XVI International School and Symposium on Synchrotron Radiation in Natural Science





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# Synchrotron Radiation in Natural Science

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#### **Polish Synchrotron Radiation Society**

#### Location:

ul. Radzikowskiego 152 31 – 342 Kraków

#### Address:

dr hab. inż. Anna Wolska Instytut Fizyki PAN, al. Lotników 32/46 02 – 668 Warszawa

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https://synchrotron.org.pl

## SYNCHROTRON RADIATION IN NATURAL SCIENCE

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## CONTENTS

Welcome to the 16 <sup>th</sup> ISSRNS	5
Committees	6
Invited speakers	8
The PSRS Award Winners 2024	10
Polish participation in the ESRF in a nutshell	11
Programme	12
Posters	17
Abstracts	19

#### Welcome to the 16<sup>th</sup> International School and Symposium on Synchrotron Radiation in Natural Science

The International School and Symposium on Synchrotron Radiation in Natural Science (ISSRNS) gathers synchrotron radiation users from Poland and abroad for over 30 years. The 1st ISSRNS was organized by the Polish Synchrotron Radiation Society (PSRS) in 1992. Its chairs were prof. Julian Auleytner from Institute of Physics PAS in Warsaw and prof. Andrzej Kisiel from the Jagiellonian University in Krakow, both the outstanding scientists, group leaders and users of synchrotron radiation. On their initiative, a year earlier during the first National Symposium of Users of Synchrotron Radiation in Przegorzały the PSRS was established. The main aim of the Society had been the popularization of research with the use of synchrotrons. It was done by the regular organization of scientific schools during which participants could learn about the synchrotrons and synchrotron radiation as well as to obtain information what kind of research can be performed and where. The conferences had helped to bring together Polish scientists interested in using synchrotron radiation and created a forum not only for learning about techniques but also for exchanging experiences in practical matters. From the beginning the scientists from major synchrotron centers have been invited to share their knowledge and expertise. After more than 30 years Polish scientists are a part of a synchrotron world but the ISSRNS conferences are still a place to learn about fascinating science and new techniques, to meet interesting scientists from all over the world, establish new collaborations and discuss ones results.

The ISSRNS conferences are organized by members of the PSRS form different scientific centers in Poland. Their location changes from little mountain resorts to big cities like Warsaw or Krakow. This year, the 16<sup>th</sup> ISSRNS is organized by the Institute of Physics, Polish Academy of Sciences, in Szczyrk, a mountain resort in the south of Poland.

Thanks to the effort of PSRS represented by prof. Krystyna Jabłońska from Institute of Physics PAS, Poland joined the ESRF consortium as an associate member already in 2004. Since then Polish scientists can perform their research in this modern and innovative facility. From the beginning the access is ensured by the grants of Ministry of Science and Higher Education. The recent one is titled "*Polish contribution to the European Synchrotron Radiation Center*" (agreement No. 2021/WK/11) and it also offers funding for participation in conferences for scientists with Polish affiliation presenting the results of research conducted at the ESRF. In addition, current grant supports the organization of the 16<sup>th</sup> ISSRNS conference, which provides a good occasion to organize a special session dedicated to the ESRF-EBS to show supreme opportunities for the exploration of materials and living matter in a very wide variety of fields which are open to the scientific community after the recent upgrade.

Poland also belongs to the XFEL consortium. The Network of Excellence XFEL Centers was established in order to provide support to the Polish scientific community in utilizing the European XFEL. Within this intention the XFEL session is organized, to show and promote time-resolved research and give space for the users to discuss their ideas.

From the beginning, one of the goals of the PSRS had been to initiate and support initiatives leading to the construction of a Polish synchrotron radiation source. It was successful and in 2018 SOLARIS National Synchrotron Radiation Centre in Kraków welcomed first users. During our meeting you can learn about the research conducted there which will be presented by the users as well as the beamline scientists.

We also give floor to the winners of the Polish Synchrotron Radiation Society Award from the fourth and fifth edition for the best PhD thesis and publications containing the results of research using synchrotron radiation or free electron lasers.

We welcome all participants. I believe you would find exciting science here, nice place to share and discuss your research, form new acquaintances and get new ideas.

Conference Chair Anna Wolska

## 16<sup>th</sup> International School and Symposium on Synchrotron Radiation in Natural Science

25 - 20 May 2025, Szczyrk, Poland

Organized by Institute of Physics Polish Academy of Sciences in cooperation with Polish Synchrotron Radiation Society with the support of the Polish Ministry of Science and Higher Education within a grant no. 2021/WK/11 which provides Polish scientists with access to the European Synchrotron Radiation Facility.

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## With the support of:

The XVI ISSRNS conference is supported by the Polish Ministry of Science and Higher Education within a <u>grant no. 2021/WK/11</u> which provides Polish scientists with access to the European Synchrotron Radiation Facility.



## Ministry of Science and Higher Education Republic of Poland



## Invited speakers

#### Marine Cotte, ESRF, France

Shedding EBS light on artworks to get insight into their creation and to better preserve them

**Catherine Dejoie**, ESRF, France Advances in high-resolution powder diffraction for applications in materials chemistry

**Thomas Feurer**, European XFEL GmbH Introduction to XFELs

**Magdalena Fitta**, Institute of Nuclear Physics PAS, Poland Unraveling charge transfer dynamics in switchable molecular magnets through X-ray absorption spectroscopy

**Zbigniew Kaszkur**, Institute of Physical Chemistry PAS, Poland In situ nanopowder diffraction- between Bragg and diffuse scattering

**Michael Knapp**, Karlsruhe Institute of Technology, Germany Probing Batteries with X-rays and Neutrons from Cradle to Grave

**Esko Kokkonen**, MAX IV Laboratory, Sweden Ambient pressure XPS at MAX IV Laboratory and how it is used for atomic layer deposition research

**Dorota Koziej**, University of Hamburg, Germany In situ X-Ray Synchrotron Methods for Exploring the Length Scale in Nanostructured Materials and Devices

**Benedikt Lassalle**, ITODYS, CNRS/Université Paris Cité; Synchrotron SOLEIL, France In situ/operando X-ray absorption spectroscopy coupled to electrocatalysis

Henrik Till Lemke, Paul Scherrer Institute PSI, Switzerland

**Kirill Lomachenko**, ESRF, France *Operando XAS for catalysis at BM23 and ID24-DCM beamlines of the ESRF* 

**Maciej Lorenc**, University of Rennes, France; CNRS, The University of Tokyo, Japan *Ultrafast x-rays reveal new opportunities in nanostructured quantum materials* 



**Paola Luches**, Istituto Nanoscienze, CNR, Italy Ultrafast dynamics of photoexcited states in cerium oxide investigated by pump-probe XAS at the EuXFEL

**Christopher Milne**, European XFEL, Germany *Probing dynamics in biology, chemistry and physics using ultrafast X-rays* 

**Przemysław Nogły**, Dioscuri Centre for Structural Dynamics of Receptors, Poland *Time-resolved serial crystallography at synchrotrons and XFELs* 

**Ewa Partyka-Jankowska**, SOLARIS, Poland *SOLARIS National Synchrotron Radiation Centre: infrastructure for research* 

**Oliver Rader**, Helmholtz-Zentrum Berlin, Germany *Angle-resolved photoemission of topological materials* 

**Francesco Sette**, ESRF, France *The delivery of the ESRF EBS and its Upgrade Programme* 

**Montserrat Soler-López**, ESRF, France *Advancements in Biological Sciences at the European Synchrotron* 

**Yoshio Takahashi**, The University of Tokyo, Japan High energy resolution fluorescence XAFS for the speciation of various elements in geochemical and environmental samples

## The PSRS Award Winners 2024

We are pleased to announce the Winners of the 5<sup>th</sup> edition of the Polish Synchrotron Radiation Society (PTPS) Award, as decided by the Award Committee:

Dr. Ashutosh S. Wadge received the award in Category I for his doctoral dissertation titled:

## "Exploring electronic properties of topological semimetals TaAs<sub>2</sub> and NbP: crystal growth, electron transport and ARPES studies."

https://www.ifpan.edu.pl/nauka/rozprawy-doktorskie/2023/ashutoshwadge.html

The award in Category II for a scientific publication was granted to the Authors of the article published in Advanced Functional Materials (2023, 33, 2304359):

Mohammad S. Shakeri, Żaneta Świątkowska-Warkocka, Oliwia Polit, Tatiana Itina, Alexey Maximenko, Joanna Depciuch, Jacek Gurgul, Marzenna Mitura-Nowak, Marcin Perzanowski, Andrzej Dziedzic, and Jarosław Nęcki, for the publication:

### "Alternative local melting solidification of suspended nanoparticles for heterostructure formation enabled by pulsed laser irradiation."

https://onlinelibrary.wiley.com/doi/full/10.1002/adfm.202304359

Prof. Wojciech M. Kwiatek was recognized in Category III for his outstanding contribution to the development of the Polish synchrotron community, including long-term dedication to the advancement of Polish PS and FEL users and the promotion of synchrotron research in Poland.

