing. The story continues... ***Jean-Paul Itié***
OC-24	Counterfeiting silver coins, from Roman republic to middle age ***Vincenzo Palleschi***

Asteroid Ryugu Chemical Characterisation from Synchrotron Spectroscopy in the Mid to Far Infrared, and by AFMIR, of Hayabusa2 Samples

E.Dartois[1], Y. Kebukawa[2], H. Yabuta[3], J. Mathurin[4], C. Engrand[5], J. Duprat[6], L. Bejach[5], A. Dazzi[4], A. Deniset-Besseau[4], C. Sandt[7], F. Borondics[7] and IOM and Stone Initial Analysis Teams

[1] Institut des Sciences Moléculaires d'Orsay, CNRS, Univ. Paris- Saclay, 91405 Orsay, France
[2] Department of Chemistry and Life Science, Yokohama National University, 79-5 Tokiwadai, Hodogaya-ku, Yokohama 240-8501, Japan.
[3] Department of Earth and Planetary Systems Science, Hiroshima University, Higashi-Hiroshima, Hiroshima 739-8526, Japan
[4] Institut Chimie Physique (ICP), UMR 8000, Université Paris-Saclay, CNRS, Orsay, 91405, France
[5] IJCLab, UMR 9012, Université Paris-Saclay, CNRS, 91405 Orsay, France
[6] Institut de Minéralogie, Physique des Matériaux et de Cosmochimie, Museum National d'Histoire Naturelle, CNRS, Sorbonne Université, Paris 75231, France
[7] Synchrotron SOLEIL, CNRS, CEA, Paris-Saclay, France
And "IOM" and "Stone" Initial Analysis Teams

ABSTRACT

The 6th December 2020, the Hayabusa2 mission from the Japanese space agency (JAXA) returned samples to Earth, collected on the Cb-type dark carbonaceous asteroid Ryugu. For the first time, Hayabusa2 brought back samples from the surface of a known carbonaceous asteroid. These Solar System objects preserved ancient material that escaped planetary accretion and differentiation, providing genuine mineral and organic phases formed within the first million years after the proto-Sun gravitational collapse. The amount of material collected by Hayabusa 2 (5.4g) is about 50 times larger than the one hundred mg planned. The InfraRed (IR) analyses of these samples provide unprecedented information on the formation and early evolution of the Solar System and allow to make the link between primitive interplanetary material and astronomical observations. A key goal of such studies initiated by the "IOM" and "Stone" Initial Analysis Teams is to elucidate the distributions and chemical characteristics of macromolecular organic materials and minerals in a C-type asteroid. In this work, we provide hyperspectral analyses showing the content and distribution of both the organics and minerals of Hayabusa2 particles that enables the comparison with other types of asteroidal and cometary samples available in the laboratory.

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A BAG for Major-to-trace Elemental Mapping and Speciation of Flat Fossils at the PUMA Beamline

P. Gueriau (1, 2) and S. Schöder (3)

(1) Université Paris-Saclay, CNRS, ministère de la Culture, UVSQ, MNHN, Institut photonique d'analyse non-destructive européen des matériaux anciens, Saint-Aubin 91192, France
(2) Institute of Earth Sciences, University of Lausanne, Géopolis, Lausanne CH-1015, Switzerland
(3) Synchrotron SOLEIL, L'Orme des Merisiers, Gif-sur-Yvette, France

ABSTRACT

Synchrotron-based X-ray fluorescence major-to-trace elemental mapping and chemical speciation of elements of interest using X-ray absorption spectroscopy are increasingly used for the anatomical and/or taphonomic study of flat fossils. The standard route for obtaining beamtime at the synchrotron can, however, prove complicated for 'novice' palaeontologist users and is poorly adapted to the timing of some palaeontological research: calls for proposals are highly technical and typically require 6–12 months from submission to experiment. As an alternative, we used the 'block allocation group (BAG)' route to implement facilitated access at the PUMA beamline. This BAG aims to provide access for studies relying on only one or too few fossils to justify individual beamtime, as well as easier and more efficient access for new groups, while also benefiting the 'synchrotron palaeontology' community by enhancing synergy among existing 'expert' user groups. In this contribution, we will describe the objectives of this BAG, with particular emphasis on the expected gains in terms of data processing and interpretation—especially of X-ray absorption spectra—and present the envisioned workflow through examples from recently collected data. We will also address possible future directions.

Cu Redox Speciation at Magnetite Surface:
A Cu $L_{3,2}$-Edge Study

J. Scaria,[1] M. Pédrot,[1] F. Choueikani,[3] L. Fablet,[1]
A-C. Pierson-Wickmann,[1] Y. Sivry,[2] C. Catrouillet,[2]
A. Dia,[1] and R. Marsac[2]

[1] Univ Rennes, CNRS, Géosciences Rennes – UMR 6118, F-35000 Rennes, France
[2] Université Paris Cité, Institut de physique du globe de Paris, CNRS, F-75005 Paris, France
[3] Synchrotron SOLEIL, l'Orme des Merisiers, Départementale 128, 91190 Saint-Aubin, France

ABSTRACT

Copper is an essential micronutrient, abundantly found in diverse rocks and minerals, yet exhibits toxicity at elevated concentrations. The behaviour and fate of copper in both aquatic and terrestrial environments may be influenced by the redox active iron oxide, such as magnetite (Fe_3O_4) nanoparticles. However, the molecular scale mechanisms involved between Cu and Magnetite nanoparticles in aqueous solutions are still poorly understood. Especially, stoichiometric magnetite (R = Fe(II)/Fe(III) = 0.5) may partially oxidize to non-stoichiometric one ($0 < R < 0.5$) or to maghemite (R = 0). Several studies established the strong influence of magnetite stoichiometry either on its redox reactivity[1] and or on its surface complexation properties[2] towards contaminants. However, their combined effects on the thermodynamics of Cu redox transformation at magnetite surface has not been considered.[3]

This study examines the interaction Cu with 10 nm-sized magnetite exhibiting different stoichiometries.[2] The effect of solution pH (~3 to ~10), initial Cu(II) concentration (25 µM and 500 µM), and dissolved Fe(II) ions (($[Fe(II)]_{aq}$ ~1000 µM) were additionally studied. The Cu speciation was monitored in the aqueous phase, by using ion selective electrode and ICP-MS, and at the magnetite surface by using soft X-ray absorption spectroscopy (XAS) and magnetic circular dichroism (XMCD) at the Cu and Fe $L_{2,3}$-edges on DEIMOS beamline at SOLEIL Synchroton (France).

The surface Cu redox speciation depended strongly on R. At pH = 8, where all Cu was adsorbed to magnetite, Cu(II) prevailed onto oxidized magnetite (R = 0.1), but was almost completely reduced to Cu(I) when R = 0.5. High [Cu] (0.5 mM) promoted the formation of small Cu(0) amounts, which could be enhanced by the addition of an excess of dissolved Fe(II), while Cu(I) appeared stabilized by surface complexation in such reducing condition at lower Cu levels. Cu adsorption to stoichiometric magnetite was drastically affected by solution pH. Both solution and surface speciation revealed the co-occurrence of Cu(II) and Cu(I), for pH < 8, with no or negligible Cu(0) species. The [Cu(II)]/[Cu(I)] values increased with decreasing pH values, in agreement with the expected pH-redox potential conditions in magnetite suspensions.[4]

This study reveals the complex interactions between Cu and magnetite nanoparticles, with significant implications for understanding Cu biogeochemistry and developing effective remediation strategies.

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Determination of Optical Parameters of Water Ice Using Infrared Synchrotron Radiation: Characterisation of Ice Clouds Emission

C. Taverna [1], J.-B. Brubach [1], M. Verseils [1], L. Labonnote [2],
Q. Libois [3], J.-L. Dufresne [4], R. Roehrig [3], P. Roy [1]

[1] Synchrotron SOLEIL, [2] Laboratoire d'Optique Atmosphérique, [3] Centre National de Recherches Météorologiques, [4] Laboratoire de Météorologie Dynamique

ABSTRACT

The spectral distribution of the infrared energy emitted by Earth is of utmost importance for an accurate study of the planet's energy budget, especially under climate change. In this context, ice clouds are among the atmospheric constituents that have the largest impact on the infrared emission of Earth. To accurately estimate their radiative properties, a detailed knowledge of the ice complex refractive index (CRI) is required, as it fundamentally controls the cloud-radiation interactions. However, the ice CRI is still poorly known, especially in the far-infrared (FIR, 100-700 cm^{-1}) and for temperatures of interest for atmospheric ice clouds (170-270 K), for which the CRI variation is expected to be significant. This lack of data over a critical temperature range is due to experimental issues. Most of the previous experiments on the subject used direct condensation of water vapor on a cold finger (around 100 K) to create ice film, making it impossible to reach temperatures above 195 K due to the sublimation of ice at this level of vacuum (around 10^{-6} mbar). To overcome the technical limits of previous studies, we developed a specific cell allowing the formation of ice films from 10 K up to 270 K. This new cell combined with the synchrotron radiation of the AILES beamline allows to obtain transmission spectra in all the infrared range available with optimized signal to noise ratio. Furthermore, with our setup we are also able to directly measure the fundamental parameters of the experiment (thickness of the ice film and temperature on the sample) during the experience, reducing to the minimum the uncertainties on the CRI retrieval and temperature evolution. Finally, we implemented a retrieval algorithm for extrapolating the CRI of a material from transmission measurements that considers all the specificity of the setup (multilayer system). Thanks to this approach and the developed setup we were able to obtain for the first time the refractive index of water ice in the far-infrared in the temperature range 150-270 K from experimental data. In this presentation we will present new optical indexes in a wide temperature range and will compare them with the data available in the literature.

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Operando VIS-NIR Microscopic Hyperspectral Imaging to Monitor Possible SR X-ray Damage on Paintings

A. Suzuki [1,3], C.S. Cheung[1], F. Di Benedetto[2], C. Riminesi[3],
S. Schoeder[4], L. Tranchant[4] and H. Liang[1]

(1) School of Science and Technology, Nottingham Trent University, Nottingham NG11 8NS, (UK)
(2) Department of Physics and Earth Science, University of Ferrara, via Saragat 1, Ferrara (Italy)
(3) Institute of Heritage Science, ISPC-CNR, via Madonna del Piano, 10 Sesto Fiorentino (Italy)
(4) Synchrotron SOLEIL, l'Orme des Merisiers, Saint-Aubin, BP 48, 91192 Gif-sur-Yvette (France)
email address: amelia.suzuki@ispc.cnr.it

ABSTRACT

Synchrotron X-ray (SR) techniques (mainly X-ray diffraction XRD, fluorescence XRF, and absorption XAS) are invaluable for the characterization of precious and heterogeneous artists' materials, thanks to low detection limits, high lateral resolution and exceptional chemical sensitivity. However, their application in the Cultural Heritage (CH) field is hindered by the higher damage probability of SR X-ray radiations, which are orders of magnitude more intense and brighter than conventional laboratory sources, hence the importance of assessing the safe limits of dosage has been recognised with more efforts devoted to such research [1,2]. Still little is known regarding the characteristics and reversibility of alterations induced on paintings by SR X-rays. The current mitigation strategies (e.g. cryogenic and/or vacuum environment, and sample spinning) are mainly focused on the assessment of the reliability of the data collected rather than the integrity of the object. The monitoring of spectral change over time during XAS measurements is a common practice, but sometimes the method fails in the detection of some types of damage [3]. Therefore, it is important to develop complementary monitoring methods capable of detecting subtle, early-warning signs of beam damage.

Here we present our in house developed VIS-NIR microscopic hyperspectral imaging system and its successful alignment with the X-ray micro beam at the PUMA beamline at Soleil synchrotron [4]. *Operando* VIS-NIR hyperspectral imaging was performed during a synchrotron experiment to assess the possible beam damage while performing XAS for the speciation of painting's materials. Common artists' pigments that are known to be sensitive to radiation damage were selected for painting mock-ups (lead white, vermilion, Prussian blue and realgar) with different binders (linseed oil and animal glue). Commonly used energies for CH materials characterisation were tested on the mock-ups varying flux density, beam spot and fluence. This monitoring method contributes to the improvement of analytical protocols that are necessary to reassure and support the increasing SR users community from the CH field and it represents a valuable strategy to preserve the integrity of the artworks analysed in large-scale facilities. We expect that this method could be incorporated for routine safe operation of SR X-ray techniques on real historical materials and beyond.

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SR-XRPD Investigation of Artificially and Naturally Aged Weighted Silk Fibers

A. Krotova[1,2], C. Vettorazzo[1,2], E. Avranovich Clerici[2], A. Gestels[2],
K. Janssens[1,2], N. Ortega Saez[1], G. Van der Snickt[1,2]

1 ARCHES group, University of Antwerp, Mutsaardstraat 31, 2000 Antwerp, Belgium
2 AXIS group, University of Antwerp, Groenenborgerlaan 171, 2020 Antwerp, Belgium

ABSTRACT

Treating silk fibers with metal salts to increase their weight and drapeability was a common practice in Europe in the 19th and 20th century. Today, such treatment, known as weighting, is believed to accelerate the deterioration and loss of unique mechanical properties of historical silks [1]. The data we previously obtained by means of in situ elemental analysis in museum collections seems to support this hypothesis as 60% of damaged fabrics contain elements typically used in weighting [2]. Nevertheless, some salt-treated objects remained well-preserved, suggesting that the degradation process is more complex than often assumed. Relatively little research has been done to explain the mechanisms of the degradation of salt-treated silks on the microscopic level, with most of the published works describing analyses performed at the macro scale [3]. This knowledge gap leads to difficulties in reporting the state of conservation to the cultural heritage community and choosing efficient conservation treatment for such material.

Since the decay of mechanical properties of weighted silk fabrics has been linked to the loss of the alignment of crystallites within the main silk protein fibroin [4], investigating the effect of weighting and aging on the preferential orientation within crystalline part of a single fiber can give more insights into the degradation mechanisms of salt-treated silks.

To evaluate the applicability of X-ray diffraction methods in addressing this issue, the first systematic study of salt-treated silk threads from degraded historical fabrics and corresponding artificially aged model samples was conducted at the PUMA beamline at SOLEIL, following a preliminary characterization at the University of Antwerp using MA-XRPD with conventional Cu-Kα source and SEM-EDX. Areas of 42 x 50 µm2 were scanned along single fibers using an X-ray beam with a focal spot of around 5 µm, energy of 18 keV with exposure time of 200 s. By collecting the resulting µ-XRPD and µ-XRF signals, the effect of artificial or natural aging of weighted silk threads was explored using radial and azimuthal intensity profiles. The obtained results could be used for the improvement in investigation of microstructural changes in salt-treated silk fibers using XRPD, and deepens the understanding of their degradation path, which is an essential step in the future development of conservation treatments.

The research was conducted in frames of SAFESILK project (Flanders Research Foundation, G060422N and the Slovenian Research Agency, project N1-0250, program P6-0283). The authors are grateful to the beamline scientists Dr. Sebastian Schröder and Dr. Katharina Müller for their help in planning and conducting the measurements.

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The Magic Scroll of the Future Ampus Museum of Writing. The Story Continues…

A. King[1], J-P. Itié[1], S. Schoeder[1], L. Tranchant[1], F. Pizzorni-Itié[2],
L. Bachelot[2], M. Gorea[3], C. Chilli[2]

1Synchrotron SOLEIL, 2Musée des Écritures Ampus, 3Laboratoire Arscan UMR 7041

ABSTRACT

The magic scroll belongs to the collection of the future “Musée des Ecritures” in Ampus (Var, France). A few engraved characters can be observed on the outside of the roll (fig. 1a). Due to the corrosion of the metal, it is impossible to unroll it physically without damage.

To read the scroll non-invasively, an initial tomographic study was performed on the PSICHÉ beamline. Due to the unexpectedly strong absorption of the metal, we were forced to increase the photon energy to the maximum available 120 keV in pink beam mode and develop a modified acquisition strategy. The metal is lead, confirmed by fluorescence measurements performed at the PUMA beamline (fig. 1b). These reveal flakes of copper alloy on the surface of the scroll, indicating that it was probably stored in a bronze container, now lost.

A second beamtime in 2024 allowed the object to scanned completely, revealing that the roll is made up of two engraved sheets (fig. 1c). A tomograph of the full axial length of the scroll (~45mm) was acquired using a helicoidal scanning acquisition. The scroll has been virtually unrolled using Python routines and visualized using Avizo software. The reconstruction shows that both faces of the metal foil are engraved with text. The engraved text has been identified as Mandean, revealing the origins of the artefact in Mesopotamia, and indicating its age as 3-5th century CE. An initial translation of the start of the text indicates that the scroll is a protective amulet for a pregnant woman. The complete translation is in progress, and the latest results will be presented at the meeting.

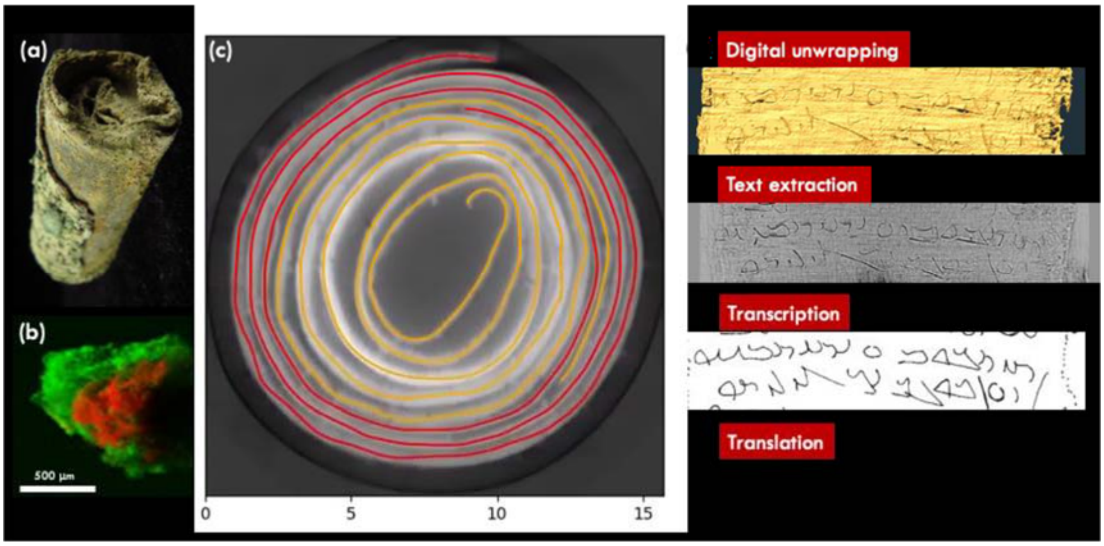


Figure 1: (a) Image of the magic scroll. (b) X-ray fluorescence map of a corrosion particle, red=copper, green=lead. (c) Tomographic slice, showing that the roll is made of two sheets of metal, overlaid with the trace used for virtual unwrapping. (d) Steps of the data analysis and interpretation of the text.