Prof. Kwiatek is a founding member of PTPS and has actively participated in organizing the ISSRNS and KSUPS conferences as a member of the Organizing and Program Committees. He was the main organizer and Chair of the International XAFS 2018 Conference, significantly contributing to the international visibility of Polish PS users. In 2020, he was awarded the title of Ambassador of Polish Congresses by the Program Council of the Polish Congress Ambassadors initiative.

The PTPS Award Winners will give lectures during the special session, which will take place on Friday, 30 May 2025.



#### Polish participation in the ESRF in a nutshell

A. Wolska<sup>1</sup>, A. Reszka<sup>1</sup> and J. Libera<sup>1</sup>

<sup>1</sup>Institute of Physics Polish Academy of Sciences, PL-02668 Warsaw, Poland

e-mail: esrf-polska@ifpan.edu.pl

The European Synchrotron Radiation Facility (ESRF) is a modern research infrastructure located in Grenoble (France) open to users since 1994. It has been built and operates as an international consortium and it is financed by the contributions paid by the Member Countries (contribution above 4 %) and Scientific Associated Members (below 4 %). Poland is an Associate Member since 2004, first, with contribution of 0.6 % and since 2006 with contribution of 1% of the infrastructure budget. From the beginning Polish contribution to the ESRF is possible thanks to the Ministry of Science and Higher Education grants. The current one started in April 2021 and will end in March 2026. The grant is led by the *National Consortium of Scientific Institutions Interested in the Use of the European Synchrotron Radiation Source in Grenoble* and is coordinated by the Institute of Physics, Polish Academy of Sciences.

At the ESRF 46 beamlines are open to users offering possibility to conduct research in the fields of physics, chemistry, cultural heritage, environmental science, structural biology, medical applications and many others. All scientists with Polish affiliation can submit their projects. The beamtime to scientists from each country is granted in accordance with its contribution level.

The current Ministry grant additionally offers a co-financing for conference participation for scientists with Polish affiliation who want to present the results of research conducted at the ESRF. The full funding regulations can be found on the project website: https://esrf.ifpan.edu.pl/ together with other information.

Research results presented at conferences or published in scientific journals must include acknowledgments to the grant financing Poland's access to the ESRF: *The access to the ESRF was financed by the Polish Ministry of Science and Higher Education, dec. no. 2021/WK/11.* 



SUNDAY, 25 MAY 2025		
17:00 - 19:00	Arrival & Registration	
19:00 - 22:00	Welcome & Barbecue	

MONDAY, 26 MAY 2025		
9:00 - 9:20	Opening	
Unlocking new X-ray Science: ESRF after EBS Upgrade - part I		
	Chair: Karolina Jurkiewicz	
9:20 - 10:20	Francesco Sette The delivery of the ESRF EBS and its Upgrade Programme	M0_I1
10:20 - 11:00	Montserrat Soler Lopez Advancements in Biological Sciences at the European Synchro	otron MO_I2
11:00 - 11:30	Coffee break	
	Unlocking new X-ray Science: ESRF after EBS Upgrade - part II	
	Chair: Francesco Sette	
11:30 - 12:10	<b>Marine Cotte</b> Shedding EBS light on artworks to get insight into their creation and to l preserve them	better MO_I3
12:10 - 12:50	<b>Catherine Dejoie</b> Advances in high-resolution powder diffraction for applications in m chemistry	aterials MO_I4
12:50 - 13:10	<b>Houri S. Rahimi Mosafer</b> Effect of cationic substitution on structural properties of Ca <sub>3</sub> orthovanadates	(VO <sub>4</sub> ) <sub>2</sub> MO_01
13:10 - 13:30	<b>Cecilia Zito</b> Unraveling the mechanism of iron sulfide nanostructures formation: insight situ X-ray diffraction and spectroscopy studies	nts from in MO_O2
13:30 - 15:00	Lunch	
18:00 - 19:00	Dinner	
	Unlocking new X-ray Science: ESRF after EBS Upgrade - part III	
	Chair: Ewa Partyka-Jankowska	
19:00 - 19:40	Kiril Lomachenko Operando XAS for catalysis at BM23 and ID24-DCM beamlines of th	e ESRF MO_I5
19:40 - 20:00	<b>Lukasz Rogal</b> Polymorphisms and Early-Stage Crystallization of Zr-Based Glasses Induc Pressure	ed by High MO_O3
20:00 - 20:20	Karolina Jurkiewicz         Insight into the structure of liquid-crystalline itraconazole drug	M0_04
20:20 - 20:40	Maciej Jankowski Advanced Software Tools and Workflows for Synchrotron X-Ray Sca Analysis at ESRF: A User's Perspective	ittering MO_05



TUESDAY, 27 MAY 2025		
XFEL application to natural science - part I		
	Chair: Przemysław Nogły	
9:00 - 9:40	<b>Thomas Feurer</b> An introduction to X-ray Science and hard X-ray Free Electron Lasers	TU_I1
9:40 - 10:20	<b>Christopher Milne</b> Probing dynamics in biology, chemistry and physics using ultrafast X	(-rays <b>TU_l2</b>
10:20 - 11:00	<b>Paola Luches</b> Ultrafast dynamics of photoexcited states in cerium oxide investigated by probe XAS at the EuXFEL	r pump- T <b>U_I3</b>
11:00 - 11:30	Coffee break	
	Material science, nanotechnology and magnetism- part I	
	Chair: Dorota Koziej	
11:30 - 12:10	Oliver Rader Angle-resolved photoemission of topological materials	TU_14
12:10 - 12:50	<b>Esko Kokkonen</b> Ambient pressure XPS at MAX IV Laboratory and how it is used for ator deposition research	nic layer TU_I5
12:50 - 13:10	<b>Elżbieta Guziewicz</b> Scanning Photoelectron Microscopy (SPEM) of N-doped ZnO films: in carbon on the electronic band structure at the nanometer scale	mpact of TU_01
13:10 - 13:30	<b>Romisaa Abdelrahman</b> Integrating XRF and XCT for Understanding Medieval Ceramic F from Wawel Royal Castle	Production TU_02
13:30 - 14:30	Lunch	
14:30 - 19:15	Excursion	
19:15 - 20:30	Dinner	
20:30 - 22:00	Poster session Hall "Sala Wiosenna" – a	8. Floor



WEDNESDAY, 28 MAY 2025		
Material science, nanotechnology and magnetism- part II Chair: Marcin Sikora		
9:00 - 9:40	<b>Magdalena Fitta</b> Unraveling charge transfer dynamics in switchable molecular magnet X-ray absorption spectroscopy	ts through WE_I1
9:40 - 10:20	<b>Dorota Koziej</b> In situ X-Ray Synchrotron Methods for Exploring the Length Scale in Nan Materials and Devices	ostructured WE_12
10:20 - 10:40	<b>Sarah-Alexandra Hussak</b> Visualizing the Competition between Etching and Sulfurizatio X-ray ptychography and X-ray Absorption Spectroscopy	on via in situ WE_ <b>01</b>
10:40 - 11:00	Vitaliy Bilovol XAS and XMCD studies of spinel-type HEOs	WE_02
11:00 - 11:30	Coffee break	
	XFEL application to natural science - part II	
	Chair: Christopher Milne	
11:30 - 12:10	<b>Henrik Till Lemke</b> Insights on condensed matter functions by FEL x-ray pulses	WE_I3
12:10 - 12:50	<b>Maciej Lorenc</b> Ultrafast x-rays reveal new opportunities in nanostructured quantum m	naterials WE_I4
12:50 - 13:10	Michał Nowakowski Fs-XES of the NIR-active spin-flip emitters	WE_O3
13:10 - 13:30	<b>Jerzy Antonowicz</b> Bridging experiment and simulation: X-ray free-electron laser probin optically induced melting of Palladium	ng of WE_O4
13:30 - 15:00	Lunch	
16:30 - 18:00	Ryszard Sobierajski       Polish research at the European XFEL         XFEL - round table       Hall "Sala Audiowizualna" –	WE_15 3. Floor
19:00 - 23:00	Conference dinner	



THURSDAY, 29 MAY 2025		
Life science, structural biology and chemistry		
	Chair: Wontserrat Soler Lopez	
9:00 - 9:40	Zbigniew Kaszkur In situ nanopowder diffraction- between Bragg and diffuse scatteri	ng TH_I1
9:40 - 10:20	Przemysław Nogły Time-resolved serial crystallography at synchrotrons and XFELs	TH_12
10:20 - 10:40	<b>Piotr Guńka</b> Kinetic studies of the formation of arsenolite inclusion compound with hy	rdrogen TH_01
10:40 - 11:00	Pawel Grochulski The health and life sciences research in the Canadian Light Source	TH_02
11:00 - 11:30	Coffee break	
	X-ray Research for Ecologically Sustainable Society - part I Chair: Hitoshi Abe	
11:30 - 12:10	<b>Michael Knapp</b> Probing Batteries with X-rays and Neutrons from Cradle to Grave	тн_із
12:10 - 12:50	<b>Yoshio Takahashi</b> High energy resolution fluorescence XAFS for the speciation of cesil earth elements in geochemical and environmental samples	ım and rare TH_I4
12:50 - 13:10	<b>Muhmmad Jamshaid Shabbir</b> Structural characterization of carbon materials derived chestnut biomass	from TH_O3
13:10 - 13:30	<b>Diana Havryliuk</b> Electronic structure and chemical segregation process induced by rec conditions in $PbHfO_3$ single crystal	lox TH_O4
13:30 - 15:00	Lunch	
16:00 - 18:00	General meeting of PTPS (Polish Synchrotron Radiation Society) Hall "Sala klubowa" -	- 5. Floor
18:00 - 19:00	Dinner	
X-ray Research for Ecologically Sustainable Society - part II Chair: Ryszard Sobierajski		
19:00 - 19:40	<b>Benedikt Lassalle</b> In situ/operando X-ray absorption spectroscopy coupled to electroc	atalysis TH_I5
New opportunities: ELI, SOLARIS		
19:40 - 20:00	<b>Tomas Parkman</b> Ultrafast laser-driven X-ray sources at ELI Beamlines: From HHG and ray to LPA-driven sources	plasma X- TH_O5
20:00 - 20:20	Henryk Fiedorowicz Laser-plasma soft X-ray sources based on a gas puff target for an absorption spectroscopy and coherence tomography	pplication in TH_O6
20:20 - 20:40	Katarzyna Sowa PolyX@SOLARIS – beamline for multimodal microimaging and spectr	omicroscopy TH_07
20:40 - 21:00	<b>Maciej Kozak</b> Recent Progress in Construction of SMAUG Beamline at SOLARIS Nation Synchrotron Radiation Centre	al TH 08



#### FRIDAY, 30 MAY 2025 **Research at SOLARIS** Chair: Yoshio Takahashi Ewa Partyka-Jankowska SOLARIS National Synchrotron Radiation Centre: infrastructure for 9:00 - 9:40 research FR I1 Rafał Fanselow Unveiling the electronic structure of Zinc Selenide Quantum Dots with X-ray 9:40 - 10:00 FR\_O1 Spectroscopy Edyta Piskorska-Hommel Study of the occupation sites in the lattice crystal by different elements 10:00 - 10:20 composed of high-entropy alloys FR\_O2 Aleksandra Drzewiecka-Antonik Platinum complexes with thiourea derivatives as ligands: looking 10:20 - 10:40 for their bonding scheme FR\_O3 Alexey Maximenko Unveiling ASTRA: A Versatile tender XAS workhorse beamline at SOLARIS 10:40 - 11:00 Synchrotron FR\_O4 11:00 - 11:30 **Coffee break PTPS** award session Chair: Agnieszka Witkowska Mariusz Jaskólski Crystal structures of the elusive Rhizobium etli l-asparaginase reveal a peculiar 11:30 - 12:00 active site FR\_I2 Ashutosh Wadge Exploring electronic properties of topological semimetals TaAs<sub>2</sub> and NbP: crystal 12:00 - 12:30 growth, electron transport and ARPES studies FR\_I3 Mohammad Shakeri Aternative local melting solidification of suspended nanoparticles for 12:30 - 13:00 heterostructure formation enabledby pulsed laser irradiation FR\_I4 13:00 - 13:30 Closing 13:30-15:00 Lunch 15:00 Departure of conference bus to Bielsko-Biała



TUESDAY, 27 MAY 2025 - POSTER SESSION		
P1	S. Piotrowska Cathodoluminescence studies of gallium nitride nanowires with oxide layers coatings	
P2	<b>A. Kertmen</b> Characterization of Iron Oxide and Zinc Sulfite Nanoparticles in Atmospheric Pollution: PCA and LCF Analysis of XAS Data Using the SOLARIS ASTRA Beamline	
Р3	W. Saeed Computational Investigation of the Elastic Characteristics of Heusler Compounds	
P4	<b>A. Sulich</b> Defect structure of CdTe-based single- crystals for X-ray and gamma radiation detection purposes	
P5	<b>E. Guziewicz</b> Electronic structure of transition metals and rare-earth ions incorporated in the SnTe matrix	
P6	<b>W. Paszkowicz</b> Equation of state of Ca <sub>3</sub> Cr <sub>2</sub> Ge <sub>3</sub> O <sub>12</sub> garnet	
P7	<b>W. Gumulak</b> Experimental study of a new Heusler compound from chrome group - $Ti_2CrAl$ and $Ti_2MoAl$	
P8	<b>M. Pilch</b> Lanthanum distribution in highly La-doped SrTiO <sub>3</sub> crystals	
Р9	<b>T. Fok</b> Near edge X-ray absorption fine structure (NEXAFS) spectroscopy in the 0.2-1.5 keV spectral range using a laser-plasma soft X-ray source	
P10	<b>B.J. Kowalski</b> Nitride nanowire surface modifications induced by chemical treatment and oxide layer deposition	
P11	<b>W. Lulka</b> Orthorombic domains in powder RuO <sub>2</sub> at ambient conditions	
P12	<b>R. Anwar</b> Polaronic transport and magnetic field dependence of conduction parameters in rGO- CFO nanocomposites	
P13	<b>P. Piszora</b> Regulating the crystal structure of $LiMn_2O_4$ material by Zn doping	
P14	<b>M. Sikora</b> Resonant photoemission study of MnFePAs compounds exhibiting giant magnetocaloric effect	
P15	H. Abe Solid-liquid interface of water/SAE304 probed by TREXS (Total REflection X-ray Spectroscopy)	
P16	<b>I. Jacyna</b> Structural and surface morphology modifications of $Pd_{100-x}Si_x$ thin film induced by ultrashort pulsed laser annealing	
P17	<b>R. Stachura</b> Surface analysis of TiO <sub>2</sub> and Ti nanolayers modified with highly charged Xe <sup>q+</sup> ions using synchrotron radiation based XRR and GIXRF methods	
P18	<b>F. Aziz</b> Thermostructural properties of 6 and $\gamma$ polymorphs of lead orthovanadate (Pb <sub>3</sub> V <sub>2</sub> O <sub>8</sub> ) in a broad temperature range (298 K-973 K): An X-ray diffraction study	



P19	<b>R. Nietubyć</b> THz radiation yield from Polfel undulator absorption
P20	<b>A. Witkowska</b> Structural study of transition metal- doped ceria reduced in hydrogen atmosphere: XAS analysis
P21	<b>K. Balin</b> Surface and electronic properties of Mn-modified $PdTe_2$ and $Bi_2Te_3$
P22	<b>A. Wolska</b> Local structure around the Co, Ni and Cu dopants in the calcium orthovanadates as seen by X-ray
P23	<b>A. Wolska</b> Structural studies of bioactive Ag(I) complexes with coumarin derivatives
P24	<b>K. Rucińska</b> Impact of Copper Oxide Nanoparticles on Human Cystatin C - Cell Viability and In Vivo-like Flow Studies
P25	J. Maksim Influence of gold nanoparticles on amyloid beta aggregation process
P26	A. Drzewiecka-Antonik Mn(II) complexes with 2,4-D and MCPA herbicides: IR and XAFS studies
P27	<b>Y. Chen</b> Support Labs at the MAX IV Synchrotron Facility
P28	J. Majewski X-Ray Synchrotron Studies of Biomembrane - Protein Interactions at Air-Liquid Interfaces
P29	<b>P. Grzywa</b> PM10 Under the X-ray: Elemental Composition and Seasonal Variability in the Air in Krakow, Poland
P30	J.J. Kubacki Analysis of in situ localization of elements in the cellular structure of cancers of the head and neck region
P31	<b>M. Dominów</b> Degradation and regeneration analysis of noble metal ex-solution catalysts for dry and steam methane reforming
P32	<b>A. Sabik</b> Electronic structure of GeS <sub>0.5</sub> Se <sub>0.5</sub> crystal: band alignment, valence bands, and core- levels
P33	A. Sabik NAP-XPS - new end station at SOLARIS synchrotron
P34	<b>B. Wolanin</b> PHELIX beamline as an advanced tool dedicated to surface science
P35	<b>L. Alluhaibi</b> Spectroscopic Synergy at the ASTRA Beamline: Combining X-ray Absorption and Raman Spectroscopy for Advanced Analysis of Complex Materials
P36	K. Kujawska Tailoring the ceria properties by lanthanide and transition metal co-doping
P37	<b>K. Matlak</b> The Scanning Transmission X-Ray Microscope at SOLARIS optimized for fast scanning and environmental experiments
P38	<b>A. Figura-Jagoda</b> Understanding of mechanism of Cu reduction induced by photon beam in Cu thin films
P39	<b>O. Liubchenko</b> Time-Resolved X-ray Diffraction Investigation of Ultrafast Structural Changes in Fe
P40	M. Jankowski The ESRF After the EBS Upgrade: A New Research Opportunities



### The delivery of the ESRF EBS and its Upgrade Programme

F. Sette

**MO I1** 

European Synchrotron Radiation Facility, B.P.220, F-38043 Grenoble Cedex, France

\*e-mail: sette@esrf.fr

The ESRF has been carrying out a major reconstruction programme, which started in 2009 and ended in 2023. This programme, referred to as the ESRF Upgrade Programme, is grounded on ESRF role and mission of pioneering synchrotron science to the benefit of Europe and society at large. Synchrotron based science has in fact continued growing and is increasingly associated with innovative applications and supporting industrial programmes.