Counterfeiting Silver Coins, from Roman Republic to Middle Age

M. Baldassarri[1], A. Gianoncelli[2], V. Palleschi[3], S. Raneri[4]

[1] *Department of Historical Studies, University of Milan, Milan (Italy)*
[2] *Elettra Synchrotron, Area Science Park, Basovizza, Trieste (Italy)*
[3] *Laboratory of Applied and Laser Spectroscopy – ICCOM/CNR, Pisa (Italy)*
[4] *Department of Earth Sciences, University of Florence, Florence (Italy)*

ABSTRACT

The counterfeiting techniques of several silver coins, going in time from Roman republic to Middle Age, were studied at the PSICHÉ beamline of the SOLEIL synchrotron. We exploited the possibility of obtaining high-resolution tomographic images for determining the thickness and the technique of deposition of the silver layer that the forgers used to put over a core of vile metal (copper) for gaining advantage of the counterfeiting.

The comparison between a forged Roman denarius (108-107 BCE) and a Medieval *quattrino* of Lucca (coined around 1510), found in the remains of a clandestine mint in Godano (La Spezia, Italy), evidenced that the former was obtained by applying a thin layer of silver on a copper base. The thickness of the layer is uniform, suggesting the application of a silver lamina on the copper planchet before the coinage. On the other hand, the medieval coin evidenced on the surface the presence of a thinner and uneven silver layer, which becomes more evident when compared with a genuine coeval *quattrino*.

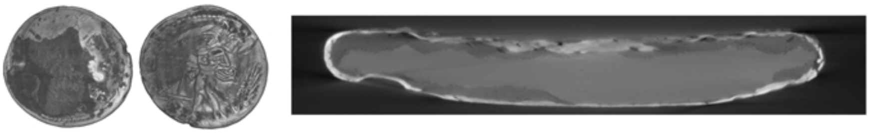


Fig. 1 – Counterfeit L. Valerius Flaccus Denarius, 108-107 BCE. At the right, the tomographic image showing the silver layer on the surface.

Fig. 2 – Left: Counterfeited *quattrino* of Lucca. Right: coeval genuine *quattrino* of Lucca.

The nature of the layer on the surface of the counterfeit *quattrino* was determined using the beamline ED X-Ray Fluorescence and diffraction setup as an amalgama of silver and mercury. The combination of appropriate slits on the beam and on the detector allows the acquisition of fluorescence and diffraction spectra with a resolution of 20×20×200 μm^3.

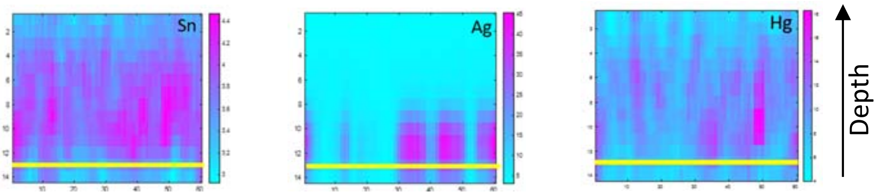


Fig. 3 – Depth elemental maps on the counterfeited *quattrino* of Lucca. The yellow line represents the surface of the coin. The tin map spans uniformly over the core of the coin, while silver and mercury are present only near the surface where the Ag+Hg layer was deposited, probably by dipping the coin in the amalgama.

Parallel Session Schedule

Physics & Advanced Materials (Structural, electronic and magnetic properties of matter / Physical surfaces and interfaces)

SOLEIL Auditorium - Main Building

Chairpersons: Debora Pierucci, Fabien Cheynis, Sara Gonzalez

IT-09	Atomic structure of novel 2D materials unveiled by Surface X-ray diffraction ***Geoffroy Prevot***
OC-25	Unveiling electric field distribution in quantum dotbased devices by operando scanning photoemission microscopy ***Dario Mastrippolito***
OC-26	Mapping of stress/strain distribution within chromium layers deposited on Zr substrates at the ESRF facility ***Clément Ribart***
OC-27	Development of a high-throughput XRD wide-surface furnace for In Situ hydration study of proton conductor electrolytes ***Giulio Cordaro***
IT-10	Structure and magnetic properties of FeRh nanomagnets deposited on perovskite surfaces ***Florent Tournus***
OC-28	On the cuprates’ universal waterfall feature: Evidence of a momentum-driven crossover ***Benjamin Bacq-Labreuil***
OC-29	A combination of real-time diagnostics probing the impact of nitrogen on silver thin film growth ***Michał Kamiński***
OC-30	Inverse freezing and melting of FeGd ferrimagnetic domains at the spin-reorientation transition ***Matthias Riepp***

Atomic structure of novel 2D materials unveiled by Surface X-ray diffraction

Geoffroy Prévot

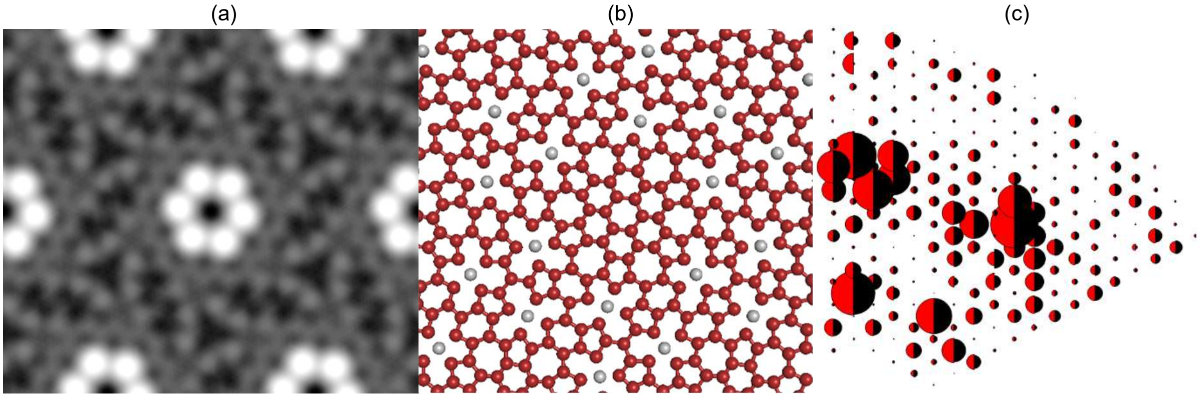
Sorbonne Université, CNRS, Institut des Nanosciences de Paris, INSP, F-75005 Paris, France

ABSTRACT

Unlike graphene or transition metal dichalcogenides, some 2D materials cannot be obtained by exfoliation of a lamellar crystal. This is for example the case of blue phosphorene, or of the group IVA Xenes such as silicene, germanene, stanene and plumbene. Such novel 2D materials can however be synthesized through CVD or PVD methods on suitable substrates.

As they have no bulk counterpart, the knowledge of their atomic structure is a key point for the understanding of their properties (electronic, optical, mechanical, etc.). For this purpose, surface X-ray diffraction is by far the most quantitative method. Indeed, the diffracted intensities measured in reciprocal space are proportional to the squared modulus of the Fourier transform of the electronic density.

In this talk, I will present the classical methods used to retrieve the atomic positions from the diffraction measurements, the recent developments achieved with phase retrieval algorithms (see Figure) and some perspectives, applied to the case of epitaxial silicene and germanene layers recently synthesized on metal substrates, and measured on the SixS beamline.[1–7]



"Germanene" layer on Ag(111). a) STM image (6.0 x 5.2 nm^2). b) corresponding atomic model (red: Ge atoms, grey: Ag atoms). c) comparison between experimental (red) and theoretical (black) in-plane structure factors.

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Unveiling Electric Field Distribution in Quantum Dot-based Devices by Operando Scanning Photoemission Microscopy

D. Mastrippolito[a,b], M. Cavalloa, E. Bossavit[a,b], P. Dudin[b], Y. Prado[a],
J. Avila[b], D. Pierucci[a], E. Lhuillier[a]

[a]*Sorbonne Université, CNRS, Institut des NanoSciences de Paris, INSP, F-75005 Paris, France*
[b]*Synchrotron SOLEIL, L'Orme des Merisiers, Départementale 128, 91190 Saint-Aubin, France*

ABSTRACT

Colloidal quantum dots (CQDs), with their broadband spectral tunability, are becoming a well-established building block for solution-processable electronic and optoelectronic devices. As device architectures become more sophisticated and miniaturized, local probing techniques are needed to investigate their electronic structure at the nanoscale, especially under realistic operational conditions (where materials are integrated with electrodes and subjected to applied electric fields). In this talk, we explore the potential of scanning photoemission microscopy at the ANTARES beamline[1] to study infrared CQD-based devices under *operando* conditions. This technique exploits the sub-micron spatial resolution of the focused soft X-ray beam to map the energy landscape in detail while biasing the devices *in situ*, directly correlating energy shifts with device geometry[2]. Beyond scalar energy mapping, the technique high resolution provides direct access to the current flow pathway and the vectorial distribution of the electric field in the CQD film, enabling its quantification and further optimization of involved device geometry[3,4]. Our findings demonstrate that this method can be effectively applied to various devices, including transistors and integrated circuits, proving valuable for nanoelectronics with applications extending beyond CQD-based device technologies.

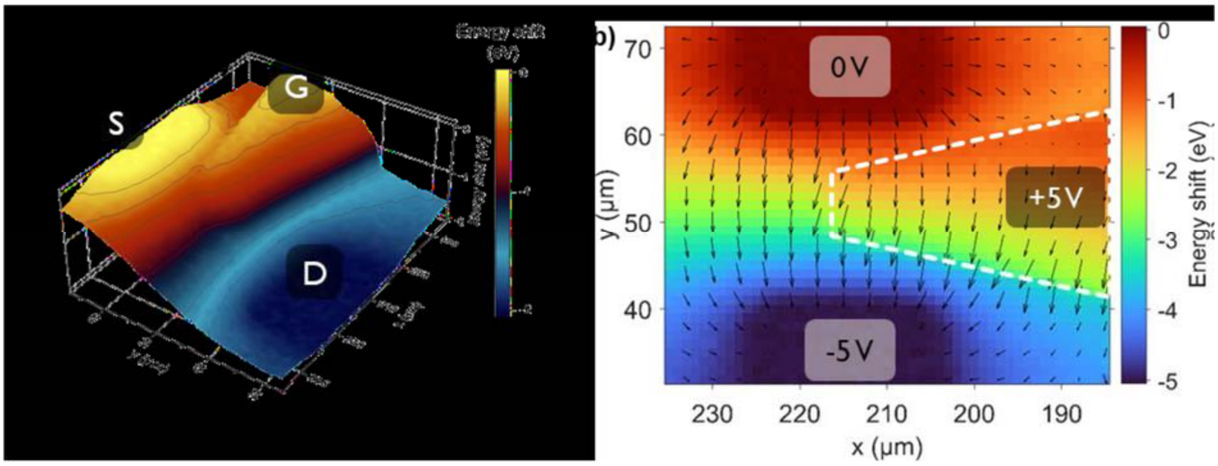

Figure 1. (a) Energy landscape of a back-gated field effect transistor (V_{DS}=−2 V and V_{GS}=+5V). (b) Energy shift map (color map) and electric field map (arrow plot) in which the fielddirection and magnitude are given by the arrow for each position (V_{DS}=−5 V and V_{GS}=+5 V).

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Mapping of Stress/strain Distribution within Chromium Layers Deposited on Zr Substrates at the ESRF Facility

C. Ribart[1], J-S. Micha[2], R. Guillou[3], S. Tardif[1], J. Eymery[1], J-L. Béchade[4]

[1] *Univ.Grenoble Alpes, CEA Grenoble, IRIG, MEM, NRX, 17 rue des Martyrs, F-38000, Grenoble, France*
[2] *Univ.Grenoble Alpes, CNRS, UMR SyMMES CEA/IRIG, 17 avenue des Martyrs, Grenoble 38000, France*
[3] *Univ Paris-Saclay, CEA, SRMA, 91191 Gif-sur-Yvette, France*
[4] *Univ.Paris-Saclay, CEA, SRMP, 91191 Gif Sur Yvette, France*

ABSTRACT

Following the Fukushima events, there is a strong international concern to develop enhanced Accident Tolerant Fuel (ATF) nuclear fuel cladding materials that would exhibit enhanced resistance to hypothetical accidental conditions, such as “LOss of Coolant Accident” (LOCA). The development of Cr-coated zirconium-based alloys is being investigated by the CEA, EDF and FRAMATOME as a near-term solution since it is an evolution of the standard zirconium-based cladding [1], [2]. The coating serves as a protective oxidation barrier by improving the mechanical properties of the cladding following “high temperature steam oxidation” (i.e. ductility and strength) simulating LOCA and slightly beyond conditions. However, Cr-coatings exhibit a specific microstructure characterized by a strong crystallographic texture and internal stresses, which differs from conventionally manufactured (bulk) chromium. Depending on the deposition parameters which may affect the microstructure of the coating, mechanical properties of chromium thin film have to be characterized in details. Recent XRD and FIB-DIC measurements have already been able to give insight about the residual stresses in the coating [3]. This contribution aims to refine the characterization of the stress/strain distribution around the Zr-Cr interface by taking advantage of the scanning micro-diffraction Laue set-up, available on BM32 beamline at the ESRF facility [4], which allows to probe locally, in-depth and non-destructively these microstructures. One as-deposited configuration of interest was selected with planar and cylindrical specimen geometries. In-plane raster maps were scanned using the conventional µLaue technique from which contributions of each crystallographic phase can be discriminated, allowing to reconstruct integrated orientation and strain fields for each grain along the illuminated depth. Subsequently, regions of interest can be identified and probed with the depth-resolved DAXM variant [5] to get micrometric voxelized fields through the thickness. Here, preliminary results will be shown, providing fresh perspectives on the internal local stress spatial distributions within the wall thickness of the Cr-coating.

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Development of a High-throughput XRD Wide-surface Furnace for *In Situ* Hydration Study of Proton Conductor Electrolytes

G. Cordaro[1], J. Sirvent[2], O. Rahmouni[1,3], F. Baiutti[2], C. Mocuta[4],
D. Thiaudière[4], A. Tarancón[2], G. Dezanneau[1]

[1]Université Paris-Saclay, CentraleSupélec, CNRS, Lab. SPMS, Gif-sur-Yvette 91190, France
[2]Department of Advanced Materials for Energy Applications, Catalonia Institute for Energy Research (IREC), Jardins de les Dones de Negre 1, Barcelona 08930, Spain
[3]Univ. Lille, CNRS, Centrale Lille, Univ. Artois, UMR 8181 – UCCS, F-59000 Lille, France
[4]Synchrotron Soleil, Gif-Sur-Yvette 91192, France

ABSTRACT

Discovering efficient and innovative materials for protonic ceramic cells requires the characterization of countless compositions. The knowledge of hydration thermodynamics and electrochemical performances is essential for understanding the proton conduction mechanisms and selecting the most promising electrolyte materials. However, traditional measurements are performed on individual samples and require extremely long stabilization times. A high-throughput approach was chosen to evaluate the electrochemical performance of hundreds of compositions inside the $Ba(Ce,Sn,Zr)_{0.8}Y_{0.1}Yb_{0.1}O_{3-\delta}$ (BCSZYY) ternary system. Thin film combinatorial libraries were produced by pulsed laser deposition on 100 mm diameter wafers. The composition gradient is obtained through alternate depositions of $BaCe_{0.8}Y_{0.1}Yb_{0.1}O_{3-\delta}$ (BCYY), $BaSn_{0.8}Y_{0.1}Yb_{0.1}O_{3-\delta}$ (BSYY), and $BaZr_{0.8}Y_{0.1}Yb_{0.1}O_{3-\delta}$ (BZYY) centered on three opposite edges of the substrate to recreate the ternary diagram. XY-resolved characterization techniques were performed to map the elemental, structural, and hydration properties of each composition inside the BCSZYY ternary system. A custom-made furnace was specifically developed to perform high-temperature XRD measurements on wide-surface samples in dry and wet conditions to measure the effect of proton incorporation on unit cell expansion.

In situ measurements were also performed at the DiffAbs beamline of the SOLEIL synchrotron by simultaneously collecting X-ray diffraction and fluorescence signals. XRF allowed the evaluation of the elemental distribution of the BCSZYY samples at room temperature and gave insight into thickness variation, complementary to spectroscopic ellipsometry[1]. Due to the large surface of the hotplate, a calibration of the radial thermal losses was performed through a platinum reference sample. With a setpoint of 735 °C, the distribution of the calculated temperature of the Pt deposition showed a maximum of 653 °C near the center and 80 °C lower at the edges. The BCSZYY sample was subsequently measured in dry and wet N_2 to calculate the thermal expansion curves and extract information about hydration thermodynamics for the entire BCSZYY ternary system. A custom-made setup for electrochemical impedance spectroscopy is under development to measure the electrochemical performance.

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Structure and Magnetic Properties of FeRh Nanomagnets Deposited on Perovskite Surfaces

F. Tournus[1], S. Gonzalez[2], I. Cañero-Infante[2], A. Reyes[1], G. Herrera[1], P. Capiod[1], O. Boisron[1], D. Le Roy[1], V. Dupuis[1], G. Saint-Girons[2], R. Bachelet[2], M. Bugnet[3], A. Resta[4], P. Ohresser[4], E. Otero[4], P. Schoeffmann[4], L. Martinelli[5], X. Weng[5], G. Renaud[5,6]

[1]Institut Lumière Matière (ILM), CNRS-Univ. Lyon 1, Villeurbanne;
[2]Institut des Nanotechnologies de Lyon (INL), CNRS-ECL-INSA-UCBL-CPE, Villeurbanne;
[3]Matériaux, ingénierie et science (MATEIS), CNRS-INSA, Villeurbanne;
[4]Synchrotron SOLEIL, Saint-Aubin;
[5]Institut Néel, CNRS-UGA, Grenoble;
[6]IRIG-MEM, CEA-UGA, Grenoble

ABSTRACT

Multiferroic composites are promising systems where the interfacial coupling between different ferroics can be driven by magnetic, voltage and strain mediation approaches to explore new technological applications [1]. Bulk FeRh alloy chemically ordered in the CsCl-type B2 phase, is characterized by a remarkable magneto-structural transition close to room temperature that can be controlled through an external stimulus for thin films on perovskite substrates [2]. As in nanoscale systems the magnetic order is very sensitive to interfaces, strain and surface terminations [3,4], we have studied FeRh nanocrystals on perovskite oxides substrates. Here, we focus on 3 to 7 nm FeRh size-selected particles, prepared by low-energy cluster beam deposition (laser vaporization) and deposited under ultrahigh vacuum on $SrTiO_3$ (001) or thin film of $BaTiO_3$ epitaxially grown on STO.