In 2004, together with Jose Goulon, I pointed out the growing importance of convergence among X-ray imaging and microscopy methods with established X-ray science scattering and absorption techniques, which had the potential to open innovative approaches on the exploration of the micro- and nano-world, and enable much more powerful investigations on the different structural hierarchies present in many complex materials and in living matter.

To this purpose, new adapted concepts – enhancing X-ray source and instrument performance to explore with high spatial resolution condensed and living matter – had to be developed. These considerations rapidly became the basis of a powerful Science Case supporting what then became the ESRF Upgrade Programme.

The implementation of the ESRF Upgrade Programme, divided in two phases – Phase I (2009-2015, ESRF Purple Book – 2007) and EBS (2015-2023, ESRF Orange Book – 2014), has enabled an almost complete reconstruction of the ESRF facility, with 27 new beamlines, a new storage ring-based X-ray synchrotron source (EBS the Extremely Brilliant Source), and new scientific and IT infrastructures and instruments.

Th new EBS storage ring came to operation with revolutionary performance in August 2020, enabling and opening new frontiers in X-ray science, and becoming the new role model for modern X-ray synchrotron sources around the world. Since summer 2024, a second EBS-like machine, the APS-U, in Argonne National Laboratory, has started operation, and with the ESRF-EBS is contributing marking the new paradigm of synchrotron science.

I will present a summary of the ESRF programme with some particular emphasis to the recent construction and commissioning of the EBS storage ring [1], and on its impact to new opportunities in X-ray science and applications.

#### References

1. Raimondi, P., Benabderrahmane, C., Berkvens, P., Sette, F. *et al.*, The Extremely Brilliant Source storage ring of the European Synchrotron Radiation Facility, Commun. Phys. 6 (2023) 82. https://doi.org/10.1038/s42005-023-01195-z



#### Advancements in Biological Sciences at the European Synchrotron

M. Soler López\*

MO I2

European Synchrotron Radiation Facility, CS40220, F-38043 Grenoble Cedex, France

#### \*e-mail: solerlop@esrf.fr

Biology's inherent complexity continues to challenge our understanding of life's fundamental processes. Achieving a holistic view requires integrating discoveries at the molecular, cellular, and organ/systems levels to elucidate the intricate interplay between structure and function. The ESRF leads the way in developing and applying unique biological technologies within an internationally collaborative, visitor-oriented environment, creating an ideal setting for cutting-edge research.

Among its diverse research portfolio, the ESRF dedicates significant efforts to unravelling the pathogenesis of Alzheimer's disease, a debilitating neurodegenerative condition. By probing bioenergetic mechanisms within neuronal circuits of the brain, we aim to uncover potential root causes of the disease [1]. Through a suite of advanced beamlines dedicated to structural biology [2,3], high-resolution X-ray imaging [4], and spectroscopy [5], the ESRF enables comprehensive, detailed investigations into biological processes across spatial and temporal scales.

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

- 1. McGregor & Soler-Lopez, Curr. Opin. Struct. Biol. 80 (2023) 102573.
- 2. Giachin et al., Angew. Chem. Int. Ed. Engl. 60(9) (2021) 4689-4697s.
- 3. McGregor et al., Nat. Commun. 14(1) (2023) 8248.
- 4. Walsh et al., Nat. Methods 18(12) (2021) 1532-1541.
- 5. Chourrout et al., Acta Biomater. 170 (2023) 260-272.



### Shedding ESRF-EBS light on artworks to get insight into their creation and to better preserve them

**MO I3** 

M. Cotte<sup>1,2</sup>, C. Holé<sup>1</sup>, G. Robertson<sup>1</sup>, S. Delcamp<sup>1</sup>, M. Bodin<sup>1</sup>, V. Gonzalez<sup>3</sup>, F. Vanmeert<sup>4</sup>, L. Monico<sup>5,6,7</sup>, I. Fazlic<sup>1,4</sup>, C. Dejoie<sup>1</sup>, M. Burghammer<sup>1</sup>, W. de Nolf<sup>1</sup>, L. Huder<sup>1</sup>, E. Gutierez- Fernandez<sup>2</sup>, S. Fisher<sup>1</sup>, H. Castillo-Michel<sup>1</sup>, M. Salomé<sup>1</sup>, V. Fernandez<sup>1</sup>, P. Tafforeau<sup>1</sup> and L. Paolasini<sup>1</sup>

<sup>1</sup>European Synchrotron Radiation Facility France <sup>2</sup>Laboratoire d'Archéologie Moléculaire et Structurale, CNRS UMR 8220, Sorbonne Universités, France <sup>3</sup>Université Paris-Saclay, ENS Paris-Saclay, CNRS, PPSM, 91190, Gif-sur-Yvette, France <sup>4</sup>*Rijksmuseum, Amsterdam, Netherlands* <sup>5</sup>CNR-SCITEC, 06123 Perugia, Italy <sup>6</sup>Centre of Excellence SMAArt, University of Perugia, 06123 Perugia, Italy <sup>7</sup>Antwerp X-ray Imaging and Spectroscopy laboratory (AXIS) Research Group, NANOLab Centre of Excellence, University of Antwerp, Belgium

\*e-mail: cotte@esrf.fr

In 2019, the European Synchrotron's core machine was completely dismantled to install a revolutionary machine producing a much brighter X-ray source. This upgrade has not only dramatically improved the properties of the X-ray beam, but also triggered several projects to improve the user experience, upstream and downstream data collection. As far as the cultural

heritage community is concerned, the results are numerous<sup>1</sup>.

For many beamlines, the increase in brightness translates into higher flux, smaller beams and faster acquisitions. This has motivated the implementation of a facilitated community access for structural analyses of historic materials (known as "BAG" access<sup>2, 3</sup>). Access is granted every six months to two X-ray powder diffraction (XRPD) beamlines (ID13 for µXRPD and ID22 for high angular resolution XRPD) and beamtime is shared by a network of more than 120 international collaborators (Fig. 1). Such analyses have led to major discoveries such as the identification of a very unusual lead carbonate (plumbonacrite) in Mona Lisa's and in the Last Supper's ground layers, giving insights into Leonardo da Vinci's painting techniques<sup>4</sup>.

**ID22** : High-angular resolution XRD



Figure 1. The two X-ray diffraction beamlines accessible through the "historical materials BAG" access: ID22 and ID13

The throughput of these beamlines (>200 samples analysed per experiment) is so high that this has motivated the creation of a dedicated database to make the data collected more FAIR and facilitate its re-use by anyone (project "SHARE", funded by the OSCARS European Call)<sup>5</sup> (Fig. 2).





Figure 2. The many objectives of the OSCARS SHARE project:

In parallel, in the field of X-ray micro-spectroscopy, the ID21 beamline has just been refurbished and its new nanoscope provides unprecedented performance for 2D nano-XRF mapping and nano-XANES in the 2.1-10 keV energy range (Fig. 3). These two techniques can now be efficiently combined for hyper-spectral XRF mapping, to identify and locate species at the nanoscale, over millimetric regions. These assets are very important to tackle subtle chemical modifications related to artefact manufacturing (e.g. ceramics firing) or alteration (e.g. pigment degradations). Efforts are also being made to provide users with easy-to-use graphical interfaces for data acquisition and to automate data processing and analysis.



Figure 3. The scanning X-ray microscope and nanoscope at ID21: X- ray absorption spectroscopy at the sub- micrometric scale

Regarding X-ray computed tomography, a new flagship beamline, BM18, has been built and optimised for multiresolution phase-contrast imaging of large objects. Thanks to a wide beam of up to 30 cm horizontally and an energy of up to about 300 keV in filtered white beam, several large fossils and a dozen of music instruments have recently been successfully imaged. In summary, the last five years have been instrumental in revolutionizing the user experience at the ESRF and, as recent examples will show, the benefits to the cultural heritage community are enormous.

**Acknowledgements:** The SHARE database is funded through the OSCARS project, which has received funding from the European Commission's Horizon Europe Research and Innovation programme under grant agreement No. 101129751

- 1. M. Cotte, Synchrotron Radiation News, 35 (2022) 1-7.
- 2. M. Cotte, Molecules, 27 (2022) 27(6) 1997.
- 3. https://www.esrf.fr/BAG/HistMat.
- 4. V. Gonzalez, Journal of the American Chemical Society, 145 (2023) 23205–23213.
- 5. https://oscars-project.eu/projects/share-synchrotron-x-ray-analysis-heritage-accessible-and-reusable- everyone.