While the particles are initially in the fcc chemically disordered phase A1, after annealing we observe by grazing incidence X-ray diffraction preferential orientation of B2 FeRh nanocrystals on the surface, with unconventional epitaxy relationships [5]. The particles are found to remain in the ferromagnetic state down to low temperature, as for FeRh nanomagnets embedded in carbon. Concerning the metallicity of Fe atoms, which is intimately linked to the magnetic properties, we observe that unprotected FeRh particles are (partially) oxidized but can be reduced by annealing at a moderate temperature, lower than the one necessary to promote the chemical order transition from the A1 to B2 phase of FeRh. This reversible process has been established using laboratory in situ X-ray photoelectron spectroscopy investigations (XPS), and has also been observed using X-ray absorption spectroscopy (SOLEIL synchrotron), simultaneously to X-ray magnetic circular magnetic dichroism to follow the impact on the magnetic properties. In addition, the effect of in situ annealing and oxidation has been followed on the intensity of grazing incidence X-ray diffraction FeRh peaks, confirming that the oxidation is occurring at the outer surface of the metallic clusters rather that at the interface with the oxide substrate. These results illustrate the specific size effects that can be met, both for the structure and the magnetic properties of bimetallic nanomagnets.

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On the Cuprates’ Universal Waterfall Feature: Evidence of a Momentum-driven Crossover

B. Bacq-Labreuil, C. Fawaz, S. Biermann, M. d’Astuto and B. Lenz

CPHT, CNRS, Ecole Polytechnique, Institut Polytechnique de Paris, F-91128 Palaiseau, France
Département de physique, Regroupement québécois sur les matériaux de pointe & Institut quantique
Université de Sherbrooke, 2500 Boul. Université, Sherbrooke, Québec J1K2R1, Canada
Univ. Grenoble Alpes, CNRS, Grenoble INP, Institut Néel, F-38000 Grenoble, France
European Theoretical Spectroscopy Facility, F-91128 Palaiseau, Europe IMPMC, Sorbonne
Université, CNRS, MNHN, 4 place Jussieu, F-75252 Paris, France

ABSTRACT

We present a study of two related universal anomalies of the spectral function of cuprates, so called waterfall and high-energy kink features, combining cellular dynamical mean-field theory and angle-resolved photoemission (ARPES) measurements for the oxychloride NaxCa2−xCuO2Cl2 (Na-CCOC) [1]. Tracing their origin back to an interplay of spin-polaron and local correlation effects both in undoped and hole-doped (Na-)CCOC, we establish them as a universal crossover between regions differing in the momentum dependence of the coupling and not necessarily in the related quasiparticles’ energies. The proposed scenario extends to doping levels coinciding with the cuprate’s superconducting dome and motivates further investigations of the fate of spin-polarons in the superconducting phase. This works leverages ARPES measurements carried out at the SOLEIL Cassiopee beamline.

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A Combination of Real-time Diagnostics Probing the Impact of Nitrogen on Silver Thin Film Growth

M. Kamiński[1], G. Abadias[2], D. Babonneau[2], A. Coati[3], Y. Garreau[3],
A. Michel[2], A. Resta[3], K. Solanki[2], A. Vlad[3] and B. Krause[1]

[1]Karlsruhe Institute of Technology (KIT), Institute of Photon Science and Synchrotron Radiation (IPS),
Hermann-von-Helmholtz-Platz 1, D-76344 Eggenstein-Leopoldshafen, Germany
[2]Institut Pprime, Département Physique et Mécanique des Matériaux UPR 3346 CNRS -
Université de Poitiers - ENSMA TSA 41123, 86073 Poitiers Cedex 9, France
[3]Synchrotron SOLEIL - Seminar Coordinator, L'Orme des Merisiers, Départementale 128,
91190 Saint Aubin, France

ABSTRACT

Application-related properties of thin films are closely related to their microstructure. Given that the structure of the thin film can evolve even under high vacuum conditions, commonly used *ex situ* diagnostics may yield misleading information. Therefore, a comprehensive understanding of the nanoscale mechanisms involved in thin film formation necessitates real-time techniques [1]. At the SIXS beamline (SOLEIL synchrotron) we performed simultaneously real-time grazing incidence small-angle x-ray scattering (GISAXS), grazing incidence diffraction (GID) and substrate curvature measurements to get information about polycrystalline thin film evolution during growth. In particular, GISAXS reveals changes in nanoscale morphology, GID gives insight into the crystallinity of thin films, and substrate curvature measurements provide information about the average intrinsic stress. With our methodology we can study the interdependence between stress state, thin film structure and morphology, using the quantitative information obtained from the scattering techniques. Since the influence of the substrate curvature can be crucial for grazing incidence condition x-ray techniques, we prove with rigorous calculations that in the curvature regime encountered in our experiment the effect on GISAXS is negligible.

The use of the real-time *in situ* diagnostics is demonstrated on the example of silver thin films deposited by magnetron sputtering. On weakly interacting oxide substrates (like SiO_x, silver tends to form 3D-structures, what prevents its application as transparent and conductive layers. It was reported that the use of gaseous additives, in particular nitrogen, promotes a formation of a continuous layer [2]. Using the information from all three techniques, we will discuss the impact of nitrogen on all growth stages (from initial stages of island nucleation, growth, and coalescence, up to formation of percolated and continuous films), including the relaxation of the film during growth interruptions.

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Inverse Freezing and Melting of FeGd Ferrimagnetic Domains at the Spin-reorientation Transition

M. Riepp[1,2], C. Rodriguez Cortez[3], M. Hennes[3], M. Hemili[1], T. Ferté[2], B. Vodungbo[1], E. Jal[1], C. Boeglin[2], M. Hehn[4], H. Popescu[5], N. Jaouen[5], and N. Bergeard[2]

[1]*Sorbonne Université, CNRS, Laboratoire de Chimie Physique—Matière et Rayonnement, LCPMR,75005 Paris, France*
[2]*Institut de Physique et Chimie des Matériaux de Strasbourg, Université de Strasbourg, CNRS, 67000 Strasbourg, France*
[3] *Sorbonne Université, CNRS, Institut des NanoSciences de Paris, 75005 Paris, France*
[4]*Institut Jean Lamour, Université de Lorraine, 54011 Nancy, France*
[5]*Synchrotron SOLEIL, Saint-Aubin, Boite Postale 48, 91192 Gif-sur-Yvette Cedex, France*

ABSTRACT

Ferrimagnetic FeGd-based alloys are promising candidates for novel types of ultrafast magnetic recording applications, switching the magnetization by all-optical means without the need of external magnetic field [1]. Up to date, information on all-optical switching in nanometer-sized magnetic domains of FeGd alloys is still scarce. We report on reversible geometric phase transitions of FeGd ferrimagnetic domains upon heating and cooling across the so-called spin-reorientation transition (SRT), where the effective magnetic anisotropy changes from out-of-plane (OP) to in-plane (IP). Using temperature-dependent x-ray resonant magnetic scattering (XRMS) at synchrotron SOLEIL beamline SEXTANTS, we find that the OP stripe-domain phase at room temperature turns into a more disordered labyrinthine domain phase at the SRT (T_{SRT} = 280 K) reducing OP demagnetizing energy at the cost of domain-wall (DW) energy. Extremely rare is the second phase transition back to the more ordered (IP) stripe-domain phase at T = 150 K that can be understood by the reduction of DW energy at the cost of IP demagnetizing energy, a phenomenon that is typically observed only in few-monolayer thin films named inverse freezing [1]. We find indications for an about 5 nm magnetic surface layer and identify counter-clockwise Bloch-type DWs in the stripe-domain phase employing XRMS in reflection geometry. A final answer to the origin of the geometrical phase transitions, however, will require additional structural investigations. Our results are the foundation for an upcoming time-resolved XRMS experiment, employing the femtosecond infrared laser currently installed at SEXTANTS, to bring new insights to the nature of ultrafast all-optical switching in FeGd-based alloys on nanometer length scales.

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PARALLEL SESSIONS
on TECHNIQUE / METHODOLOGY

Parallel sessions on technique / methodology

Tuesday, January 28th

X-ray Absorption Spectroscopy (soft / tender / Hard X-ray)

Chairpersons: Amélie Bordage (S2UO), Sara Gonzalez (S2UO), Delphine Vantelon (SOLEIL)

SOLEIL - Reception Building Auditorium

09:00 - 09:20	Introduction Beamlines presentation, overview and perspectives First feedbacks from the survey for XAS
09:20 – 10:00	Round table discussion / questions from the users

Imaging / Microscopy / Tomography

Chairpersons: Andrew King (SOLEIL), Simona Raneri (S2UO), Pierre Asselin (S2UO)

SOLEIL Auditorium - Main Building

09:00 - 09:10	Introduction ***Andrew King*** *– SOLEIL Synchrotron (PSICHE and SOLEIL referent for imaging techniques)*
09:10 - 09:30	Beamlines presentation, overview and perspectives
09:30 - 10:00	Round table discussion / questions from the users

Photoelectron Spectroscopies

Chairpersons: Debora Pierucci (S2UO), Fabien Cheynis (S2UO)

CEA BLOCH Auditorium

09:00 - 09:20 (15'+5')	Nano-ARPES facility at ANTARES beamline for direct electronic structure determination of 2D materials ***José Avila*** *- SOLEIL Synchrotron, St Aubin, France*
09:20 - 09:40 (15'+5')	TEMPO beamline: high-resolution and time-resolved photoemission spectroscopy from UHV to near ambient pressure ***Matthieu Silly*** *- SOLEIL Synchrotron, St Aubin, France*
09:40 - 10:00	Round table and discussion

Parallel sessions on technique / methodology

Tuesday, January 28th

Diffraction & Scattering / Coherence

Chairpersons: Florent Carn (S2UO), Benoît Masquida (S2UO)

PHENIX Room

09:00 - 09:10 (6'+4') ***Philippe Fontaine*** *– SIRIUS beamline*

09:10 - 09:20 (6'+4') ***William Shephard*** *– Proxima-2A beamline*

09:20 - 09:30 (6'+4') ***Thomas Bizien*** *– SWING beamline*

09:30 - 09:40 (6'+4') ***Alessandro Coati*** *– SixS beamline*

09:40 - 09:50 (6'+4') ***Dominique Tiaudière*** *– DiffAbs beamline*

09:50 - 10:00 (6'+4') ***Pierre Legrand*** *– Proxima-1 and Polaris beamlines*

Networks for students & postdoc

Chairperson: Charlotte Catrouillet (S2UO), Asma Tougerti (S2UO)

LIBRA Room

09:00 - 10:00

Nano-ARPES Facility at ANTARES Beamline for Direct Electronic Structure Determination of 2D Materials

J. Avila

SOLEIL Synchrotron, Saint-Aubin, France

TEMPO Beamline: High Resolution and Time Resolved Photoemission Spectroscopy from UHV to Near Ambient Pressure

M. G. Silly[1], S. Chaouchi[1], A. Bendounan[1], L. Dudy[1], P. Holander[1],
F. Picca[1], F. Bournel[2], J.-J. Gallet[2], D. Krizmancic[3]

[1] *Synchrotron SOLEIL L'Orme des Merisiers Départementale 128 91190 Saint-Aubin, France*
[2] *Laboratoire de Chimie Physique-Matière et Rayonnement (LCPMR) 4 place Jussieu*
Tour 43/44, Etage 1 75252 Paris Cedex 05, France
[3] *Officina dei Materiali - CNR-IOM Q2 Building - Area Science Park Strada Statale 14 km 163,5 -*
34149 Basovizza – Trieste, Italy

ABSTRACT

TEMPO, a soft X ray beamline dedicated to time resolved experiments, is optimized to perform pump-probe experiments ranging from seconds to sub-picoseconds time scales[1].Two Apple II undulators provide photons in the energy range 50-1500 eV with a variable polarization (linear to circular). Highly sensitive to organic chemistry (C, N, O), semiconductors (Si, Ge), transition metals and rare earth species, the energy range is well suited for studies on organic molecular systems on a large range of semiconductor and metal substrates. Two end stations provide a state-of-the-art apparatus to investigate the electronic properties of surfaces under various environment and excitation. On the main branch, the high photon flux is coupled to the excellent energy resolution of the MBS A-1 electron energy analyzer. A delay-line detector[2] allows us to investigate the dynamic behavior of excited states using photoelectron spectroscopy by measuring the evolution of electronic properties under laser excitation of selected chemical species at the surface. On the second branch, a near ambient pressure photoemission experiment permits to investigate the chemistry and the electronic properties of surfaces exposed to various gases at pressures up to 25mbars. The two experimental end stations are coupled to synchronized fs pulsed LASER covering the wavelength range between 400 and 800nm to perform operando measurement. The main results obtained and the perspectives on the main scientific subjects studied on the beamline will be presented.

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POSTERS SESSION

List of Posters

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PO-BIO-02	Flocculosine A - Liposaccharide isolé du champignon Anthracocystis flocculosa. Comment se débrouiller avec des fibres cristallines ***Thierry Prangé***
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PO-GEO-18-S	Organic matter exposed to an emerging contaminant: platinum (Pt) speciation changes ***Justine Le Doare***
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PO-PHY-26	Quarter wave plate galvanometer for fast polarisation switching in RIXS ***Jack Harrison***
PO-PHY-27	Ultrafast dynamics of magnetic anisotropy and magnetic structure in ferrimagnetic alloys CoTb. ***Mohamed El Moundji Hemili***

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PO-PHY-29	Charge transfer processes in functional materials studied with hard X-ray core-hole clock spectroscopy ***Fredrik O.L. Johansson***
PO-PHY-30-S	Mixtures of water, ammonia and methane under extreme pressure and temperature conditions ***Nour Aicha Kacem***
PO-PHY-31	Dichroism and SRARPES studies of W(321) and W(-3-2-1) ***Yves Lassailly***
PO-PHY-32-S	Structure and ferroelectric control of GeTe thin films using vicinal Si substrates ***Léa Meynier***
PO-PHY-33-S	Comparative study of hematite-based photoanodes using operando STXM and Raman spectroscopy - Methods and first results ***Meyssa Mockbel***
PO-PHY-34-S	Electronic structure study of alloys of 2D transition-metal dichalcogenides ***Harold Rojas-Páez***
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PO-PHY-36	In situ monitoring of yttrium films structural evolution under very low hydrogen pressure using X-ray diffraction at SIXS beamline ***Olivier Thomas***

Dimerization of a Self-cleaving Intronic Ribozyme May Control the Expression of a Protein Enhancing Memory in Mammals

T. D’andrea[a], A. Limani[b], B. Masquida[b]

[a] UPR 9002, CNRS - Université de Strasbourg, IBMC, 2 allée Konrad Roentgen, 67084 Strasbourg, France
[b] UMR 7156, CNRS - Université de Strasbourg, IPCB, 4 allée Konrad Roentgen, 67084, Strasbourg, France

ABSTRACT

The Cytoplasmic polyadenylation element-binding protein 3 (CPEB3) plays a crucial role in synaptic plasticity and long-term memory. Its structure, which includes a prion-like domain and RNA-binding domains, enables it to regulate neuronal protein expression by controlling mRNA maturation (1). The expression, and thus the activity, of CPEB3 is regulated by a self-cleaving ribozyme located within intron 2 of the gene. This ribozyme's activity has been shown to inhibit CPEB3 expression by cleaving the 5' end of the pre-messenger RNA, thereby preventing proper splicing. This HDV-like ribozyme uses its structural scaffold to carry out a transesterification reaction, facilitated by a catalytic site formed by the P1.1 mini-helix and the C75 residue. Inhibition of this catalytic site, using antisense oligonucleotides (ASOs) that bind by base pairing to the ribozyme cleavage site sequence, confirmed its regulatory role on the expression of CPEB3 and associated protein networks (2). SAXS analysis and native electrophoresis further confirmed a homodimeric conformation of the ribozyme in solution, originally observed in the crystal structure of the ribozyme (3). The homodimer is stabilized by an anticodon-like loop with a palindromic sequence (5'-ACGU-3') that could mimick the action of ASOs. The dimerization, by preventing the formation of the catalytic site, could inhibit the self-cleavage activity of the ribozyme. The evidence of this homodimerization in solution suggests the hypothesis that neuronal stimulation may increase the expression of the cpeb3 gene and thus the local quantity of RNA transcripts. The ribozyme concentration would then foster dimerization and further ribozyme cleavage inhibition resulting in increasing the expression of the CPEB3 protein.

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Flocculosine A - Liposaccharide isolé du champignon Anthracocystis flocculosa. Comment se débrouiller avec des fibres cristallines

T. Prangé[1], M. Ponchet[2], M. Mehiri[3], W. Shepard[4]

[1] CITCoM CNRS UMR 8038, Faculté de Pharmacie 75006 Paris, France.
[2] INRAE 1355, CNRS UMR 7254 Sophia Agrobiotech. 06903 Sophia Antipolis, France.
[3] CNRS UMR 7272 Université Nice Côte d'Azur, 06108 Nice, France. NIXE Sarl,
BP 291, 06560 Sophia Antipolis, France.
[4] Synchrotron SOLEIL,

ABSTRACT

La flocculosine A [1] est un métabolite à forte activité antifongique sécrété par le champignon *Anthracocystis flocculosa* (anciennement *Pseudozyma flocculosa*). Il cristallise sous forme d'aiguilles longues et souples semblables à de l'amiante. Ces fibres sont généralement observées en présence du mycélium dans le milieux de culture du champignon [2-3]. Toutes les tentatives de cristallisation de la floculosine A purifiée ont toujours donné lieu à des aiguilles minces, allongées et courbes, généralement d'une épaisseur inférieure à 5 microns. Les diagrammes de diffraction de ces cristaux (ligne microfocus PROXIMA 2A) montrent toujours des taches allongés, souvent multiples, rappelant une structure désordonnée. Nous rapportons comment la forme et la taille des plaques de cristallisation, et le gradient de température, influent sur la qualité des cristaux et comment de "bons" cristaux peuvent être finalement obtenus.
utilisables.