## Advances in high-resolution powder diffraction for applications in materials chemistry

C. Dejoie<sup>1\*</sup>, A. Fitch<sup>1</sup>

MO\_I4

<sup>1</sup>European Synchrotron Radiation Facility, B.P.220, F-38043 Grenoble Cedex, France

\*e-mail: catherine.dejoie@esrf.fr

High-angular resolution powder diffraction, with well-defined and resolved diffraction peaks, is used generally to obtain high-quality data for complex structure refinements. The ID22 beamline at ESRF has very high angular resolution, arising from the combination of the highly collimated beam from the new EBS ring, wavelength selection via a cryogenically-cooled Si 111 monochromator, and use of a 13-crystal Si 111 multianalyser stage between the sample and a Dectris Eiger2 X 2M-W CdTe pixel detector [1]. Such an arrangement, combining multi-analyzer stage and 2D detector, is the first of its type, allowing the low-angle asymmetry in the peak shape to be removed, angular resolution to be gained, and statistical quality of the high-angle data to be improved. Higher quality data can be collected faster, allowing complex in situ and operando experiments to be performed with applications in diverse areas of Materials Science. One of the applications is in relation with the current environmental challenges, to selectively trap green-house gases such as carbon dioxide (CO2) and methane (CH4) from flue gases (mix CO2, H2O, N2) and biogas (mix CO2, CH4, H2O) using microporous materials. For nonmixed gases, in situ adsorption experiments as a function of the gas pressure are carried out, and adsorption isotherms are retrieved after Rietveld refinement against the highresolution powder diffraction data. Knowing the location of the gas molecules in the material allows a description of the adsorption process not only at the macroscopic scale, but also at the microscopic one, as each individual site is modelled with a site-specific isotherm with specific thermodynamic parameters. This deeper knowledge of the adsorption process opens the way to new strategies to optimize the selective removal of green-house gases from a competitive environment, as found in flue gases or biogas.

- 1. Fitch, A., Dejoie, C., Covacci, E., Confalonieri, G., Grendal, O., Claustre, L., Guillou, P., Kieffer, J., de Nolf, W., Petitdemange, S., Ruat, M., Watier, Y., J. Synchrotron. Rad. 30 (2023) 1003.
- 2. Lill, J., Dejoie, C., Giacobbe, C., Fitch, A.N., J. Phys. Chem. C 126 (2022) 2214-2225.



### Effect of cationic substitution on structural properties of Ca<sub>3</sub>(VO<sub>4</sub>)<sub>2</sub> orthovanadates MO O1

H.S. Rahimi Mosafer <sup>1\*</sup>, R. Minikayev<sup>1</sup>, A. Wolska<sup>1</sup>, M. Klepka<sup>1</sup>, M Berkowski<sup>1</sup>, C. Martin<sup>2</sup>, A. Fitch<sup>3</sup>, A. Maximenko<sup>4</sup> and W. Paszkowicz<sup>1</sup>

<sup>1</sup>Institute of Physics, Polish Academy of Sciences, Warsaw, Poland <sup>2</sup>Laboratoire CRISMAT, Normandie Université, ENSICAEN, UNICAEN, CNRS, 14050 Caen, France <sup>3</sup>European Synchrotron Radiation Facility, Grenoble 38000 <sup>4</sup>SOLARIS National Synchrotron Radiation Centre, 30392, Krakow, Poland

\*e-mail: rahimi@ifpan.edu.pl

Orthovanadates have attracted significant research attention due to their potential applications across various fields, including optoelectronics, biomedicine, and catalysis. Calcium orthovanadate  $(Ca_3(VO_4)_2)$  is known to crystallize in R3c space group<sup>1</sup>. The structure of natural and synthetic compounds of this family is complex and constructed in a way allowing for various substitutions, with two columnar building units. As the structure supposes five cationic sites (M1-M5) where various substituents can be located at various site, at low or high concentration (up to 10% in specific cases), the determination of structure requires refinement of the occupancies at all these sites.

In this presentation, we explore the structural properties of calcium orthovanadates, specifically  $Ca_{10.5-x}TM_x(VO_4)_7$  (x=0-1), where some of the calcium ions have been partially replaced by transition metal ions. We investigate these properties at both, room temperature and non-ambient conditions, through X-ray diffraction measurements<sup>2-4</sup>. High-resolution X- ray powder diffraction experiments were conducted at the ID22 beamline at ESRF. The solubility limit was determined for three sets of samples. The EXAFS results align with the XRD findings regarding the presence of a transition metal at the M5 site. The study of diffraction patterns at non-ambient temperatures indicates that the structure remains unchanged with temperature varying from 4 K to 1100 K, as confirmed by Rietveld refinements. In this work, we determine the unit cell size and atomic positions of  $Ca_{10.5-x}TM_x(VO_4)_7$  as functions of temperature. Additionally, we calculate the thermal expansion coefficient for these materials in relation to temperature using various models

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- 1. Gopal, R., and C. Calvo. Zeitschrift für Kristallographie-Crystalline Materials 137 (1973) 67-85.
- 2. H. S. R. Mosafer et al., Dalton Transactions 50 (2021) 14762.
- 3. J. Sánchez-Martín et al., Cryst. Eng. Comm. 25 (2023) 1240.
- 4. H. S. R. Mosafer et al., Crystals 13 (2023) 853.



## Unraveling the mechanism of iron sulfide nanostructures formation: insights from *in situ* X-ray diffraction and spectroscopy studies MO O2

C.A. Zito<sup>1,2</sup>\*, L. Klemeyer<sup>1</sup>, F. Caddeo<sup>1</sup>, B. Jessen<sup>1</sup>, M. Langfeldt<sup>1</sup>, S.Y. Harouna-Mayer<sup>1,2</sup>, A-C. Dippel<sup>3</sup> and D. Koziej<sup>1,2</sup>

<sup>1</sup>University of Hamburg, Institute for Nanostructure and Solid-State Physics, Center for Hybrid Nanostructures, 22761 Hamburg, Germany <sup>2</sup>The Hamburg Center for Ultrafast Imaging, 22761 Hamburg, Germany <sup>3</sup>Deutsches Elektronen-Synchrotron DESY, 22607 Hamburg, Germany

\*e-mail: cecilia.zito@uni-hamburg.de

Iron sulfides ( $Fe_xS_y$ ) have emerged as promising materials for several applications. In particular,  $Fe_3S_4$  greigite has attracted significant attention due to its magnetic properties and potential use in Li-ion and Na-ion batteries<sup>1</sup>. However, controlling the phase during the synthesis of the Fe<sub>x</sub>Sy nanostructures remains challenging due to the system's complexity, with seven major crystal phases exhibiting different oxidation states and stoichiometries<sup>2</sup>. Understanding the chemical processes occurring during synthesis is crucial for obtaining a high-purity phase, which directly impacts the performance of the  $Fe_xS_y$  nanomaterials in the intended applications.

Here, we illustrate how combining *in situ* X-ray diffraction and spectroscopy studies offers insights into the formation mechanism of  $Fe_3S_4$  nanosheets in solution under solvothermal conditions. Initially, *in situ* X-ray diffraction (XRD) uncovers the lack of long-range ordering in the initial stages of the reaction, followed by the initial crystallization of tetrahedral FeS phase (mackinawite) with preferred orientation along (001) plane, forming a layered structure. This phase gradually transforms into crystalline spinel  $Fe_3S_4$  (greigite).

Using *in situ* high-energy resolution fluorescence-detected X-ray absorption spectroscopy (HERFD-XAS) measurements, we identify four key components in the reaction: the iron(III) acetylacetonate precursor as the starting point, two distinct intermediates, and the final product, Fe<sub>3</sub>S<sub>4</sub>. The combination of HERFD-XAS and density functional theory (DFT) calculations suggests an initial reduction of the organo-metallic iron precursor with the simultaneous coordination of two benzyl alcohol solvent molecules, forming the first reaction intermediate. This oxygen-coordinated molecular intermediate then transforms into a tetrahedrally sulfur-coordinated compound that we identify as FeS through FEFF calculations, corresponding to the second intermediate in the reaction. Additionally, we employ an innovative approach using *in situ* valence-to-core X-ray emission spectroscopy (vtc-XES) at elevated temperatures in solution<sup>3</sup>, pinpointing the transition from the Fe-O to Fe-S coordination as the reaction progresses.

With the versatile combination of X-ray techniques, we obtain mechanistic insights into all steps of the complex formation of  $Fe_3S_4$  nanosheets under solvothermal conditions.

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- 1. G. Li, B. Zhang, F. Yu et al. Chem. Mater. 26(20) (2014) 5821-5829.
- 2. J. R. Bairan Espano, J. E. Macdonald. J. Am. Chem. Soc. 145(34) (2023) 18948-18955.
- 3. L. Klemeyer, T. L. R. Gröne, C. A Zito et al. J. Am. Chem. Soc. 146(49) (2024) 33475-33484.