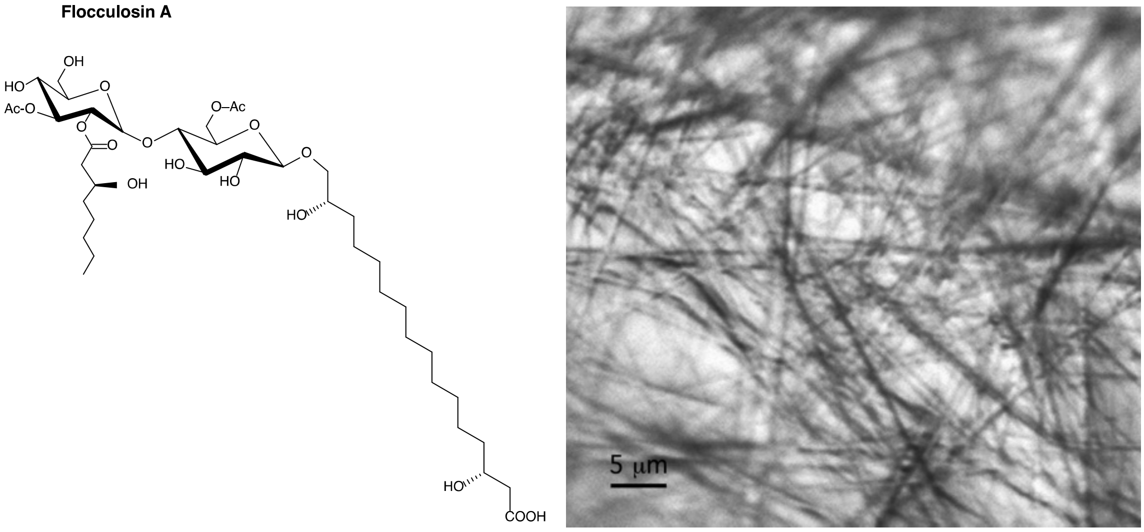

Figure 1. Flocculosin A et cristaux.

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Exploring Cryo-Correlative Light, Electron, and X-ray Microscopy (Cryo-CLEXM): Methods and Insights for Biological Imaging

H. Rista, C. Lumy, S. Kubsky, P. Legrand, P. Montaville and F. Jamme

Synchrotron SOLEIL, Experience division DISCO and PROXIMA1 beamlines

ABSTRACT

Cells and other biological samples are complex and heterogeneous systems. In order to fully comprehend such systems, it is necessary to explore them using both chemical and high-resolution ultrastructural information. To get this information, we use Cryo Correlative Light, Electron, and X-ray Microscopy cryo-workflow (Cryo-CLEXM). Each one of the methods involved in CLEXM or in one of its variations (CLEM, CLXM) spans a specific resolution range, and correlation becomes possible when these ranges overlap. The correlation allows for a deeper understanding of how biological function influences, and is influenced by, cellular structure and dynamics.

We used Cryo-CLEXM to study Liquid-Liquid Phase Separation (LLPS) in HEK293fs cells transfected with the XPA fluorescent protein expression vector and to investigate morphological changes in replication organelles in Huh7 cells infected with GLT1cc HCV.

The preservation of samples in their near-native state is the first bottleneck researchers encounter in cryo-imaging. Preservation is achieved by means of fast plunge freezing (vitrification) of the sample itself. However, the success rate is highly sample-dependent. For this reason, any multimodal bioimaging workflow would benefit from dedicated cryo-sample qualification protocols. The validation relies on specific information on the molecular as well as the macroscopic properties. X-Ray Diffraction (XRD) analysis reveals the molecular arrangement of water in the sample through the interpretation of its diffraction patterns, while quantitative information can be obtained using interferometry. This technique provides the topography of the sample's surface, therefore allowing a correlation between its thickness and its diffraction pattern.

With this poster, I would like to highlight how well-designed sample preparation protocols and correlative multimodal imaging can adapt to answer biological questions across diverse sample types and functions.

PO-CHE-04

Magnetic Enhancement of High-Entropy Oxide Electrocatalysts for High Areal-Energy Rechargeable Zinc Air Batteries

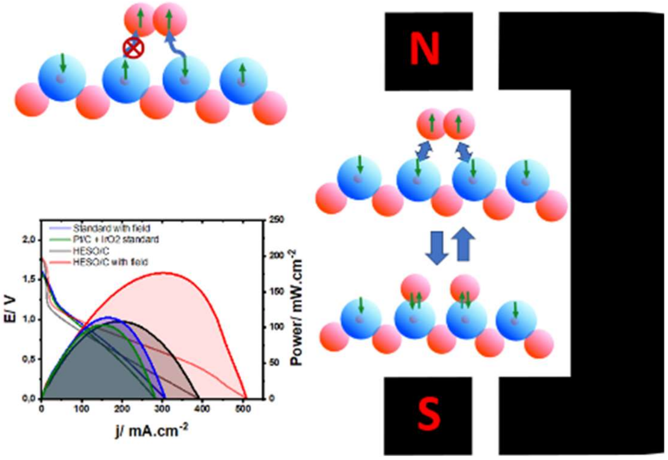
Ernst H. Hechter[1], Aderemi B. Haruna[1], Xiao-Yu Yang[1,2], Maxwell W. Terban[3], Héctor D. Abruña[4,], and Kenneth I. Ozoemena[1,*] Dean H. Barrett[1]

[1]*Molecular science Institute, School of Chemistry, University of the Witwatersrand, Johannesburg 2050, South Africa*

[2]*State Key Laboratory of Advanced Technology for Materials Synthesis and Processing, School of Materials Science and Engineering, Wuhan University of Technology, Wuhan 430070, China*

[3]*Max Planck Institute for Solid State Research, Heisenbergstr. 1, 70569 Stuttgart, Germany.*

[4]*Department of Chemistry and Chemical Biology, Cornell University, Ithaca, New York 14853, United States of America*



A high entropy spinel oxide $(CuCoFeMnNi)_3O_4$ was synthesized for use as a bifunctional oxygen evolution (OER) and reduction (ORR) reaction electrocatalyst for rechargeable zinc-air battery (RZAB) cathodes. An external magnetic field was used to induce enhancements in both the OER and ORR, as well as a dramatic improvement in the power profile of the zinc-air battery. This result is the most significant magnetic enhancement of a RZAB yet reported.

Cellulose in Solution in Ionic Liquids: Conformation and Inter-chains Correlations

Q. Zhang[a], J. Perez[b], Y. Nishiyama[c], D. Constantin[d],
P. Judeinstein[a], F. Boué[a]

a Laboratoire Léon Brillouin, UMR12 CEA-CNRS, U. Paris-Saclay, CEA, F-91191 Gif sur Yvette,
b SWING, Synchrotron SOLEIL, Saint-Aubin – BP 48, 91192 Gif sur Yvette, France.
c CERMAV-CNRS, CS40700, 38041 Grenoble Cedex 9, France.
d Institut Charles Sadron, CNRS and Université de Strasbourg, 67034 Strasbourg, France.

ABSTRACT

Ionic liquids (ILs) emerged as a non-polluting solvent that can dissolve cellulose effectively. The solvation mechanism is slightly controversial. In studies of such solutions using SAXS, in spite of low contrast and thin chain conformation giving very low intensity,

- we extracted a form factor of core-shell semiflexible chain.
- increasing concentration on a wide range (0.0005 - 0.3 g/g), we unveiled interchain effects:
    - in well dissolved cases, a structure factor can be proposed for independent softly aligned cellulose chains
    - at higher concentration, or in presence of a small percentage of water, a stronger scattering is observed suggesting biphasic structures, which can be useful to understand the first stages of the “regeneration”
- analysis of bacterial cellulose scattering evidences identical chain whereas aggregates additional low q scattering can be subtracted. This widens the way to neutron scattering experiments using deuteriated bacterial chains.

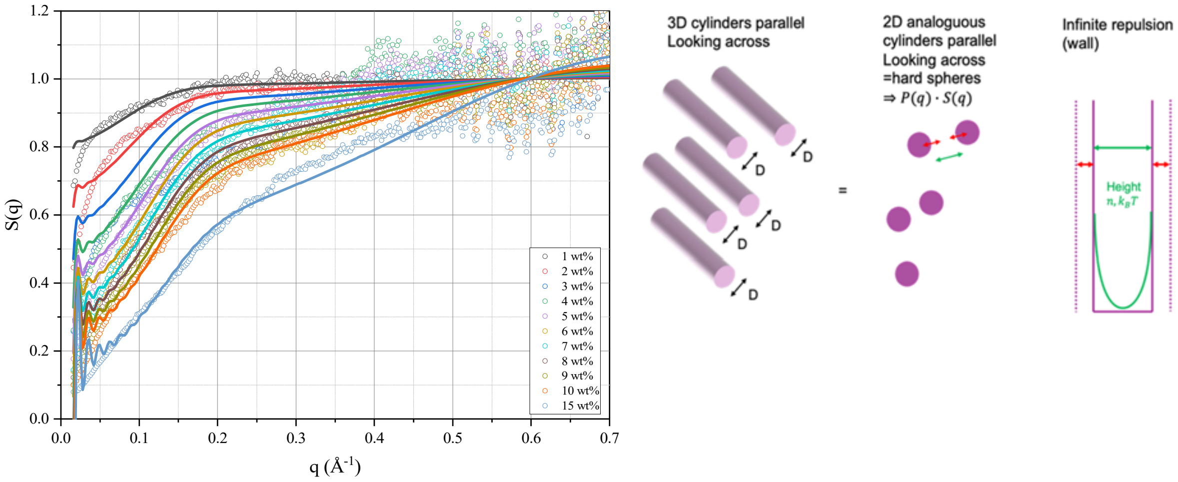


Figure: Aligned “Hard Rods” (right) effective structure factor(left) of cellulose chains in an Ionic Liquid, ButylMethylImidazolium Acetate, for concentration between 1% and 15%.

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Structural Analysis of Colloidal Titania Ribbons and their Organization upon Drying

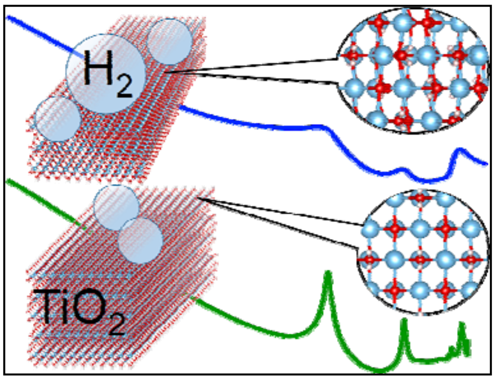
S. Boukhris,[a] A.C. Iacoban,[b] H. Badr,[c] A.C. Kuncser,[b] M. Ibrahim,[c]
M.W. Barsoum,[c] M. Florea,[b] and D. Constantin [a]

[a] *Institut Charles Sadron, CNRS and Université de Strasbourg, 67034 Strasbourg, France*
E-mail: sebastien.boukhris@unistra.fr
[b] *National Institute of Materials Physics, 405A Atomistilor Street, 077125 Magurele, Romania*
[c] *Department of Materials Science and Engineering, Drexel University, Philadelphia, Pennsylvania 19104, United States*

ABSTRACT

Titanium dioxide (TiO_2) is one of the most widely studied and used semiconductors for photocatalysis due to its stability, non-toxicity, abundance, wide availability, and low cost. Alongside its uses in environmental cleanup processes, TiO_2 shows a high potential in water splitting, a process that generates hydrogen fuel by splitting water (H_2O) into hydrogens (H_2) and oxygen (O_2) using solar energy. However, the efficiency of this process is limited by the recombination of electron-hole (e-h) pairs generated. One way to solve this issue is by using nanostructures that increase the surface area and improve charge separation, thus reducing recombination. It has been postulated that lower dimensionality in titania, and especially 1D morphologies, helps in preventing e-h recombination [1]. In this context, we have previously reported a scalable, one-pot, near-ambient process to convert low-cost, earth-abundant, non-soluble titanium precursors, such as TiO2, TiC, TiN, into 1D lepidocrocite titanate nanofilaments (NFs), henceforth referred to as 1DL [2, 3, 4].

In this work, we use synchrotron-based small and wide-angle X-ray scattering to elucidate the structure of low-dimensional titania (Figure). We show that in the colloidal state, they consist of NFs, loosely associated into ribbons, one lepidocrocite sheet thick (about 4 Å), 30 to 40 Å wide (10 to 15 NFs) and more than 300 Å long. Upon drying, they reach a final state of extended sheets, stacked three to about twenty high, whose crystallinity increases with the stack height, in parallel with a decrease in photocatalytic activity. The photocatalytic activity is higher for thinner and more disordered stacks. These findings suggest a kinetic pathway for the self-assembly of initially one-dimensional NFs into two and ultimately three-dimensional structures, providing context for a recent body of work on these low-dimensional materials.



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Deciphering the Kinetics of Photopolymerization-induced Nanostructure and Interface Formation for Submicron Additive Manufacturing

S. Chen[*#§], R.A.T.M. van Benthem[$], J.F.G.A. Janssen[%],
M.K.G. Johansson[^], P. Müller-Buschbaum[*], S.V. Roth[#^]

**: Technical University of Munich, TUM School of Natural Sciences, Department of Physics, Chair for Functional Materials, James-Franck-Str. 1, 85748 Garching, Germany*
#: DESY, Notkestraße 85, 22607 Hamburg, German
§: FRM II, Technische Universität München, Lichtenbergstraße 1, 85748 Garching, Germany
$: Eindhoven University of Technology, Groene Loper 5, 5600, MB, Eindhoven, the Netherlands
%: Covestro (Netherlands) B.V., Urmonderbaan 22, 6167, RD, Geleen, the Netherlands
^: Department of Fibre and Polymer Technology, KTH Royal Institute of Technology, Teknikringen 56, SE-100 44 Stockholm, Sweden

ABSTRACT

Photopolymerization offers spatial resolution, low energy consumption, and high curing speeds, making it a widely used technology in additive manufacturing[1]. Although the photopolymerization is widely used, the fundamental understanding of the physical structure induced by photopolymerization and interface formation during the transition from solid-liquid to solid-solid interfaces in multilayer films are more of missing pieces[2]. By modulating precursor resin components and combining grazing incidence small angle X-ray scattering (GISAXS)[3], the UV-curing induced nanostructure and the kinetic of interface formation of resin multilayer are probed. We quantify photopolymerization-induced nanostructured domains by GISAXS experiments. And we find that solvent preassembly of polymer molecular chains directly contributes to the heterogeneity of nanostructure growth induced by photopolymerization. In addition, we performed GISAXS experiments on the solid-liquid interface during the initial deposition stage of multilayer film preparation, which shows that swelling of the crosslinked layers leads to the reorganization of larger nanostructured domains (radius >100 nm) induced by photopolymerization thereby inducing the interlayer interfaces.

In order to gain a deeper understanding of the formation mechanism of typical nanostructured domains induced by photopolymerization and the heterogeneity of their conversion rates[4], our next step will be to conduct additional studies on the characterization of nanostructured domains in the established GISAXS system in conjunction with Nano-FTIR experiments.

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Crystal Structure and Phase Transitions in Azetidinium Metal Halides for Photovoltaics via in situ Low-temperature Synchrotron XRD

Y.U. Jin[1], A.N. Salak[2], B. Marler[3], E. Elkaïm[4], and D.C. Lupascu[1]

[1]Institute for Materials Science and Center for Nanointegration Duisburg-Essen (CENIDE),
University of Duisburg-Essen, 45141 Essen, Germany
[2]Department of Materials and Ceramics Engineering, CICECO-Aveiro Institute of Materials,
University of Aveiro, 3810-193 Aveiro, Portugal
[3]Institute of Geology, Mineralogy and Geophysics, Ruhr-University Bochum, 44780 Bochum, Germany
[4]Synchrotron Soleil, L'Orme des Merisiers, Saint-Aubin, BP 48, Gif-sur-Yvette Cedex, 91192 France

ABSTRACT

Organic-inorganic hybrid halide perovskites have been extensively spotlighted due to their exceptional photovoltaic property and have opened a new generation in solar cell materials.[1,2] As pioneering materials, methylammonium lead iodide ($MAPbI_3$) and formamidinium lead iodide ($FAPbI_3$) have been mainly focused in the photovoltaic field owing to their proper bandgap, high light absorption coefficient, and outstanding charge carrier mobility.[3] However, these materials face stability and environmental issues, leading to the in-depth study of the chemical composition, such as a replacement of organic A-site cations and metal lead-free B-site divalent cations species. It has been revealed that the screening effect in MA cation for charge carrier migration is extraordinarily effective in the $MAPbI_3$ despite the existence of many defect states due to a remarkably large dielectric constant.[4]

Accordingly, our group focused on the synthesis of novel lead-free hybrid halides with a new A-site cation replacement, which is the azetidinium [$(CH_2)_3NH_2^+$, Az].[5,6] Synchrotron X-ray diffraction patterns of the 7 newly synthesized materials have been obtained via in situ temperature measurement at cryostat in CRISTAL beamline over last two years: $(Az)_2AgBiBr_6$, $(Az)_3Bi_2I_9$, $(Az)_3Bi_2Br_9$, $(Az)_3Bi_2Cl_9$, $(Az)_3Sb_2I_9$, $(Az)_3Sb_2Br_9$ and $(Az)_3Sb_2Cl_9$. These azetidinium lead-free metal halides do not adopt an ideal 3D perovskite structure but a low-dimensional system such as 0D, 1D, and 2D. Recently, many studies have proven that the dimensionality of hybrid halides at a molecular level provides a broad understanding of optoelectronic and photovoltaic properties with various angles.[7] The low-dimensional hybrid halides present a unique electronic structure and optical properties in comparison with 3D hybrid halide perovskites. Here we report on the phase transitions and the azetidinium molecular behavior over a wide low-temperature range obtained via the Rietveld refinement of the Synchrotron XRD data collected at the CRISTAL beamline.

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Evaluating the Electronic Structure and Stability of Alkaline O_2 Evolution Electrocatalysts at BESSY II, in SOLIAS Cluster Tool

C. Maheu[a,b],*, C. Tian[b], X. Huang[c], K. H. Zhang[c], J. P. Hofmann[b]

[a] *Institut des Matériaux de Nantes Jean Rouxel, Centre National de la Recherche Scientifique (CNRS), Nantes Université, CEDEX 03, 44000 Nantes, France*

[b] *Department of Materials and Earth Sciences, Technische Universitat Darmstadt, 64287 Darmstadt, Germany*

[c] *State Key Laboratory of Physical Chemistry of Solid Surfaces, College of Chemistry and Chemical Engineering, Xiamen University, Xiamen 361005, P. R. China*

ABSTRACT

The oxygen evolution reaction (OER) limits the efficiency of the entire water electrolysis process. Therefore, the development of highly active and stable OER electrocatalysts is critical. The scarcity and the high cost of IrO_2- and RuO_2-based catalysts make alternatives attractive, such as $La_{1-x}Sr_xFeO_3$ (LSFO) perovskite oxide [1], or $Ni_xCo_3–xO_4$ (NCO) spinel oxides [2]. Sr-doping of LSFO ($La_{1-x}Sr_xFeO_3$) results in the oxidation of Fe^{3+} to Fe^{4+} and enhances the OER activity; filled electronic states near the Fermi level (EF), the so-called hole state, improve the formation of key OER reaction intermediates [1]. Similarly, the OER activity of NCO materials has been correlated with the Ni^{3+} content and the resulting hole states density [2]. However, electrocatalysts tipicaly undergo morphological and electronic changes during OER. It is therefore crucial to provide, ideally *in-situ*, information about true active sites and their stability.