## Operando XAS for chemistry at BM23 and ID24-DCM beamlines of the ESRF

MO\_I5

K.A. Lomachenko\*, D. Salusso, A. Molokova, O. Mathon

European Synchrotron Radiation Facility, 38043 Grenoble, France \*e-mail: lomachenko@esrf.fr

BM23 and ID24 are ESRF beamlines that specialize primarily in X-ray absorption spectroscopy, but also offer an impressive set of complementary characterization techniques. Main fields of in-house research and user activity are operando chemistry (catalysis, electrochemistry, etc), materials science, environmental science, physics and geoscience. Both beamlines have been deeply refurbished within the EBS upgrade program.

BM23 is multi-purpose XAS beamline optimized for high-quality XAS measurements between 4.5 and 90 keV (for mm beam) and 4.5-45 keV (for  $\mu$ m beam), in transmission or fluorescence mode. Its brand-new monochromator developed in the ESRF allows collecting XAS spectra in continuous mode in a few seconds with unprecedented energy and beam position stability. BM23 offers a large variety of sample environment: in situ cells, cryostats, ovens, gas infrastructure, high-pressure equipment, mass-spectrometer for gas analysis and more. Pilatus 1M detector is available for complementary XRD measurements. Simultaneous XAS and DRIFTS measurements under controlled temperature and gas flow are possible thanks to the dedicated DRIFTS/XAS/MS setup. The microXAS station for focusing the beam below 5x5  $\mu$ m<sup>2</sup> FWHM with 10<sup>9</sup> ph/s is also available for space-resolved studies.

ID24 beamline consists of two branches with very distinct instrumentation and applications. Energy dispersive branch (ID24-ED) is coupled with high-power laser facility (HPLF) and is dedicated mainly to laser shock experiments in physics and planetary science. Energy dispersive scheme allows ultra-fast measurements (down to 100 ps/spectrum), ideal for time- resolved studies.

Scanning branch of ID24 (ID24-DCM) is a high brilliance XAS beamline equipped with the same novel double-crystal monochromator as BM23. The accessible X-ray energy ranges from 5 to 45 keV. Taking advantage of the new EBS characteristics, ID24-DCM combines high flux up to 10<sup>13</sup> ph/s from undulator source with variable X-ray beam size from 1x1 mm<sup>2</sup> down to 500x500 nm<sup>2</sup> FWHM. The new DCM allows collecting XAS spectra in a few seconds even in fluorescence mode. A Pilatus 2M detector is permanently installed on the beamline for complementary XRD measurements. High energy resolution fluorescence detected XAS is available thanks to 5- crystal spectrometer. MicroXAS station allows focusing the beam down to 500x500 nm<sup>2</sup> FWHM if needed. ID24-DCM is compatible with all chemistry sample environment of BM23, including cells, gases and DRIFTS/XAS/MS setup.

The talk will be devoted to the demonstration of the performance of BM23 and ID24-DCM beamlines and their application to chemistry studies, including catalysis, synthesis of novel materials and electrochemistry. Recent highlights from both user-driven and inhouse research will be presented.



### Polymorphisms and early-stage crystallization of Zr-based glasses induced by high pressure and temperature

MO\_03

L. Rogal<sup>1\*</sup>, D. Laniel<sup>2</sup>, G. Weck<sup>3</sup>, G. Garbarino<sup>4</sup>, Z. Kobylarz<sup>1</sup>, U.A. Wdowik<sup>5</sup> and J. Dutkiewicz<sup>1</sup>

<sup>1</sup>Institute of Metallurgy and Materials Science Polish Academy of Science, 30-059 Krakow, Poland <sup>2</sup>Centre for Science at Extreme Conditions, School of Physics and Astronomy, University of Edinburgh, King's Buildings, EH9 3FD Edinburgh, United Kingdom <sup>3</sup>DIF/DPTA, CEA 91680 Bruyères-le-Châtel, France <sup>4</sup>European Synchrotron Radiation Facility (ESRF), 38000 Grenoble, France <sup>5</sup>IT4Innovations, VSB – Technical University of Ostrava, 708 00 Ostrava- Poruba, Czech Republic

\*e-mail: l.rogal@imim.pl

 $Cu_{43}Zr_{43}Ag_7Al_7$  (at. %) metallic glasses were annealed above the glass transition temperature (497 °C) under 7.8–73 GPa. Two high-pressure systems were employed: (i) a Bridgman toroidal-type apparatus with resistive heating and (ii) a Diamond Anvil Cell (DAC) equipped with double-sided YAG laser heating.

Distribution In situ Pair Function (PDF) measurements at the European Synchrotron Radiation Facility (ESRF) reveal a decrease in atomic distances associated with the densification of the metallic glasses, indicating polymorphic transitions and subsequent early-stage crystallization induced by high pressure and temperature. Post-annealing high-resolution transmission electron microscopy (HRTEM) analyses, performed at atomic resolution, distinguish multiple glassy states. Inverse and Fast Fourier Transform (FFT and IFFT) of selected HRTEM micrographs demonstrate a progressive reduction in nearest-neighbor spacing with increasing pressure and the development of ordered microdomains within the amorphous matrix at higher temperatures (Fig. 1). These findings imply a precedes structural reorganization that crystallization reorganization preceding the onset of crystallization.



Fig. 1 Representative HRTEM image and corresponding Fast Fourier Transform (FFT) showing a diffuse ring characteristic of an amorphous structure with incipient ordering.

Additionally, a notable decrease in the effective glass transition temperature was observed at higher sintering temperatures, implying a correlation between densification mechanisms and the stability of the amorphous phase. The combined results provide new insights into pressure- driven polymorphic transformations and the fundamentals of crystallization kinetics in Zr- based metallic glasses under extreme conditions.

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#### Insight into the structure of liquid-crystalline itraconazole drug

K. Jurkiewicz1\*, T. Lamrani1 and A. Aminian1

**MO\_04** 

<sup>1</sup>Institute of Physics, University of Silesia in Katowice, 41-500 Chorzów, Poland

\*e-mail: karolina.jurkiewicz@us.edu.pl

Nowadays, there is an increasing interest in the application of amorphous pharmaceutical formulations in tablet form since they exhibit better solubility and bioavailability than their crystalline counterparts. One of the very poorly soluble active pharmaceutical ingredients (APIs) is itraconazole (ITZ), which demonstrates superior antifungal activity. Moreover, ITZ is a well-known thermotropic liquid-crystalline (LC) compound. When cooled down from melt, ITZ exhibits transformation to the nematic and then to the smectic phase, which subsequently is trapped in the glass state at room temperature. Recently, we demonstrated that by cryo- milling it is possible to produce very stable solid ITZ particles with a lower degree of LC organization (nematic) compared to ordinary glass obtained by vitrification<sup>1</sup>. Importantly, the more disordered form resulted in better solubility in the conditions simulating gastrointestinal passage<sup>1</sup>.



Figure 1. X-ray scattering (diffraction) data of the vitrified and cryo-milled ITZ collected using synchrotron radiation (ID22, ESRF).

In order to deepen the knowledge of the subtle differences in the local non-crystalline, structure of amorphous-like APIs, total X-ray scattering data collected using synchrotron sources may be used. Here, we will present the results of different X-ray scattering experiments and molecular dynamics simulations, which in combination allowed us to create coherent models of the atomic- scale structure of ITZ obtained using various methods: vitrification, cryo- milling, and solvation-evaporation.

The analysis of the obtained intermolecular structure factors and configurations changes the conventional thinking about the LC organization of molecules, which, in the case of the ITZ smectic glass,

is difficult to distinguish from completely amorphous at first sight. Moreover, based on the analysis of the intramolecular structure factors, one may ask whether temperatureactivated transitions between different diastereomers are possible.

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#### References

1. T. Lamrani, et al. J. Mol, Liq. 414 (2024) 126106.



## Advanced Software Tools and Workflows for Synchrotron X-Ray Scattering Analysis at ESRF: A User's Perspective MO\_O5

M. Jankowski<sup>1\*</sup>

<sup>1</sup>The European Synchrotron Radiation Facility – ESRF, Grenoble , 38043 France

\*e-mail: maciej.jankowski@esrf.fr

The recent launch of the Extremely Brilliant Source (EBS)<sup>1</sup> at the ESRF has been accompanied by numerous advancements in software development, introducing sophisticated tools and workflows designed to provide the user community with the capability to generate high-quality scientific data. Understanding these tools is essential for efficient data analysis in synchrotron experiments.

Key collaborative software technologies employed at ESRF are introduced, including Extensible Workflow System (EWOKS)<sup>2</sup> for automated, reproducible, and traceable data processing integrated with Beam Line Instrumentation Support Software (BLISS)<sup>3</sup> for beamline control, Scientific Library for eXperimentalists (SILX)<sup>4</sup> as a Python package for data science and analysis, and specialized tools for reducing X-ray diffraction data from 2D detectors<sup>5,6,7</sup>.

The digital ecosystem at ESRF is also introduced, highlighting the computing cluster for data analysis, the Virtual Infrastructure for Scientific Analysis (VISA) platform, and the web-based environment for interactive Jupyter notebooks. These tools and platforms are vital in accelerating scientific discoveries and enhancing synchrotron radiation research.