Quasi in-situ measurements were performed in the SOLIAS endstation of BESSY II, where it is possible to navigate under a controlled atmosphere (vacuum or argon purged) between the electrochemistry and the spectroscopic analytic chambers [3]. Inside the first one, cyclic voltammetry (CV) measurements were performed in 1 M KOH. Inside the second one, near-edge X-ray absorption fine structure spectroscopy (NEXAFS) and synchrotron X-ray photoemission spectroscopy (SXPS) were performed, after each electrochemical treatments (3 CV, 10 CV and 30 CV in total). The excitation energies were adjusted for each core level to measure the photoelectrons at identical kinetic energies, and therefore investigate each element at a comparable depth of analysis.

In short, the enhanced activity of the NCO series is due to surface reconstruction that builds up oxygen deficiency and overcomes the detrimental EF shift as well as the stable hole state content [2]. Electronic modifications have a greater impact along the LSFO series for which $LaFeO_3$ has a decreased activity after CV cycles because it becomes more p-doped and the hole state density is increased. On the contrary, $La_{0.33}Sr_{0.67}FeO_3$ becomes less p-doped, and the hole state content decreases, resulting in a lower OER activity after the 30 CV cycles.

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Chemical and Structural Evolution of the $Rb_{2\text{-}2x}K_{2x}Ti_2O_5$ Layered Titanates when Exposed to Ambient Air

N. Meziani[1], D. Berardan[1], M. Parent[4], G. Rousse[2], B. Leridon[2] and P. Giura[3]

[1] *ICMMO, CNRS, Université Paris-Saclay, Orsay, France*
[2] *LPEM, ESPCI, PSL Sorbonne Université, Paris, France*
[3] *IMPMC, CNRS, Sorbonne Université, Paris, France*
[4]*Laboratoire de Chimie du Solide et Énergie, CNRS, Collège de France, Paris, France*

ABSTRACT

The search for new materials containing non-critical mineral resources with outstanding physical properties for energy storage is a major objective today. $M_2Ti_2O_5$ compounds (Figure 1), where M is an alkaline element, are solid electrolytes that exhibit interesting electrical and electrochemical properties when hydrated. Despite the recent interest in these materials, the nature of the ionic species responsible for their transport properties remains a widely debated topic. It is therefore essential to study the nature and formation processes of mobile ionic species. [1-2]

In this study, we combine ATG, XRD and IR spectroscopy to investigate the chemical and structural evolution of alkali titanates $Rb_{2-2x}K_{2x}Ti_2O_5$ ($0 \leq x \leq 1$) when exposed to the ambient atmosphere. Our results demonstrate that a complete solid solution exists between the potassium and rubidium end-members, with mixed compounds exhibiting intermediate behavior. On exposure to ambient air, all compounds degrade progressively, forming first hydrates and then bicarbonates. Rubidium titanate degrades significantly faster than potassium titanate. Conversely, when hydrated in the absence of CO_2, the crystal structure of potassium titanate ($K_2Ti_2O_5$) alters more rapidly than rubidium titanate ($Rb_2Ti_2O_5$)[3].

These observations enable us to trace an initial description of the physicochemical evolution of these materials under controlled hydration conditions, both in the presence and absence of CO2. The results obtained provide the groundwork for ageing control and tuning of the hydration process, helping to optimize the energy transport and storage properties of these new alkali titanates.

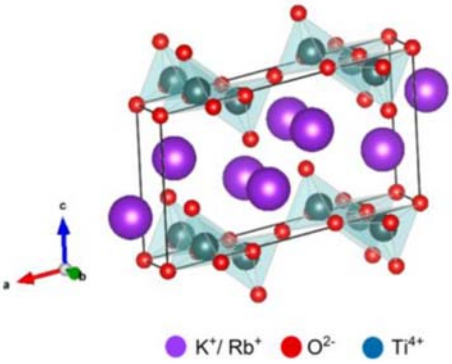

Figure 1 : Cristal structure of $M_2Ti_2O_5$

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Catalytic PVC-model Pyrolysis over H-ZSM5 Probed by Photoelectron Photoionization Coincidence Spectroscopy

J. Cabana[ab*], Z. Zhang[a], Z. Pan[ab], P. Kumar[a], A. Bodi[a], G.A. Garcia[c], L. Nahon[c], J.A. van Bokhoven[ab], and P. Hemberger[a]

a Reaction Dynamics Group, Laboratory for Synchrotron Radiation and Femtochemisty,
Paul Scherrer Institut, Villigen, Switzerland
b Eidgenössische Technische Hochschule Zürich, Zurich, Switzerland
c Synchrotron SOLEIL, L'Orme des Merisiers, St. Aubin, BP 48, 91192 Gif-sur-Yvette, France
** jovanni.cabana@psi.ch*

ABSTRACT

The mechanism of bulk plastic waste pyrolysis is not yet fully understood due to several variables such as undefined intermediates, uncharted routes, and unselective products, thus limiting optimization of this thermochemical recycling process. While the typical pyrolysis coupled to GC/MS set-up[1] detects only stable products, it is difficult to catch elusive intermediates that are the key to unlock reaction pathways necessary to optimize a higher product selectivity, relevant for a feasible commercialization.

In this study, polyvinyl chloride, modeled by 1,3-dichlorobutane, is pyrolyzed over H-ZSM5 coupled with Photoelectron Photoion Coincidence Spectroscopy (PEPICO), a well-suited method for isomer-resolved analysis of reactive intermediates.[2] The zeolite catalyst achieved high conversion (H-ZSM5: 100%) even at low temperatures. Dehydrochlorination proceeds rapidly over H-ZSM5 with transient chlorine species expressed through HCl desorption. After dehydrochlorination, butadiene is formed, oligomerized, and subsequently cracked to lower molecular weight products. In turn, a broad range of products can be observed from H-ZSM5 at 350°C but shifts selectivity at 500°C to light olefins and monocyclic aromatic hydrocarbons.

Mass-selected threshold photoelectron spectra revealed a pool of isomeric butadiene-dimers (C_8H_{12}) primarily of five and six-membered ring monocyclic aromatics, which eventually undergo ring contraction, expansion, demethylation, hydrogenation, and cracking reactions. The model compound follows the general PVC mechanism: dechlorination, polyene formation, aromatization, and further cracking. This project sheds light on fundamental aromatization pathways of PVC pyrolysis products specifically benzene, toluene, and xylenes (BTX); its detection and fingerprinting, its oligomer decomposition routes, and its intermediate species.

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Deperturbation Analysis of the $A^1\Pi(v = 4)$ Level In $^{13}C^{18}O$ Based on the VUV and Vis FT Spectroscopies

S. Ryzner[1], A. Stasik[1], W. Szajna[1], M.I. Malicka[2],
R.W. Field[3], N. de Oliveira[4], A. Pashov[5], P. Jasik[6,7],
J. E. Sienkiewicz[6,8], W. Ubachs[9] and R. Hakalla[1]

[1]Institute of Physics, University of Rzeszow (Poland)
[2]The Faculty of Mathematics and Applied Physics, Rzeszow University of Technology (Poland)
[3]Department of Chemistry, Massachusetts Institute of Technology, Cambridge (USA)
[4]Synchrotron SOLEIL (France)
[5]Faculty of Physics, Sofia University, (Bulgaria)
[6]Faculty of Applied Physics and Mathematics, Gdańsk University of Technology (Poland)
[7]BioTechMed Center, Gdańsk University of Technology (Poland)
[8]Advanced Materials Center, Gdańsk University of Technology (Poland)
[9]Department of Physics and Astronomy, Vrije Universiteit (The Netherlands)

ABSTRACT

The present study reports the first deperturbation analysis of the $A^1\Pi(v = 4)$ level in the $^{13}C^{18}O$ molecule. This analysis was based on spectra acquired through two complementary Fourier-transform spectroscopic methods: *(i)* emission spectroscopy in the visible (Vis) region, utilizing a Bruker IFS-125HR spectrometer (University of Rzeszow, Poland), and *(ii)* vacuum ultraviolet (VUV) absorption spectroscopy, conducted with a wave-front-division spectrometer at the DESIRS beamline of the SOLEIL synchrotron (St. Aubin, France).

The deperturbation analysis of the $A^1\Pi(v = 4)$ level was carried out using the PGOPHER program Ref. [1], employing an effective Hamiltonian and the term-value-fitting approach. This methodology enabled the precise determination of molecular parameters for $^{13}C^{18}O$, including molecular constants, interaction parameters and the ro-vibronic terms of the $A^1\Pi(v = 4)$ level and its perturbers.

This research represents a continuation of prior investigations into the $A^1\Pi$ state and its numerous perturbing states in carbon monoxide isotopologues, previously undertaken by our research group [2–6].

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High-resolution Structure and Energetics of the DABCO Molecule Attached to Clusters for Modelling Energy Transport in Matter

A. Scognamiglio[1], O. Shviro[1], B. Zarié[1], L. Barreau[1], D. Cubaynes[1],
B. Gans[1], E. Gloaguen[1], C. Schouder[1], G. A. Garcia[2], L. Nahon[2],
A. de la Lande[3] and L. Poisson[1]

[1] *Institut des Sciences Moléculaires d'Orsay, Université Paris-Saclay, CNRS, 91405 Orsay, France*
[2] *Synchrotron SOLEIL, L'Orme des Merisiers, St. Aubin BP 48, 91192 Gif sur Yvette, France*
[3] *Institut de Chimie Physique, Université de Paris-Saclay, CNRS, 91400 Orsay, France*

ABSTRACT

The diffusion of electrons in matter generates secondary species, such as secondary electrons and radicals, that can interact with nearby species. In cells, these interactions may cause DNA damage and increase cancer risk [1]. Recently, studies have shown that data on both elastic and inelastic scattering in small water clusters can support the development of theoretical models to predict energy transport at the macroscopic scale [2]. Clusters, in particular, provide effective models for describing organized condensed matter [3]. Our approach uses a highly symmetrical molecule, DABCO, known for its atomic-like orbitals [4], deposited on argon and water clusters and ionized via a 1-photon transition with synchrotron light (DESIRS beamline). Coincident ion and electron detection allows precise identification of the products formed and their photoelectron distributions. We have gathered detailed information on the electronic and vibrational structure of the isolated molecule and small clusters – examined here with unprecedented precision. We have as well retrieved information on the photoelectron angular distribution in both cases, from which we aim to gain insights on electron scattering processes.

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VUV-FT Spectroscopy of the (*v*' - 0) Progression of the Cameron System (a$^3\Pi$ – X$^1\Sigma^+$) in the CO Molecule

A. Stasik[1], M. Malicka[2], S. Ryzner[1], A. Heays[3], R. Field[4], N. de Oliveira[5], W. Szajna[1], W. Ubachs[6], R. Hakalla[1]

[1]Materials Spectroscopy Laboratory, Institute of Physics, University of Rzeszow, Poland.
[2]Faculty of Mathematics and Applied Physics, Rzeszow University of Technology, Poland.
[3]J. Heyrovsky´ Institute of Physical Chemistry , Prague, Czech Republic.
[4]Department of Chemistry, Massachusetts Institute of Technology, Cambridge, USA.
[5]Synchrotron SOLEIL, Orme de Merisiers, St. Aubin, Gifsur Yvette Cede, France.
[6]Department of Physics and Astronomy, Vrije Universiteit, Amsterdam, The Netherlands.

ABSTRACT

Carbon monoxide (CO) has garnered significant attention from researchers due to its immense abundance in space, second only to molecular hydrogen [1]. This makes CO a key player in the modelling of the evolution of nebulae, galaxies, stars, planets and exoplanets. Additionally, the Cameron system (a$^3\Pi$ – X$^1\Sigma^+$) of CO is crucial for studies on the dayglow of Mars' atmosphere [2 - 4].

The primary objective of this study was to obtain the first four and regular bands of the (*v*' - 0) progression of the Cameron system (a$^3\Pi$ – X$^1\Sigma^+$) in $^{12}C^{16}O$. This was achieved through Fourier-transform absorption spectroscopy in the vacuum ultraviolet (VUV-FT) using a Fresnel-mirror spectrometer on the DESIRS beamline of the SOLEIL synchrotron [5-6] (see Fig. 1).

Accurate analysis of the first four vibrational levels of the a$^3\Pi$ electronic state was performed using the PGOPHER software [7], employing an effective Hamiltonian for precise spectroscopic modeling.

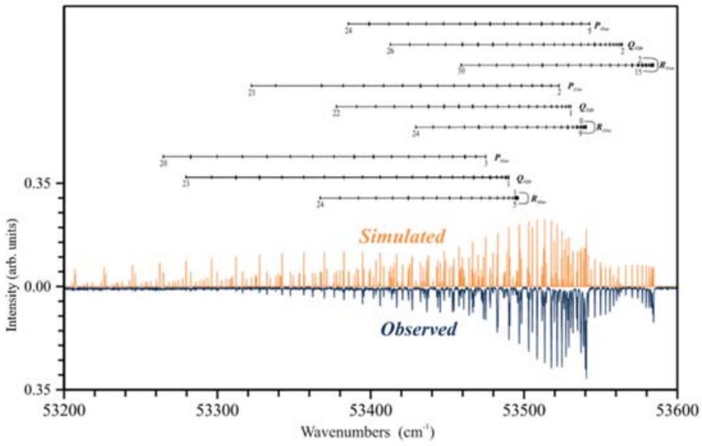

Fig. 1. Experimental spectrum of the $^{12}C^{16}O$ a$^3\Pi$ - X$^1\Sigma^+$(3,0) recorded by VUV spectroscopy technique (lower, navy blue trace) as well as the simulated one plotted on the basis of the final deperturbation analysis using the PGOPHER software (upper, orange trace).

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Evaluating Soil Carbon Responses to Fertilizer Use in Moroccan Olive Cultivation

K. Boufous[1], Y. Yusuf[1], K. Benghzial[1], L. Vergutz[2], H. Mohamed[3,4],
H. W. Pereira de Carvalho[1,5]

[1] The Global Critical Zone Science Chair, Mohammed VI Polytechnic University, Lot 660,
Hay Moulay Rachid, Ben Guerir, 43150, Morocco
[2] OCP Group, Morocco
[3] African Genome Center—Agrobiosciences, Mohammed VI Polytechnic University, Lot 660,
Hay Moulay Rachid, Ben Guerir 43150, Morocco
[4] Institut de Recherche en Biologie Végétale, Département de sciences biologiques,
Université de Montréal, 4101 Sherbrooke Est, Montréal, QC H1X 2B2, Canada
[5] Centre for Nuclear Energy in Agriculture, Sao Paulo University, Piracicaba, Brazil

ABSTRACT

Soil carbon is a major constituent of soil health, fertility, and a factor in sustainable agricultural practices and climate change mitigation. Understanding the role of fertilizer application in affecting soil carbon dynamics is very critical in optimizing nutrient management for any agricultural system. The study examines the impacts of fertilizer application on soil carbon levels in Moroccan olive farms with a view to drawing insights into sustainable practices that enhance soil health and carbon sequestration. Four parcels were considered: old fertilized and non-fertilized, young fertilized and young non-fertilized olive trees. Rhizospheric soil, bulk soil (0–20 cm) and control compartments were sampled. Carbon functional groups and their interaction with quartz were investigated by means of synchrotron-based FTIR spectroscopy while calcium speciation was investigated using XANES spectroscopy. Complementary laboratory analyses were carried out to assess total and organic carbon content, nutrients availability, and microbial activity. Preliminary results indicate that fertilized soils had 40% more calcium, with up to 290 mmol/kg in the bulk soil of old fertilized trees. Phosphorus concentrations were about 35% greater, reaching 140 mg/kg in fertilized rhizospheric soils. Fertilized soils had 15–20% more organic carbon, up to 4.5% in rhizospheric soils compared to 3.0% in non-fertilized soils. The higher SOC values found in this study, up to 4.5%, are far higher than the national average of 1–2% for Moroccan soils (Zayani et al., 2023). This could be explained by the long-term influence of olive tree cultivation, particularly for old orchards (up to 100 years), and the application of fertilizers that increase soil organic carbon compared to the national average. FTIR analysis showed that fertilized soils have better linkages between quartz and functional groups, such as carboxyl (COO-), aromatic C (C=C), and aliphatic carbon (C-H), which might imply better stabilization of organic matter. XANES spectroscopy revealed clear differences in calcium forms in unfertilized soils. Calcite was the most abundant species in both rhizospheric (77%) and control soils, with smaller contributions from marl (17%) and calcium-citrate compounds (6%). The study provides critical information on how soil carbon will respond to fertilization and will help design management practices for sustainable olive production systems.

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Co(II) oxidation to Co(III) at the $Fe_{3-\delta}O_4$ surface in oxidizing environments

L. Fablet[1,2], F. Choueikani[2], R. Marsac[3], A. Beauvois[2], T. Stephant[4]
V. Briois[2] et M. Pédrot[1]

[1]*Univ Rennes, CRNS, Géosciences Rennes – UMR 6118, F-35000 Rennes, France*
[2]*Synchrotron SOLEIL, l'Orme des Merisiers, Saint-Aubin – BP48, 91192 Gif-sur-Yvette, France*
[3]*Université Paris Cité, Institut de physique du globe de Paris, CNRS, F-75005 Paris, France*
[4]*Univ Rennes, CNRS, ISCR – UMR 6226, F-35000, Rennes, France*

ABSTRACT

Understanding and predicting the interaction mechanisms between Co and spinel iron oxide nanoparticles ($Fe_{3-\delta}O_4$) is of major interest in several contexts. Indeed, $Fe_{3-\delta}O_4$ nanoparticles can be used for soil and water decontamination due to their high surface reactivity. In addition, their magnetic properties enable them to be used for industrial applications, with the synthesis of nanoparticles decorated with metals. Interactions between Co and $Fe_{3-\delta}O_4$ nanoparticles have been well documented under anaerobic conditions, with descriptions of Co^{2+}, polymerization and surface precipitation.[1,2] However, $Fe_{3-\delta}O_4$ nanoparticles are mostly used in the presence of O_2, and the interaction mechanisms in these conditions have attracted less attention. Therefore, the influence of O_2 on Co adsorption and redox speciation at $Fe_{3-\delta}O_4$ nanoparticles surface was studied.