- 1. Raimondi, P., Benabderrahmane, C., Berkvens, P., et al., The Extremely Brilliant Source storage ring of the European Synchrotron Radiation Facility, Commun Phys, 6 (2023) 82.
- De Nolf, W., Huder, L., Svensson, O., et al., EWOKS: An Extensible WOrKflow System, Synchrotron Radiation News 37(6) (2024) 9–15.
- 3. Guijarro, M., Felix, L., De Nolf, W., et al., A Marriage Made in BLISS—An Integrated Beamline Control System, Synchrotron Radiation News 36(6) (2023) 12–19.
- 4. Vincent, T., Valls, V., Payno, J., et al. (2024). silx-kit/silx: 2.1.0: 2024/04/19 (v2.1.0). Zenodo.
- 5. Kieffer, J., Brennich, M., Florial, J. B., et al., New data analysis for BioSAXS at the ESRF, Synchrotron Radiation 29(5) (2022) 1318–1328.
- 6. Roobol, S., Onderwaater, W., Drnec, J., et al., BINoculars: data reduction and analysis software for twodimensional detectors in surface X-ray diffraction, Applied Crystallography 48(4) (2015) 1324–1329.
- 7. Kieffer, J., Karkoulis, D. (2013, March). PyFAI, a versatile library for azimuthal regrouping. In Journal of Physics: Conference Series (Vol. 425, No. 20, p. 202012). IOP Publishing



### An introduction to X-ray Science and hard X-ray Free Electron Lasers

T. Feurer<sup>1\*</sup>

TU\_I1

<sup>1</sup>European X-Ray Free-Electron Laser Facility GmbH, 22869 Schenefeld, Germany

\*e-mail: thomas.feurer@xfel.eu

Hard X-ray Free Electron (XFEL) lasers produce extremely intense and ultra-short X-ray pulses, perfect for investigating the structure and dynamics of matter at atomic length and time scales. Operating for over a decade, these lasers have demonstrated a wide range of applications in physics, chemistry, materials science, and structural biology.

The European XFEL, one of the latest large-scale research infrastructures in Europe, recently celebrated five years of successful user operations. The facility features a 3.5 km long tunnel, including a 2 km long superconducting accelerator, stretching from DESY in Hamburg to Schenefeld in Schleswig-Holstein, where the experimental hall houses seven instruments. These instruments offer a broad range of experimental capabilities.

Since beginning operations, numerous exciting user experiments have been conducted in fields such as physics, chemistry, bio-crystallography, and material science. In my talk, I will present the main principles of the science performed at the European XFEL, with a focus on high energy density science and attosecond science. I will also provide examples from recent experiments in various scientific areas.



Figure 1. A glimpse of the 2 km long superconducting accelerator.



### Probing dynamics in biology, chemistry and physics using ultrafast X-rays

TU\_I2

Chris J. Milne

European XFEL GmbH, Schenefeld, Germany 22869

\*e-mail: christopher.milne@xfel.eu

X-ray spectroscopy and scattering allow a unique combination of electronic and structural information to be obtained from a variety of different types of samples in many different forms (solid, liquid, gas). The extension of these methods into the time domain has allowed measurement of dynamic processes, for example the tracking the photoinduced charge carriers in a functional material [1] or following the photocycle in a light activated protein [2]. In recent years X-rays have started to become routinely used to measure light-activated processes using a pump-probe scheme, where the sample is photoexcited with light and then probed after a variable time delay using an X-ray pulse. These methods can measure dynamics over a broad range of timescales, allowing them to probe everything from protein dynamics to ultrafast electronic spin-state changes in molecular systems. With the recent development of X-ray free electron lasers (XFELs), time-resolved X-ray techniques have moved into the ultrafast regime, where the timescales of electron and nuclear motion can be accessed using the femtosecond X-ray pulses available from these facilities.



Fig. 1. Examples of the science and X-ray techniques performed at the FXE Instrument of the European XFEL

This talk will present an overview of how X-ray techniques are being used at XFELs to address topics in the natural sciences and the type of information the measurements can provide. The presentation will introduce the time-resolved capabilities of the European XFEL, a unique high-repetition rate XFEL facility located in northern Germany. Examples will be shown of the types of measurements XFELs can perform and the scientific questions that can be answered using ultrafast X-ray techniques, with a specific focus on research performed at the Femtosecond X-ray Experiments (FXE) instrument[3].

- 1. Penfold, T. J. et al. (2018), Nat. Comm. 9, 478. Rittmann-Frank, M. H. et al. (2014). Angew. Chem. Int. Ed. 53 5858–5862.
- 2. Bacellar, C. et al. (2020). P Natl Acad Sci Usa 117, 21914–21920. Kinschel, D. et al. (2020). Nat. Comm. 11 4145.
- 3. Khakhulin, D. et al. (2020). Appl. Sci. 10, 995. Galler, A. et al. (2019). Journal of Synchrotron Radiation 26 1–16.



### Ultrafast dynamics of photoexcited states in cerium oxide investigated by pump-probe XAS at the EuXFEL TU

TU I3

S. Pelatti<sup>1,2</sup>, E. Spurio<sup>1,2</sup>, J.S. Pelli Cresi<sup>3</sup>, S. Benedetti<sup>2</sup>, A. di Bona<sup>2</sup>, S. D'Addato<sup>1,2</sup>, P. Zalden<sup>4</sup>, Y. Jiang<sup>4</sup>, Y. Uemura<sup>4</sup>, H. Wang<sup>4</sup>, D. Vinci<sup>4</sup>, X. Huang<sup>4</sup>, F. Lima<sup>4</sup>, M. Biednov<sup>4</sup>, D. Khakhulin<sup>4</sup>, Chris Milne<sup>4</sup>, S. Turchini<sup>5</sup>, G. Ammirati<sup>5</sup>, P. O'Keeffe<sup>6</sup>, D. Catone<sup>5</sup>, F. Boscherini<sup>7</sup> and P. Luches<sup>2\*</sup>

<sup>1</sup>Dipartimento FIM, Università degli Studi di Modena e Reggio Emilia, 41125 Modena, Italy <sup>2</sup>Istituto Nanoscienze, CNR (NANO-CNR),41125 Modena, Italy <sup>3</sup>Elettra-Sincrotrone Trieste, 34012 Basovizza, Trieste, Italy <sup>4</sup>European XFEL, Holzkoppel 4, 22869 Schenefeld, Germany <sup>5</sup>Istituto di Struttura della Materia – CNR (ISM-CNR), EFSL, Rome, Italy <sup>6</sup>Istituto di Struttura della Materia – CNR (ISM-CNR), EFSL, Monterotondo Scalo, Italy <sup>7</sup>Dipartimento di Fisica e Astronomia, Alma Mater Studiorum – Università di Bologna, Bologna, Italy

\*e-mail: paola.luches@nano.cnr.it

Pump-probe spectroscopies utilizing X-ray free-electron lasers (XFELs) offer elementspecific insights into the processes occurring in photocatalysts following photoexcitation, which are essential for the rational optimization of these materials' efficiency. At the FXE instrument of the European XFEL facility<sup>1</sup> we investigated the dynamic evolution of photoexcited sates in cerium oxide, a material widely used as a catalyst or photocatalyst.

The sample was a stoichiometric  $CeO_2$  film grown by reactive magnetron sputtering on a sapphire substrate. The measurements were performed at grazing incidence, to minimize sample damage and maximize the sensitivity of the method to the thin film investigated. The sample was excited using a laser pump pulse with energy above the band gap and probed by XFEL pulses. We employed pump-probe X-ray absorption spectroscopy (XAS) at the Ce L<sub>3</sub> edge in both the near-edge and extended energy ranges to simultaneously probe the ultrafast electronic and local atomic structure modifications following photoexcitation. The data were acquired in the total fluorescence yield within a delay time range up to 300 ps with approximately 50 fs resolution.

The results revealed a rapid de-excitation pathway occurring within the first few hundred femtoseconds, followed by the formation of a metastable excited state with a lifetime on the order of hundreds of picoseconds. The analysis of pump-probe XAS in the extended energy range identified a structural distortion consistent with the formation of a photoinduced small polaron state, that was hypothesized by a previous study done by optical pump-probe transient absorption spectroscopy.<sup>2</sup> The observed time correlation between the photoinduced electronic and structural changes further reinforces the hypothesis of photoinduced polaron formation.

Such transient charge localization is expected to have a positive impact on cerium oxidebased photocatalysts by weakening the bond between the cation and oxygen, thereby reducing the energy required for oxygen vacancy formation, which is crucial for the material's functionality. At the same time, it can extend the lifetime of the photoexcited carriers.

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- 1. A. Galler, W. Gawelda, M. Biednov, C. Bomer, A. Britz, S. Brockhauser, T.-K. Choi, M. Diez, P. Frankenberger, M. French, et al., J. Synchrotron Radiat. 26 (2019) 1432.
- J.S. Pelli Cresi, L. Di Mario, D. Catone, F. Martelli, A. Paladini, S. Turchini, S. D'Addato, P. Luches, P. O'Keeffe, J. Phys. Chem. Lett. 11 (2020) 5686.