Magnetite (Fe_3O_4) nanoparticles were synthesized by co-precipitation of iron salts under anaerobic conditions, then oxidized with H_2O_2 to obtain $Fe_{3-\delta}O_4$ with R = [Fe(II)]/[Fe(III)] = 0.1. Adsorption experiments were carried out under aerobic conditions with Co concentrations ranging from 0.1 to 12 mM, at pH 8, at room temperature. A part of these samples was analyzed at SOLEIL synchrotron by X-ray spectroscopy (XAS) and X-ray magnetic circular dichroism (XMCD) at $L_{2,3}$-edges on DEIMOS beamline. To follow the adsorption kinetics of Co on $Fe_{3-\delta}O_4$ nanoparticles, Quick-XAS experiments were performed at *K*-edge on ROCK beamline. The recorded spectra were processed by PCA and MCR-ALS analysis, and EXAFS fitting.

Adsorption experiments show two different behaviors of Co onto $Fe_{3-\delta}O_4$ nanoparticles, according to Co concentration, with (i) the polymers at low Co concentration and (ii) the Co precipitation at high Co concentration. These changes in surface speciation are also distinguished by the change in oxidation state of Co^{2+} to Co^{3+} as determined by XAS and XMCD analyses at high concentration at $L_{2,3}$-edges. For high Co concentration, the adsorption kinetic at *K*-edge have enabled to differentiate three species over time with (i) the Co^{2+} in solution, (ii) the formation of a mixture of several Co(II) species and (iii) the partial oxidation of Co(II) to Co(III), leading to the formation of a Co_3O_4 precipitate.

This study highlights the formation of mixed species of Co(III) and Co(II) onto the magnetite surface, depending on the initial Co concentration, which might play a role in Co behavior and fate in oxic environments with the presence of $Fe_{3-\delta}O_4$ nanoparticles, but also to control the synthesis of $Fe_{3-\delta}O_4$ nanoparticles decorated with Co for magnetic or catalytic applications.

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Iron Speciation of Metamorphic Silicates at Microscale

Françoise, M.[1], Dubacq, B.[1], Bourdelle, F. [1], Verlaguet A.[1]

[1] Sorbonne Université, CY Cergy Paris Université, CNRS INSU, Institut des Sciences de la Terre de Paris, ISTeP, F-75005 Paris, France

ABSTRACT

The crystal chemistry of metamorphic minerals allows assessing the pressure-temperature history of crustal rocks, via phase diagrams constructed with thermodynamic models. Conversely, an error or approximation in the determination of the phase chemistry can result in discrepancies in the outcomes of thermodynamic models, and to major misinterpretations of the conditions undergone by the rock.

Routinely, the chemistry of metamorphic minerals is measured with electron microprobe (EPMA) and hence considers all iron as divalent. However, silicates and in particular phyllosilicates (chlorite, mica, serpentine) may include ferric and ferrous iron, and the speciation can vary on short distances, i.e. on few micrometers. Neglecting iron speciation in these Fe-rich minerals strongly impacts their estimated temperature of crystallisation**[1,2,3]**.

The *Schistes Lustrés* complex (Western Alps) is a well-studied area**[4]** where the thermobarometry via thermodynamic models is particularly challenging. This study reports on X-ray Absorption Spectroscopy (XAS) measurements carried out at the GALAXIES beamline of SOLEIL synchrotron to determine the iron speciation of a selection of metamorphic minerals from this complex. The beamline micro-beam (<500 µm²) and circular polarization enabled to collect XANES at the K edge of Fe in anisotropic micrometric metamorphic minerals, following the procedure of Wilke et al. (2001)**[5]**. Iron in chlorite is mainly divalent (0-40% Fe^{3+}) but reaches non-negligible Fe^{3+} levels, while it is mainly – or even completely – trivalent in mica (40-100% Fe^{3+}). These high Fe^{3+} content greatly differ from the generally-assumed crystal chemistry, in particular for estimated octahedral vacancies in chlorite, pointing out to the importance of the di-trioctahedral substitution ($2Al^{3+}$ + vacancy $\leftrightarrow 3Mg^{2+}$), in addition to the Tschermak substitution (${}^{IV}Si^{4+} + {}^{VI}Mg^{2+} \leftrightarrow {}^{IV}Al^{3+} + {}^{VI}Al^{3+}$) and homovalent substitutions ($Al^{3+} \leftrightarrow Fe^{3+}$ and $Mg^{2+} \leftrightarrow Fe^{2+}$). These measurements will now help refining thermodynamic modelling of the di-trioctahedral substitution.

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Organic Matter Exposed to an Emerging Contaminant: Platinum (Pt) Speciation Changes

J. Le Doare[1], C. Catrouillet[1], M. Benedetti[1], G. Landrot[2], A. Gelabert[3]

1 Institut de physique du globe de Paris, 1 rue Jussieu, 75238 Paris cedex 05, France
2 Synchrotron SOLEIL, l'Orme des Merisiers, 91190 Saint-Aubin, France
3 Centre de Recherche et d'Enseignement en Géosciences de l'Environnement,
Technopôle de la Méditerranée, 13545 Aix-en-Provence, France

ABSTRACT

Due to the use of Pt in anthropogenic activities, its biogeochemical cycle has become unbalanced during the last century.[1,2] Increasing environmental dispersion leads to an inevitable change in Pt speciation in the environment, which is known to occur mainly as Pt(0, II, IV).[3,4] Recognized as a soft acid, the empirical Pearson's theory explains the strong affinity of Pt for thiol (-SH) groups of organic matter. These sites are known to be redox sensitive and therefore affect the bioavailability of platinum in the environment. However, there are no experimental results to confirm these hypothèses

This study provides insight into (i) understanding the evolution of the redox state of Pt and (ii) identifying the atomic environment of Pt when it interacts with organic matter. The humic acid Leonardite (LHA, 1g L^{-1}) has been exposed to Pt(+IV) (667 µg of Pt per g LHA) for 59 days at pH 7 and with an ionic strength adjusted to 0.01mol L^{-1} using $NaNO_3$. Two sets of experiments were carried out: one under aerobic conditions and the other under anaerobic conditions, both in absence of light. During these kinetic experiments, six samples (collected after 1, 3, 8, 10, 13 and 59 days) were analyzed at the Pt L3 edge at the SAMBA beamline at SOLEIL to determine their redox state.

The XANES results showed a reduction from Pt(+IV) to Pt(+II) and possibly to Pt(0) through the formation of Pt nanoparticles over time for experiments performed in anaerobic conditions. Conversely, in the presence of O2, no reduction occurred over time. EXAFS data revealed that Pt's first neighbors were O atoms under aerobic and anaerobic conditions.

Additional equilibrium experiments were performed to determine the atomic environment of Pt(II) when complexed with LHA (1 g L^{-1}). Different Pt(II) salts ($PtCl_2$, $PtBr_2$ and PtI_2) were dissolved in the presence of LHA in dialysis bags, which allowed us to obtain different Pt/LHA concentration ratios due to the different solubility product of the salts.

These observations will contribute to develop initial predictions of Pt behavior in the presence of organic matter, using modeling tools.

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Visual Apparatus of 300 Million-year-old Amphibians Revealed by X-ray Fluorescence Mapping

A. Logghe[1], P. Gueriau[2], F. Clarac[1], J-S. Steyer[1], S. Sanchez[3,4]

[1]Centre de recherche en paléontologie – Paris, UMR 7207 – CNRS, MNHN, SU, Muséum national d'Histoire naturelle, 8 rue Buffon, CP38, Paris, France
[2]Université Paris-Saclay, CNRS, ministère de la Culture, UVSQ, MNHN, Institut photonique d'analyse non-destructive européen des matériaux anciens, Saint-Aubin, France
[3]Department of Organismal Biology, Uppsala University, Uppsala, Sweden
[4]European Synchrotron Radiation Facility, Grenoble, France

ABSTRACT

Amphibians are one of the most diverse clades of tetrapods (four-limbed vertebrates) and they are unique in being able to metamorphose. Metamorphosis is defined as a hormonally induced phenotypic remodelling resulting in a condensation of developmental events[1]. In lissamphibians, this shift often results in a transition from aquatic to (semi-)terrestrial lifestyle, including changes not only in the skeletal apparatus but also the skin, vision, and diet. Soft-tissues are therefore crucial for characterising the palaeoecology of amphibians and thus the metamorphic process. However, so far only skeletal developmental trajectories could be investigated with precision from the fossil record of amphibians, resulting in a fragmentary understanding of their life cycle[2, 3].

Here, we present the results of analyses that are focused on soft-tissues of exceptionally preserved amphibians from the late Carboniferous (~300 million-years-old) of the Montceau-les-Mines fossil site from the Massif Central in France. Dozens of amphibians exhibit soft-tissue remains including dermal tissues, guts, gills and 3D- preserved eyes. 7 specimens were scanned in 2023 at the PUMA beamline. We used a state-of-the-art Micro X-ray fluorescence (μXRF) major-to-trace elemental mapping to highlight the anatomy and chemistry of the eyes, in addition to the skeleton, of these amphibians in order to understand their link to both palaeoecology and life-cycle.

We thus report the oldest preliminary evidence of a tetrapod eye structuration in these early amphibians with the presence of a circled-shape pupil similar to that of early diverging amphibians such as gymnophionans, salamanders and tadpoles. This suggests that a circle-shaped pupils, highly correlated to aquatic life[4], was the ancestral state of the amphibian eye. The ontogeny of the pupil shape in extant amphibians (always circle in larvae) could therefore reflect the eye evolution in regard to terrestrialisation, through metamorphosis. Moreover, one of the main changes in the amphibian visual apparatus through metamorphosis is the apparition of the eyelids[5]. The eyelid is indeed absent in both aquatic and larval anurans and only appears after metamorphosis in both salamanders and anurans. Interestingly, in one of our biggest specimens, a thin veil of arsenic was observed partly covering the orbit suggesting the possible presence of skin surrounding the eye, a feature not observed in other specimens. During the XRF mapping, we could also unveil an enrichment of the eye of our fossils in zinc (Zn), congruent with the observations in extant tetrapod for which the lens contains a substantial amount of zinc[4]. The speciation of Zn in the eyes was also probed using XAS at the Zn K-edge in some mapped eyes.

Thus, our results suggest a structuration of the eyes of early amphibians similar to that of extant larval amphibians, and possibly the first evidence of a change in the eye in agreement with a change of lifestyle. Further investigations could enhance our understanding of visual capacities and changes in the eye from aquatic to terrestrial lifestyles thanks to a new scan session at the PUMA beamline in February of this year.

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Chemical Mechanisms in Radioecology: Cesium in Bivalve Molluscs

A. Plasajec [a], M R. Beccia [a], C. Den Auwer [a], J. Aupiais [b],
M. Monfort [b], P.L. Solari [c]

[a] Université Côte d'Azur, Institut de Chimie de Nice, 06108 Nice, France
[b] CEA, DAM, DIF, F-91297 Arpajon, France
[c] Synchrotron Soleil, L'Orme des Merisiers, Saint-Aubin, BP 48, F-91192 Gif-sur-Yvette, France

ABSTRACT

Of all the environmental compartments that serve as outlets for various metal contaminants, the oceans can be considered the ultimate receptacle for rivers and watersheds. Therefore, the oceans act as a long-term reservoir (i.e., source) for pollution such as metals and radionuclides. Indeed, the marine environment has often been monitored as a marker of pollution. Specifically, the presence of trace metal radionuclides, such as cesium, in seawater is primarily anthropogenic in origin, arising from sources such as atmospheric nuclear testing, waste disposal, mining activities, and accidental releases. This research aims to clarify the mechanisms underlying the accumulation of cesium in sentinel marine organisms, with a focus on improving the understanding of metal speciation, localization, and the associated biological impacts. Model organisms used for contamination are mollusks—particularly mussels— because they are commonly employed as bioindicators of radioactive pollution due to their ability to filter large volumes of seawater and their sedentary nature. Through the use of quantification, spectroscopy, and imaging techniques, this research aims to provide critical data on transfer constants, ecotoxicity, and the distribution and speciation of cesium within the marine ecosystem. This approach will also enhance our understanding of how cesium interacts with biomolecules, offering insights into the broader implications of cesium accumulation in marine organisms and ultimately improve assessments of health risks.

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Unveiling and Optimizing Interface Properties of $NiFe_2O_4/BaTiO_3$

H. Lin[1,2], C. Mocuta[2], R. Arras[3], C. Blaess[1,2], B. Sarpi[4], P. Ohresser[2], H. Magnan[1], J-B. Moussy[1], C. L. Rountree[1], A. Barbier[1]

[1] Université Paris-Saclay, CEA, CNRS, SPEC, 91191, Gif-sur-Yvette, France
[2] Synchrotron SOLEIL, L'Orme des Merisiers Saint-Aubin, BP 48, 91192 Gif sur Yvette Cedex, France
[3] CEMES, Université de Toulouse, CNRS, 29 rue Jeanne Marvig, F-31055 Toulouse, France
[4] Diamond Light Source, Chilton, Didcot, Oxfordshire, UK

ABSTRACT

In this study, we investigate the properties of $NiFe_2O_4$ (NFO)/$BaTiO_3$ (BTO) multiferroic heterostructures, examining individual layer thicknesses ranging from 3 to 12 nm. X-ray diffraction performed at the DiffAbs beamline (synchrotron SOLEIL) reveals that as BTO thickness increases, NFO transitions from a non-strained cubic lattice to a compressively strained tetragonal lattice. In contrast, thicker NFO layers introduce enhanced tensile stresses on the BTO layer, counteracting the compressive strain originating from the $SrTiO_3$ (STO) substrate. Piezoresponse force microscopy demonstrates that the polarization switching voltage escalates with increasing BTO layer thickness. Through X-ray magnetic circular dichroism measurements, performed at the DEIMOS beamline (synchrotron SOLEIL), coupled with multiplet theory, we elucidate variations in the magnetic moments and ionic distributions within the NFO layers. Remarkably, a larger BTO thickness is associated with a chemical reduction of Fe ions in the NFO layer, indicative of increased oxygen vacancies, which are induced by the increasing compressive strain as evidenced by first-principles calculations. Thinner NFO layers showed increased tetrahedral (Td) site vacancies and oxygen vacancies concomitant with reduced magnetic moments, which can be optimized by either increasing NFO thickness or through air annealing at 450°C. Combining the ionic distribution variation with in-plane lattice parameter evolution during growth, we postulated that the reduced magnetic moments originate from a 2−3 nm antiferromagnetic rock salt NiO/FeO formed at the beginning of NFO growth, while the air annealing restores the magnetism by oxidizing and arranging the NiO/FeO into spinel NFO under the synergistic effect of oxygen and heat. We could demonstrate that air annealing at moderate temperature appears as a very efficient method to restore the magnetization of ultrathin layers of NFO, allowing us to overcome a major drawback for these compounds, which hampers their utilization in spintronics applications so far.

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Using Alkali Metal Atoms to Tune the Electronic Properties of Narrow-bandgap semiconductor Nanocrystals

D. Mastrippolito[1], M. Cavallo[1], E. Bossavit[1,2], Y. Prado[1],
M.G. Silly[2], D. Pierucci[1], E. Lhuillier[1]

1 Sorbonne Université, CNRS, Institut des NanoSciences de Paris, INSP, 75005 Paris, France.
2 Synchrotron SOLEIL, L'Orme des Merisiers, Départementale 128, 91190 Saint-Aubin, France.

ABSTRACT

Infrared-active colloidal quantum dots (CQDs) have recently emerged as a powerful alternative to traditional epitaxially grown semiconductors, notably for photodetection. An essential device structure for this aim is the photodiode, which can extract photogenerated charges thanks to the unique electronic band structure provided by a multi-material stack. Realizing high-performance photodiodes, however, requires the ability to fine tune the band alignment at each material interface.

For traditional semiconductors, this fine tuning can be achieved through the introduction of extrinsic impurities, leading to precise levels of doping. In the case of CQDs, this strategy is usually not viable due to fabrication and stability concerns. Carrier density control is often achieved through surface ligand exchanges instead. These capping molecules generate surface dipoles and charge transfers toward the CQDs, which shift the position of the bands with respect to the Fermi and vacuum levels. However, the most appropriate ligands for a given band alignment are not necessarily compatible with efficient charge conduction. There is thus a need for new strategies.

As an alternative, we explored a method to induce surface dipoles in nanocrystals through the deposition of alkali metal atoms. We applied this strategy to HgTe nanocrystals, one of the most mature infrared-active CQD materials, investigating two different sizes.

Using X-Ray Photoemission Spectroscopy, we showed that for all studied CQDs, the deposition of potassium on the surface of a nanocrystal film leads to a significant and continuously tunable shift of the material's work function, reaching 1.3 eV in some cases. We then evidenced that the dipole arises from the polarization of the adatoms without involving any charge transfer toward the CQDs. This seemingly very general approach appears very promising as a way to shift the absolute energy of a band gap, which could ease the integration of colloidal materials into high-performance diodes.

X-ray Natural Circular Dichroism, X-ray Magnetic Circular Dichroism and X-ray Natural Linear Dichroism in Chiral Crystals

A. De Frenza[1], E.A. Hillard[2], P. Rosa[2], N. Kordestani[2], J.M. Ablett[3],
A. Rogalev[4] F. Wilhem[4], N. Yutronkie[4], M-A. Arrio[1], A. Juhin[1],
P. Sainctavit[1,3]

1 IMPMC, CNRS, Sorbonne Université, UMR 7590, 75252 Paris, France
2 Univ. Bordeaux, CNRS, Bordeaux INP, ICMCB, UMR 5026, F-33600 Pessac, France
3 Synchrotron SOLEIL, L'Orme des Merisiers, BP 48, 91192 Saint Aubin, France
4European Synchrotron Radiation Facility (ESRF), 38043 Grenoble, France
e-mail: alessandro.de_frenza@sorbonne-universite.fr

ABSTRACT

Chirality, defined as the absence of improper symmetry operations in a crystal, is frequently associated with optical activity. While this phenomenon has been extensively studied in the UV-visible range, its exploration in the X-ray range remains limited. This study seeks to investigate X-ray natural circular dichroism (XNCD), X-ray magnetic circular dichroism (XMCD) and X-ray natural linear dichroism (XNLD) in two chiral systems belonging to the hexagonal crystal class.

We focus on chiral crystals of cobalt and nickel-based compounds, which exhibit significant XNCD signals as shown by experiments [2]. The two compounds, [Co(en)3](NO3)2 and [Ni(en)3](NO3)2, crystallize in space group P6322 at room temperature. As expected [1], these systems also display angular dependence for both the electric dipole and the electric quadrupole contributions to the X-ray absorption (XAS) cross-sections.

This study includes the computation of XAS, XNCD XMCD and XNLD cross-sections and through an analytical investigation of the angular dependence in XAS, we address the question of the connection of the angular dependence with the electronic structure of the crystals [1],[3]. The various factors influencing the intensity of the XAS, XNCD, XMCD and XNLD signals are thoroughly analyzed based both on the experimental and computational results within the framework of Density Functional Theory (DFT), utilizing the FDMNES code [4].

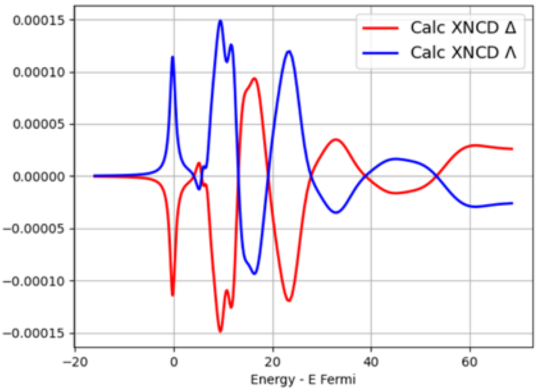

Figure 1: Calculated XNCD and XMCD of $[Co(en)_3](NO_3)_2$

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Tools to Prepare Experiments: Fast Estimates of Electronic and Phononic Dispersions, Powder Diffractograms, and Fluorescence Spectrum

E. Farhi

Synchrotron SOLEIL L'Orme des Merisiers - Départementale 128 - 91190 Saint-Aubin, France

ABSTRACT

We shall present tools proposed by the Data Reduction and Analysis Group at SOLEIL, to estimate some of the material properties, all accessible via web services. To date, we propose two distinct services, all available on our HPC servers [1-2].

The first service is using fast ab-initio/DFT [3] to provide estimates of:

1. the electronic density of states (using DFT)
2. the electronic dispersions in the full Brillouin Zone (coarse sampling)
3. the vibrational density of states
4. the vibrational dispersions in the full Brillouin Zone

The input is a material structure. Results are given as data files as well as in a full report. Typical computation times are of the order of 10-60 minutes for simple structures (e.g. 10 atoms), but can be larger for complex compounds. Once started, computations run in the background, and results can be retrieved later.

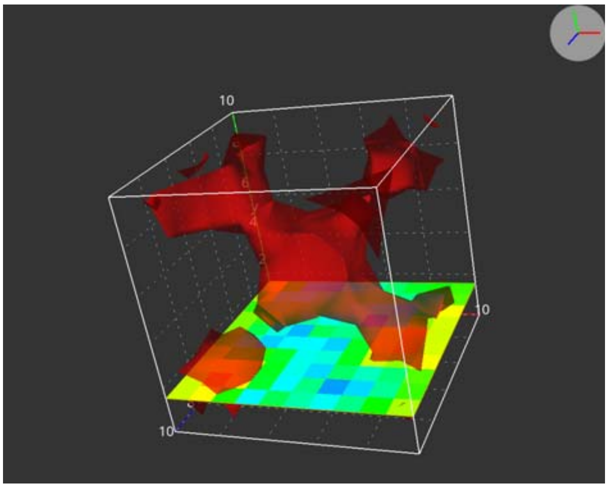

Figure 1: Mo_5PB_2 electronic level isosurface in Brillouin zone.

The second service is using a digital twin [4-5] of a diffraction beam-line to provide estimates of:

1. the powder diffractogram
2. the fluorescence spectrum

The input is a material structure. Results are given as data files as well as in a full report. Typical computation times are of the order of 1-2 minutes, and include some resolution and background effects.

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Biopolymer-templated Deposition of Hierarchical 3D-structured Graphene Oxide/Gold Nanoparticle Hybrids for Surface-enhanced Raman Scattering

Y. Guo[1,2], G. Pan[2], C. Harder[1,2], S. Tu[2], Y. Bulut[1,2], G. Nemeth[3],
F. Borondics[3], B. Sochor[1], S.K. Vayalil[1,4], D. Söderberg[5],
P. Müller-Buschbaum[2] and S.V. Roth[1,5]

1. Deutsches Elektronen-Synchrotron, Notkestr. 85, 22607 Hamburg, Germany
2. Technical University of Munich, TUM School of Natural Sciences, Department of Physics, Chair for Functional Materials, James-Franck-Str. 1, 85748 Garching, Germany
3. Synchrotron SOLEIL, L'Orme des Merisiers, Départementale 128, 91190 Saint-Aubin, France
4. UPES, Dehradun, Uttarakhand, 248007, India
5. KTH Royal Institute of Technology, Department of Fibre and Polymer Technology, Teknikringen 56-58, SE-100 44 Stockholm, Sweden

ABSTRACT

Cellulose has emerged as a promising bio-based template for sensors, smart windows, and bioelectronics. Typically, Surface-Enhanced Raman Scattering (SERS), an advantageous analytical technique, allows for the rapid detection and structural analysis of biological and chemical compounds through their spectral patterns in nanotechnology [1]. Crucial for SERS is fabricating the substrates with strong and reproducible enhancements of the Raman signal over large areas and with a low fabrication cost. Herein, we propose a simple and scalable approach utilizing a layer-by-layer spray coating technique followed by a subsequent annealing method to fabricate cellulose nanofiber (CNF) films loaded with gold nanoparticles (Au NPs) and graphene oxide (GO) hybrids to serve as SERS substrates. On the one hand, the hybrid 3D structures comprising Au NPs/GO significantly boost SERS sensitivity due to the synergistic effects of electromagnetic and chemical enhancements. On the other hand, incorporating three-dimensional (3D) CNF substrates contributes to a more uniform distribution of Au NPs/GO, and thermal annealing further induces remarkable hotspots. Most notably, grazing incidence small-angle X-ray scattering (GISAXS) combined with nano-Fourier-transform infrared spectroscopy (nano-FTIR) was first used to study and confirm a synergistic Raman enhancement mechanism of localized surface plasmon resonance (LSPR) and interface charge transfer (CT) at ultrahigh spatial resolution. Therefore, the proposed methodology provides a framework for facile and scalable production of universally adaptable SERS substrates with exceptional sensitivity.

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Quarter Wave Plate Galvanometer for Fast Polarisation Switching in RIXS

J. Harrison[1], J. Ablett[2], V. Scagnoli[1,3], and S. Gliga[1]

[1.] *Paul Scherrer Institute, Villigen 5232, Switzerland*
[2.] *Synchrotron SOLEIL, L'Orme des Merisiers Départementale 128,*
91190 Saint-Aubin, France
[3.] *Department of Materials, Laboratory for Mesoscopic Systems,*
ETH Zurich, 8093 Zurich, Switzerland

ABSTRACT

Diamond quarter wave plates (QWP) can be used to rotate the polarisation of hard x-rays.[1] At synchrotron radiation facilities, they are often used to convert linearly polarised light from insertion devices to circular, elliptical or vertical polarisation. They have been installed at several synchrotron endstations,[2-4] providing polarised x-rays with a variety of applications, for example magnetic measurements of compounds containing transition metal ions such as Fe or Co. The polarization rotation originates from the birefringence of perfect crystals near Bragg conditions in forward diffraction. This is energy-dependent and therefore requires accurately positioning the QWP on the order of tens of micro-radians (~1 milli-degree) of the Bragg angle. Such accuracy requires reliable rotation stages; however, these are often limited by the speed of positioning, making them impractical for fast measurements. Furthermore, beam instabilities will lead to variations in intensity when alternating between two polarisation helicities for magnetic circular dichroism studies, which could mask the dichroic signals from samples.

To overcome these difficulties, we have mounted a diamond QWP on a fast optical scanning galvanometer.[5] This allows us to oscillate the QWP between the two birefringence angles with ~10 μrad accuracy at frequencies up to 1.5 kHz, with the prospect of increasing this frequency further. This leads to millisecond polarization switching, potentially speeding-up measurements. Furthermore, rapid polarization switching allows us to largely eliminate effects of longer timescale beam fluctuations, thereby increasing the signal to noise of the measurements.

In this poster, I will outline the details of the fast-oscillating QWP setup we have implemented at the GALAXIES beamline. I will demonstrate how the QWP angle can be calibrated as a function of x-ray energy to allow for dichroic measurements of resonant inelastic x-ray scattering (RIXS) spectra. Finally, I will show preliminary data on spectra collected using this setup compared to a static (slow switching) QWP. This method may generally benefit user research performed at GALAXIES aiming to exploit polarised X-rays.

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Ultrafast Dynamics of Magnetic Anisotropy and Magnetic Structure in Ferrimagnetic Alloys CoTb

M.M. Hemili[1], J. Lüning[4], C. Von Korff Schmising[3], B. Pfau[3],
F. Capotondi[5], V. Chardonnet[1], G.S. Chiuzbăian[1], M. Hennes [2],
E. Jal[1], B. Vodungbo[1]

[1] Laboratoire de Chimie Physique – Matière et Rayonnement (LCPMR) -
CNRS - Sorbonne Université, Paris, France.
[2] INSP - Institut des NanoSciences de Paris - CNRS - Sorbonne Université, Paris, France.
[3] Max-Born-Institut f¨ur Nichtlineare Optik und Kurzzeitspektroskopie, Berlin, Germany.
[4] Helmholtz-Zentrum Berlin f¨ur Materialien und Energie, Berlin, Germany.
[5] FERMI, Elettra-Sincrotrone Trieste, Basovizza, Trieste, Italy.

ABSTRACT

30 years ago, Beaurepaire made a groundbreaking discovery that initiated the field of Femtomagnetism. Through a pump-probe experiment on a pure nickel layer, Beaurepaire observed an ultrafast demagnetization. While numerous theories have been proposed to explain this phenomenon, there is still no consensus within the scientific community.

Additionally to this fundamental quest, the ultrafast magnetization dynamic has grown in interest due to its potential applications, such as All Optical Switching (AOS). AOS could significantly decrease information encoding times and is usually observed in ferrimagnetic rare-earth (RE)-transition metal (TM) alloys. In these materials, the two sub- systems exhibit distinct ultrafast magnetization dynamics when subjected to a femtosecond infrared pulse. To bring more information about the dynamics of such alloys with a spatial resolution, we carried out a Time resolved Small Angle X-ray Scattering (Tr-SAXS) experiment on Co-Tb. This experience was carried out at the beamline DIPROI at FERMI. We used the capacity of FERMI to deliver simultaneously two X-ray pulses of different wavelengths to probe the 3d Co and 4f Tb valence electrons, using the absorption edges $M_{2,3}$ (58.9eV) and N4,5 (150.5eV) of Fe or Co and Tb, respectively.

Our samples, grown by magnetron sputtering are amorphous. They have a high perpendicular magnetic anisotropie due to local order, and possess magnetic stripes domains. The experimental idea is to scatter X-rays on magnetic domains, and to use two CCD cameras ito record simultaneously the light scattered by Co and Tb atoms. This allows us to recover the demagnetization curve of Co and Tb simultaneously, for a pump laser of 800 nm with different fluences. By looking at the azimuthal integration of the scattering intensities as a function of the time delay, we can extract the time resolved evolution of magnetization, domain size and domain distribution of both TM and RE elements. The first results will be presented and discussed.

Spatial Resolution Studies of Optical Read out MPGD Gaseous Detectors at METROLOGIE Beamline

A. Cools[1], E. Ferrer-Ribas[1], T. Papaevangelou[1], E.C. Pollacco[1],
M. Lisowska[2], F.M. Brunbauer[2], E. Oliveri[2], F.J. Iguaz[3*]

[1] *IRFU, CEA, Université Paris-Saclay, F-91191 Gif-sur-Yvette, France*
[2] *European Organization for Nuclear Research (CERN), CH-1211 Genève 23, Switzerland*
[3] *SOLEIL Synchrotron, L'Orme des Merisiers, Départementale 128, 91190 Saint Aubin, France*
** Speaker: francisco-jose.iguaz-gutierrez@synchrotron-soleil.fr*

ABSTRACT

Optically read out gaseous detectors are used in track reconstruction and imaging applications requiring high granularity images. Among resolution-determining factors, the amplification stage plays a crucial role and optimizations of detector geometry are pursued to maximize spatial resolution. To compare Micro Pattern Gaseous Detector (MPGD) technologies, focused low-energy X-ray beams at METROLOGIE beamline of the SOLEIL synchrotron facility were used to record and extract point spread function widths with MICRO-Mesh Gaseous Structure (Micromegas) and Gas Electron Multiplier (GEM) detectors. Point spread function width of ≈108 μm for Micromegas and ≈127 μm for GEM foils were extracted. The scanning of the beam with different intensities, energies and across the detector active region can be used to quantify resolution-limiting factors and improve imaging detectors using MPGD amplification stages.

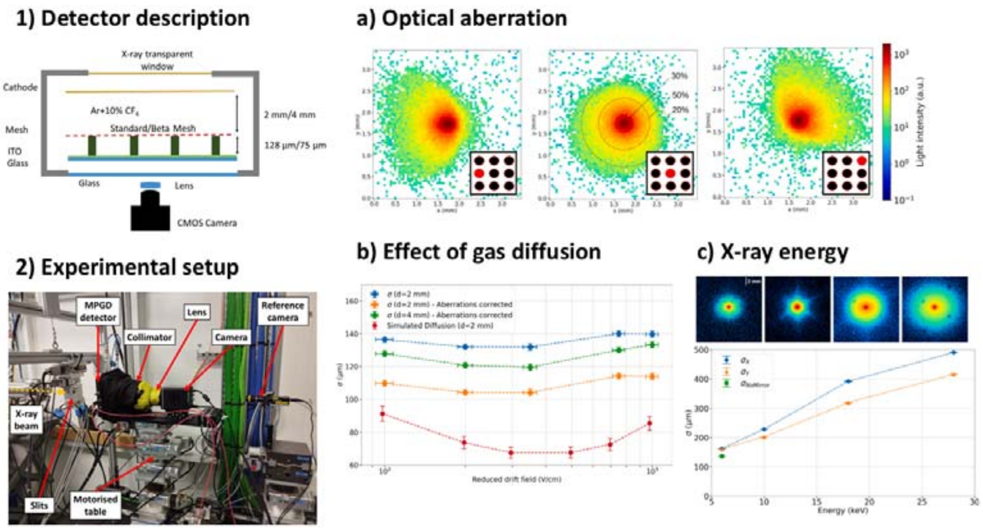

Figure 1: Schema of an optical readout detector and images from the experimental setup and different contributions to spatial resolution (optical aberration, gas diffusion and X-ray energy). Images published in [4].

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Charge Transfer Processes in Functional Materials Studied with Hard X-ray Core-hole Clock Spectroscopy

F.O.L. Johansson[1], E. Cartwright[1], B. Kammlander[1], T. Sloboda[2],
U.B. Cappel[1] and A. Lindblad[1]

[1] *Div. X-ray Photon Science, Dept. Physics and Astronomy, Uppsala University, Box 516,*
SE-751 20 Uppsala, Sweden
[2] *Div. of Applied Physical Chemistry, Dept. Chemistry, KTH - Royal Institute of Technology,*
SE-100 44 Stockholm, Sweden
Email: fredrik.johansson@physics.uu.se

ABSTRACT

One of the fundamental aspects of the performance of photovoltaic materials is charge separation which in its essence is charge transfer across interfaces. In order to study pure electronic charge transfer core-hole clock spectroscopy can be used. Following a core-resonant excitation, the decay of the metastable state is studied. This can either happen with the excited electron still localized on the atomic site (spectator decay) or it can tunnel away from the site of the core-hole (charge transfer decay), where the subsequent decay has the same final state as a normal Auger decay.

These processes have distinct spectral signatures and by monitoring these while changing the photon energy over a resonance, information on the probability of charge transfer can be extracted. Using the core-hole lifetime as an internal reference a charge transfer time can be calculated, hence core-hole clock spectroscopy. The probability of tunneling depends on the energy landscape surrounding the excited atom and using this a number of systems have been studied using this technique [1-6]. I will present how core-hole clock spectroscopy can be used to tool to study charge transfer in functional materials.

In lead sulfide quantum dots [4] and polymer heterojunction [1,5] the charge transfer exhibits different behavior dependent on the excess energy of the excited electron. For low excess energy the charge transfer is dependent on the composition or size whereas at higher excitation energy the tunneling behavior converges. It also turns out that the macroscopic best device performance can be correlated to the fastest charge transfer time measured with core-hole clock spectroscopy.

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Mixtures of Water, Ammonia and Methane under Extreme Pressure and Temperature Conditions

N. Kacem[1], L. Andriambariarijaona[1], F. Datchi[1], F. Alabarse[2],
G. Gabarino[3], B. Baptiste[1], N. Guignot[4], K. Béneut[1] and S. Ninet[1]

1. Institut de Minéralogie, de Physique des Matériaux et de Cosmochimie – IMPMC - UMR 7590
Sorbonne Université - 4, place Jussieu, Paris 75005, France
2. ELETTRA, Xpress Beamline, Trieste, ITALY
3. ESRF, ID15b beamline, Grenoble, France
4. SOLEIL, PSICHE beamline, Saint-Aubin, France

ABSTRACT

The mantle of the giant icy planets, Neptune and Uranus, is mainly composed of water, ammonia and methane ices, submitted to extreme conditions of pressure and temperature. However, the exact composition of the mixture and which compounds are stable in the ice layer are unknown. In order to build robust interior models of these planets and of the icy satellites, it is crucial to study the phase diagram of the ternary mixtures (H_2O, NH_3, CH_4), for various compositions and over a large range of pressure and temperature conditions.

Binary water/ammonia mixtures are also interesting for fundamental physics as they present both homonuclear and heteronuclear H-bonds. At ambient pressure, ammonia and water form 3 stoichiometric compounds ($xH_2O:yNH_3$): ammonia monohydrate (AMH,1:1), ammonia hemihydrate (AHH, 1:2) and ammonia dihydrate (ADH, 2:1). In the last years, our group has determined the HPHT phase diagrams of these three hydrates and discovered several phases that are common to the three compositions [1-4]. In the present work, we extended the experimental exploration of the water-ammonia mixtures by studying the HP-HT phase diagrams of several other compositions of the (H_2O-NH_3) system, using visual observations, Raman spectroscopy and X-ray diffraction experiments conducted in different synchrotron facilities (SOLEIL/ESFR/ELETTRA). The latter has enabled to draw the isothermal binary diagrams (pressure, composition) over the temperature range 300-700 K. More recently, a first synchrotron experiment at the PSICHE beamline of SOLEIL was conducted on different compositions of the (H_2O, NH_3, CH_4) mixtures, in order to study the influence of methane on the system. In this presentation, we will expose the results obtained both for the binary and ternary mixtures.

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Dichroism and SRARPES Studies of W(321) and W(-3-2-1)

Y. Lassailly[1], F. Sirotti[1], N. Lewis[2,3], E. Seddon[2,3], K. Ahrendsen[4],
T. Gay[5], S. Jenkins[6], J. Minár[7], R. Salazar[7], L. Nicolaï[7],
A. Pulkkinen[7], A. Bendounan[8]

[1]LPMC, CNRS & Ecole Polytechnique, Palaiseau, France.
[2]The Cockcroft Institute, Daresbury Laboratory, Sci-Tech Daresbury, Warrington, UK.
[3]The School of Physics and Astronomy, Photon Science Institute,
The University of Manchester, Manchester, UK.
[4]Smith College, Northampton, Massachusetts, USA.
[5]Department of Physics and Astronomy, The University of Nebraska, Lincoln, Nebraska, USA.
[6]Ysuf Hamied Department of Chemistry, University of Cambridge, Lensfield Road, Cambridge, UK.
[7]University of Western Bohemia, Pilsen, Czech Republic.
[8] Synchrotron Soleil, France.

ABSTRACT

DICHROIC INVESTIGATIONS OF CHIRAL SINGLE CRYSTAL W SURFACES

We present ARPES and CDAD experimental results that probe the first few atomic layers of W(321). Both spectroscopic techniques are complemented by ground-state DFT electronic structure calculations. The work has confirmed the presence of a Dirac-like surface-resonance confined to the first few atomic layers and at a slightly reduced binding energy to Rashba-like bands. The computational results show an intricate spin pattern. The CDAD spectra will give results that will be complementary to those obtained from the spin-polarisation of the photoemitted electrons at specific k points. These are powerful tools that have been shown to resolve the topological and quantum-geometrical structural character in momentum space.

Structure and Ferroelectric Control of GeTe Thin Films using Vicinal Si Substrates

L. Meynier, F. Cheynis, F. Leroy
Aix Marseille Université, CNRS, CINAM, AMUTECH, Marseille, France

ABSTRACT

Ferroelectric Rashba semiconductors are a novel class of materials with strong potential for spintronic applications1 . On GeTe thin films, the ferroelectric polarization has been shown to reverse in an electric field2 , and the spin chirality of the band structure has been consistently changed3 . α-GeTe has a spontaneous polarization along the pseudocubic pc direction. Since ferroelectricity and ferroelasticity are intimately related properties it is possible also to adjust the ferroelectric domains structure using stress fields4,5 . Considering the key role of atomic steps in interfacial stress relaxation mechanisms, we propose to use substrates with a controlled miscut to modify the induced stress in the GeTe film and therefore tune the ferroelectric domains structure. In this perspective we have grown GeTe films by MBE on vicinal Si substrates with a miscut angle between 2° and 10° with respect to Si(111) substrate. The ferroelectric state has been characterized by x-ray diffraction, TEM, SEM and LEEM. The role of the miscut angle on the ferroelectric domains is clearly evidenced: the miscut of the Si substrates break the interface symmetry and favor one type of ferroelectric domain. We also show that the surface morphology and the presence of mirror domains is strongly correlated to the miscut angle of the substrate. At last the size decay of ferroelectric domains during annealing is attributed to the thermal stress arising from significant differences in the linear thermal expansion coefficients between Si and GeTe5 .

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Comparative Study of Hematite-based Photoanodes using Operando STXM and Raman Spectroscopy - Methods and First Results

M. Mockbel[1,2], L. Schmidt[2], B. Meddas[3], K. Chaouchi[1], S. Blanchandin[1], O. Ersen[2], D. Stanescu[3], S. Stanescu[1]

[1] Synchrotron Soleil, l'Orme des Merisiers, Départementale 128, 91190 Saint-Aubin, France
[2] Institut de physique et de chimie des matériaux de Strasbourg (IPCMS),
23 Rue du Loess Bâtiment 69, 67200 Strasbourg
[3] Service de Physique de l'État Condensé (SPEC), CEA, CNRS UMR 3680, Université ParisSaclay,
Orme des Merisiers, CEA Saclay, 91191 Gif-sur-Yvette Cedex, France

ABSTRACT

Hematite-based (Ti:α-Fe_2O_3) photoanodes, catalytically activated with NiO_xH_y, were studied using Raman and STXM (*Scanning Transmission X-rays Microscopy*) in operando conditions.

First, we synthesized akaganeite ($Fe^{3+}O(OH,Cl,Ti)$) nanorods on F:SnO_2 (FTO) – coated glass substrates using the Aqueous Chemical Growth (ACG) method employing aqueous solutions of metal salts precursors ($FeCl_3$ and $TiCl_3$). Then, Ti:α-Fe_2O_3 nanorods were obtained by annealing in nitrogen atmosphere at 600°C. NiO_xH_y was subsequently deposited onto the hematite samples via electrodeposition in an electrochemical cell using aqueous $NiCl_3$ as electrolyte, hematite sample as the working electrode, a Pt electrode as cathode and Ag/AgCl electrode as reference.

Both Raman and STXM operando setups and associated methods used to characterize the hematite based photoanodes are described. Preliminary results demonstrate the feasibility of the operando approaches applied for our samples. NiO_xH_y oxidoreduction is evidenced for the expected electrochemistry conditions.

Electronic Structure Study of Alloys of 2D Transition-metal Dichalcogenides

H. Rojas-Páez[1,*], A. Antezak[2], J. Pazos[3], E. Frantzeskaikis[2], F. Fortuna[2], J. A. Galvis[4], P. Giraldo-Gallo[1], A. F. Santander-Syro[2]

[1]Universidad de los Andes, Bogotá, colombia;
[2] ISMO, Université Paris-Saclay, Orsay Cedex, France;
[3]Universidad Central, Bogotá, Colombia;
[4]Universidad del Rosario, Bogotá, Colombia.
[*]ha.rojas@uniandes.edu.co

ABSTRACT

Transition-metal dichalcogenides are a family of materials that have been of interest because of their 2D intrinsic character, their novel properties and exotic ground states including charge density wave, superconductivity and topological insulators (1). Alloys of TMDs are a platform to study the inherited properties of parent compounds to their mixtures, and to look for the optimization of certain properties as doping changes.

The emergence of multiferroic states was recently shown in alloys of WTe_2 and WSe_2, namely $W(Te_xSe_{1-x})_{2(1-\delta)}$ being x the tellurium and δ the chalcogen vacancies fraction (2). Photoemission (PS) and Angle-resolved Photo-emission spectroscopy (ARPES) have been used to study pure compounds WTe_2 [3-6], and WSe_2 (7-11) but to the best of our knowledge it does not exist any experimental report on the intermediate compositions $W(Te_xSe_{1-x})_{2(1-\delta)}$. It has been observed that the structure of the layered alloys $W(Te_xSe_{1-x})_2$ is modified from 2H to 1Td from changing the tellurium doping fraction.

In this work we present a study of the electronic structure performed by ARPES measurements of a set of samples $2H\text{-}W(Te_xSe_{1-x})_{2(1-\delta)}$. We will present measurement of band structure at ΓM, and ΓK high symmetry planes, and energy maps at different binding energies for samples with different tellurium doping fractions. For future work, we plan to explore the electronic structure features for both higher tellurium, $x>70\%$, and chalcogen, $|\delta|>20\%$, fractions in which the alloys are expected to be multiferroic.

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Influence of Applied Deformation on Magnetic Properties: Ferromagnetic Ni60Fe40 Thin Films Deposited on Polymeric Substrate

A. Toledano Povedano

Institut Pprime, France
alejandro.toledano.povedano@univ-poitiers.fr

Prof. Dr. P-O.Renault, Institut Pprime, France - pierre.olivier.renault@univ-poitiers.fr
Dr. E. Haltz, Laboratoire des Sciences des Procédés et des Matériaux (LSPM), France - eloi.haltz@univ-paris13.fr
Prof. Dr. A. Michel, Institut Pprime, France – anny.s.michel@univ-poitiers.fr
Prof. Dr. F. Zighem Laboratoire des Sciences des Procédés et des Matériaux (LSPM), France - zighem@univ-paris13.fr
Dr. P. Godard, Institut Pprime, France - pierre.godard@univ-poitiers.fr
Prof. Dr. D. Faurie, Laboratoire des Sciences des Procédés et des Matériaux (LSPM), France - faurie@univ-paris13.fr
Dr. D. Thiaudière, Soleil Synchrotron, France - dominique.thiaudiere@synchrotron-soleil.fr
Mr. P. Joly, Soleil Synchrotron, France - philippe.joly@synchrotron-soleil.fr
Mr. N. Girodon-Boulandet, Laboratoire des Sciences des Procédés et des Matériaux (LSPM), France - noel.girodon@lspm.cnrs.fr
Dr. C. Mocuta, Soleil Synchrotron, France - cristian.mocuta@synchrotron-soleil.fr

ABSTRACT

Metallic ferromagnetic thin films are essencial in devices including sensors, data storage, and signal processing1 . With the rise of flexible electronics, understanding the relationship between magnetic properties and mechanical deformations in the low and high strain regimes is critical. These deformations induce homogeneous elastic strains and strain heterogeneities due to crystalline defects and cracks, impacting the magnetic properties of films through magnetostriction and dipolar interactions2 . This study focuses on how mechanical strain and controlled crack propagation affect the magnetic properties of thin films on polymer substrates, aiming to reveal the relationship between controlled microstructural changes (residual stress, film thickness) and magnetic properties (from initial strain to crack onset and subsequent propagation). These insights are critical for developing flexible magnetic devices that maintain performance under mechanical stress. A unique setup was developped at Synchrotron SOLEIL (DiffAbs beamline), combining four techniques to study in situ the crystalline and magnetic properties of the sample subjected to equibiaxial or sequenced uniaxial tensile testing: X-ray diffraction to monitor the local lattice strain, digital image correlation to measure macroscopic distortions, electrical resistivity to reveal the crack onset and Magneto-Optical Kerr Effect to track the evolution of magnetic reversal. Ni60Fe40 thin films with varying thicknesses (20 and 200nm) have been deposited by ion beam sputtering on flexible polymer substrates and characterised under strain with this setup. Findings show how crack density (which varies with thickness3) influences the material's magneto-mechanical properties. The hysteresis loops initially show a square shape. As applied deformation increases, the loops change and exhibit features typical of a direction that resists magnetization, attributed to the negative magnetostrictive coefficient of Ni60Fe40. Beyond the maximum of the lattice strain, the loops appear to return to a square shape.

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In Situ Monitoring of Yttrium Films Structural Evolution under Very Low Hydrogen Pressure using X-ray Diffraction at SIXS beamline

O. Thomas[1], C. Kutyla[2], C. Bessouet[2], Y. Garreau[3], A. Coati[3],
S. Escoubas[1], C. Guichet[1], S. Lemettre[2], L. Leroy[2], A. Bosseboeuf[2],
T. Sauvage[4], O. Wendling[4], A. Bellamy[4], and J. Moulin[2]

1 Aix Marseille Université, Université de Toulon, CNRS, IM2NP, 13013 Marseille, France
2 C2N, Centre de Nanosciences et de Nanotechnologies, Université Paris-Saclay,
CNRS, UMR 9001, 91120 Palaiseau, France.
3 Synchrotron SOLEIL, L'Orme des Merisiers, Saint-Aubin, BP 48,
91192 Gif-sur-Yvette Cedex - France
4 CEMHTI, Conditions Extrêmes et Matériaux : Haute Température et Irradiation,
Université d'Orleans, UPR 3079 CNRS, 45071 Orléans Cedex 2, France.

ABSTRACT

The equilibrium phase diagram Y-H [1] shows the formation of several hydrides as well as a hexagonal Y(H) solid solution. The affinity of yttrium for hydrogen and also for oxygen makes this rare earth metal an interesting component of transition metal getter alloys [2]. Getter films are used to maintain a static vacuum in a sealed volume based on their capacity to sorb residual gas molecules. In the present work we have investigated the behavior of evaporated yttrium thin films during ramp anneals under low hydrogen pressure using X-ray diffraction at SIXS beamline at SOLEIL synchrotron. Out-of-plane radial scans are recorded on a 2D hybrid pixel detector during annealing. In-plane radial scans have been recorded only before and after annealing to preserve time resolution. 1D I(q) patterns are obtained via radial integration and Bragg peaks are fitted with an analytical function for quantifying the structural evolutions (integrated intensity, integral breadth, strain). Comparing films annealed under UHV ($P = 9. 10^{-10}$ mbar) with those annealed under hydrogen ($P_{H2} = 7.10^{-5}$ mbar) allows separating thermoelastic Bragg peak shifts from those arising from hydrogen uptake. All these eigen strains are treated in an anisotropic framework and thermo-chemo-elastic calculations allow extracting the average hydrogen concentration in the film as a function of temperature. In a later stage diffraction peaks attributed to YH_2 phase are detected. The extremely high affinity of yttrium for oxygen does not prevent film oxidation even under UHV at elevated temperature. The competition between hydrogenation and oxidation will also be presented and discussed.

ACKNOWLEDGMENTS

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Satellite & Tutorials

Tuesday, January 28th

14:00 -17:00

Tutorial 1 – The latest developments of FASTOSH

Gautier Landrot – SOLEIL Synchrotron (SAMBA beamline)

PHENIX Room

Tutorial 2 – Surface diffraction - Data treatment and analysis

Fabien Cheynis, Alessandro Coati, Andrea Resta, Alina Vlad, Yves Garreau – S2UO

LIBRA Room

Satellite – The new CryoEM - POLARIS

Paloma Fernandez-Varela, Pierre Legrand, Eric Larquet, Heddy Soufari – S2UO

SOLEIL Auditorium

TUTORIAL 1

The latest developments of FASTOSH

Tuesday, the 28th of January, 2025

Since the latest tutorial given at SOLEIL on « FASTOSH », which was held in 2023 at the 17th SOLEIL User Meeting, a number of functionalities have been added to this SOLEIL home-made XAFS data treatment software, currently available for free download on SAMBA beamline's website.

Location: SOLEIL Reception Building

Organizers: Gautier Landrot (SAMBA)

Number of participants: 50

This presentation will be essentially a live demo of the program by its author, to interactively showcase:

- Its main functionalities, especially those that are unique to the program, and demonstrate their potential usefulness in many fields of science and when conducting experiments in *operando* conditions at the XAFS beamline.
- Its newest functionalities, including:
    - Tools to visualise Wavelet Transformation of the EXAFS
    - Tools to interpret Fourier Transform or Wavelet Transform of the EXAFS, where the EXAFS is quickly modelled using single scattering paths generated by FEFF8L
    - Multiple new functionalities for MCR-ALS data treatment, including those to treat XAFS data and non-XAFS data collected by laboratory bench-top spectrometers. All these new functions were not available in the original Jaumot et al. MCR-ALS GUI 2.0 toolbox, which is customized [1]
    - Miscellaneous, including a function for multiple linear combination fitting

For this presentation, the use of a personal laptop won't be required. This meeting is essentially meant to be a convivial and interactive opportunity to get an update from the program's author on the main functions of the software and how to use them, as well as exchange and interact with each other, notably to ask specific questions on the software or request/suggest the development of additional functions that could be useful for the User Community.

REFERENCE

1- Jaumot, J., de Juan, A. and Tauler, R. (2015) MCR-ALS GUI 2.0: New features and applications. Chemometrics and Intelligent Laboratory Systems 140, 1-12.

TUTORIAL 2

Surface X ray Diffraction (SXRD) data reduction and analysis

Tuesday, the 28th of January, 2025

In this tutorial the reduction and analysis of surface diffraction and scattering data will be shown for users.

Location: **Reception Building, SOLEIL**

Organizers: SixS beamline (A. Coati, Y. Garreau, A. Resta, M. Sauvage-Simkin, A. Vlad), GRADES group (F. Picca, E Farhi) and S2UO (F. Cheynis)

Number of participants: 50

After a brief reminder on the technique basis and data collection strategy, the participants will be guided in the use of BINoculars-NG, a program developed at ESRF and refurbished at SOLEIL that allows to reduce the data, starting from images collected by a 2D hybrid detector. Data can be projected in a datacube in different spaces, depending on the experimental needs. Available spaces are *q*-space, *hkl*-space, angular-space, mixed (time, angle, q,…) – spaces. Once the dataset reduced in the desired space, data integration along desired space directions can be achieved.

The tutorial will show how to manage the data collected on a sample surface and how to obtain the surface structure starting from the diffusion rods integrated by BINoculars. ROD program developed by E. Vleg will allow to solve the structure.

All the users who want to discover surface x-ray diffraction data treatments available at the SixS beamline are welcome.

The participant needs a PC with no special requirements but a wifi connection. The program and the data will be available on a dedicated space on the DARTS platform.

(1) http://dx.doi.org/10.1107/S1600576715009607
(2) https://doi.org/10.1107/S0021889899013655

SOLEIL SYNCHROTRON

SATELLITE

The new CryoEM – POLARIS

Tuesday, the 28th of January, 2025

Location: SOLEIL Auditorium Main Building

Organizers: Paloma Fernandez-Varela, Pierre Legrand, Eric Larquet, Heddy Soufari – S2UO

Number of participants: No limit

POLARIS

1. Installation and first results of expert users (**E. Larquet/H. Soufari / P. Legrand,** synchrotron SOLEIL)
    - SPA (**C. Madru,** Ecole Polytechnique)
    - Tomography (**C. Sauvanet,** I2BC)
2. Coordination with I2BC – Glacios (**S. Bresannelli,** I2BC)
3. Operation (**A. Thompson,** Synchrotron SOLEIL)
    - Access mode
    - Grides preparation and storage
    - Data analysis and storage
4. Round table: Question/Answers (**PD. Coureux,** IBCP Lyon **/ Y. Mechulan,** Ecole Polytechnique)

LIST OF COMMERCIAL EXHIBITORS

allectra	**ALLECTRA** **Gilles ROUSSEAU**
ALTEC equipment	**ALTEC EQUIPMENT** **Arnaud AUBERT**
EDWARDS	**EDWARDS VACUUM** **Cedric SOURCIS** **Claire LE JEUNE**
FRANCESCIENTIFIQUE	**FRANCE SCIENTIFIQUE** **Denis RADOSAVKIC**

VACUUM TECHNOLOGY Hositrad	**HOSITRAD** **Jurgen TOMASSEN** **Frans VAN ROOSMALEN**
MICROVISION INSTRUMENTS	**MICROVISION** **Gilles VAILLANT**
neyco HEF® PHOTONICS	**NEYCO** **Baptiste TARIF** **Solène GAUBERT**
OMEGA PHYSICS	**OMEGA PHYSICS** **Matthieu CAVELLIER** **Sandra CAVELLIER**

Physical Instruments	**PHYSICAL INSTRUMENTS** **Ludovic RUSE**
PN Detector	**PN DETECTOR** **Andrew FRAM**
saes high vacuum	**SAES** **Dario NICOLOSI**
SPECSGROUP	**SPECS** **Brice HOFF**

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