## Angle-resolved photoemission for the investigation of topological materials

Oliver Rader\*

TU\_I4

Helmholtz-Zentrum Berlin für Materien und Energie, 12489 Berlin, Germany \*e-mail: rader@helmholtz-berlin.de

Angle-resolved photoelectron spectroscopy (ARPES) is the premier technique for probing the electronic band structure of solids<sup>1</sup>. In this talk, we introduce ARPES through examples from topological materials, a class of systems that have attracted considerable interest in condensed matter physics due to their unconventional electronic and transport properties.

Three-dimensional (3D) topological materials host symmetry-protected electronic states with distinctive spin textures, making them promising candidates for future applications in spintronics and topological quantum computing.

We will discuss key aspects of ARPES, beginning with the fundamental three-step model, where conservation laws and the probing depth are important aspects. We will address the dipole approximation and selection rules, followed by the measurement of 3D band structures and Fermi surfaces. The role of surface states and resonances will be explored as well as many-body interactions and lifetime broadening. We will discuss spin polarization effects occurring in magnetic and nonmagnetic topological insulators.

Finally, we review ARPES investigations of various topological phases<sup>2</sup>, including strong and crystalline topological insulators, magnetic topological insulators, and 3D Dirac, Weyl, nodal, and chiral semimetals, while also addressing the current status of correlated topological insulators.

- 1. Hongyun Zhang, T. Pincelli, C. Jozwiak, T. Kondo, R. Ernstorfer, T. Sato, Shuyun Zhou, Angle-resolved photoemission spectroscopy, Nature Reviews Methods Primers 2 (2022) 52, DOI: 10.1038/s43586-022-00133-7; arXiv:2207.06942
- J. Sánchez-Barriga, O. J. Clark, O. Rader, Angle-resolved photoemission of topological materials. In T. Chakraborty (Ed.), Encyclopedia of Condensed Matter Physics, 2nd ed., Vol. 4, pp. 334-369. Elsevier, Amsterdam (2024), DOI: 10.1016/B978-0-323-90800-9.00274-2; arXiv:2501.00497 (2024).



#### Ambient pressure XPS at MAX IV Laboratory and how it is used for atomic layer deposition research

Esko Kokkonen1\*

TU\_I5

<sup>1</sup>MAX IV Laboratory, Lund University, SE-221 00 Lund, Sweden \*e-mail: esko.kokkonen@maxiv.lu.se

The discovery of novel materials and new chemical processes requires the detailed understanding of their constituent sub-processes. *In situ* techniques are invaluable in such fields of science and engineering, since they enable investigating the materials under realistic conditions which are close to those the materials are subjected to in their intended operational areas. Ambient Pressure X-ray Photoelectron Spectroscopy (APXPS) is one such *in situ* tool allowing one to obtain detailed elementally specific, chemically relevant and surface sensitive information from a wide range process and samples serving different scientific disciplines. APXPS is, in its simplicity, merely a way of conducting XPS measurements under higher pressure conditions. Typical tools, such as the ones presented here, allow measurements at pressure of tens of mbar and temperatures of several hundred degrees Celsius.

Typical XPS investigations are often limited to pressures in the ultra-high vacuum regime. Such pressures are far from the values that are found in real industrial chemical and materials manufacturing processes. Therefore, it is if utmost interest to study the materials in the conditions that are relevant for their manufacture in order to find routes of optimisation, energy efficiency, and other improvements.

One such area of research is atomic layer deposition (ALD), which is nowadays the most common tool used to create nanosized devices used in many different areas of nanoelectronics research. ALD relies on the cyclic exposure of the substrate surface to different precursor molecules. These precursor molecules will react with the surface and ideally create individual atomic layers. The desired layer thickness is realized by continuing to cycle the substrates over the surface until enough material has been deposited. Due to the self-limiting nature of the half- cycles, ALD creates uniform and chemically pure surfaces, in the ideal case. However, in many processes, it is not accurately known how the chemical reactions follow one another, especially in the details of the reactions occurring in the sub-cycle timescale.

In this manner, APXPS and ALD can together form a powerful combination that can provide information which has so far been inaccessible. Due to the pressure range of typical ALD processes being in the range that can be used with APXPS, one can truly use the power of XPS to obtain information from the surface layers as they are being deposited. Such a system has been built at the SPECIES beamline at the MAX IV Laboratory [1]. The setup has been used to investigate several different ALD processes to find out details on the ALD of TiO<sub>2</sub>, HfO<sub>2</sub>, platinum, and others, on many different substrates [2-5]. Information gained so far includes details about oxygen migration, roles of pressure and temperature, and the details of the individual half-cycles.

- 1. E. Kokkonen, *et al.*, Rev. Sci. Instrum. 93 (2022) 013905.
- 2. G. D'Acunto et al., ACS Appl. Electron. Mater. 2 (2020) 3915.
- 3. G. D'Acunto, et al., Surf. Interfaces 39 (2023) 102927.
- 4. R. Jones, et al., Surf. Science 753 (2025) 122656.
- 5. E. Kokkonen, *et al.*, J. Vac. Sci. Tech. A 42 (2024) 062406.



#### Scanning Photoelectron Microscopy (SPEM) of N-doped ZnO films: impact of carbon on the electronic band structure at the nanometer scale

E. Guziewicz<sup>1\*</sup>, M. Amati<sup>2</sup>, L. Gregoratti<sup>2</sup> and O. Volnianska<sup>1</sup>

<sup>1</sup>Institute of Physics Polish Academy of Sciences, PL 02 668, Warsaw, Poland <sup>2</sup>Elettra-Sincrotrone Trieste S. C. p. A., 34149 Trieste, Italy

\*e-mail: guzel@ifpan.edu.pl

The *p*-type conductivity of ZnO has been the subject of extensive research for over 20 years. Although the problem has not yet been solved, systematic studies combining modern research techniques supported by advanced theoretical calculations and tailored growth technology provide interesting new results. Recently, it was found that the macroscopically measured *p*-type conductivity of ZnO may have a complicated microscopic origin, as shown by recent low temperature cathodoluminesence (LTCL) and scanning photoelectron spectroscopy (SPEM) studies that revealed separated donor and acceptor regions in ZnO:N.<sup>1-2</sup> A Scanning Photoelectron Microscopy (SPEM) experiment reported here was performed on the ZnO:N film deposited by Atomic Layer Deposition (ALD) under O-rich conditions, both *as grown* and post-growth annealed in oxygen at 800°C.



Figure 1. (left) SPEM image of the cross-section of the *as grown* ZnO:N film showing the points where PES spectra were taken; (right) PES spectra of the Zn3d and the valence band region taken at points O, P, Q and R.

Photoelectron spectra of *as grown* and annealed ZnO:N/Si films taken with *State-of-the-Art* resolution of 130 nm show significantly different intensities of the valence maximum close to the VB edge depending on the measured point of the film cross-section as well as a shift of some of the spectra towards the bandgap, pointing to hybridization with shallow acceptor states. We have found a significant correlation between a valence band shift and carbon content established from the intensity of the C1s state. The calculated migration properties show that complexes such as VZn(NH)O, that are the source of acceptor states, are easily formed in the presence of the interstitial  $C_iH_2$  group.<sup>3</sup> The presence of CH<sub>2</sub> groups in the samples leads to lowering of the migration energy by 0.8 eV and to zero in the ZnO and N:ZnO, respectively. These results confirm that the formation of defect complexes is very sensitive to the local geometry and lattice perturbations.

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- 1. E. Guziewicz, O. Volnianska, I.N. Demchenko, P. Zeller, M. Amati, L. Gregoratti, Phys. Rev. Appl. 18 (2022) 044021.
- S. Mishra, B.S. Witkowski, R. Jakiela, Z. Khosravizadeh, W. Paszkowicz, A. Sulich, O. Volnianska, W. Wozniak, E. Guziewicz, Phys. Stat. Solidi A 220 (2023) 2200466.
- 3. E. Guziewicz, S. Mishra, M. Amati, L. Gregoratti, O. Volnianska, Nanomaterials 15 (2025) 30.



#### Integrating XRF and XCT for Understanding Medieval Ceramic Production from Wawel Royal Castle

TU\_02

R. Abdelrahman<sup>1,3</sup>, S. Wroński<sup>1</sup>, B. Makowiecki<sup>2</sup>, L. Samek<sup>1\*</sup>

<sup>1</sup>AGH University of Krakow, Faculty of Physics and Applied Computer Science, 30-059 Krakow, Poland <sup>2</sup>Wawel Royal Castle – State Art Collection, Ceramic and Glass Department, PL 31-001 Kraków, Poland <sup>3</sup>Future University in Egypt (FUE), Faculty of Engineering and Technology, Cairo, Egypt.

\*e-mail: Lucyna.Samek@fis.agh.edu.pl

This study examines five tile fragments from Wawel Royal Castle using X-ray fluorescence (XRF) and X-ray computed tomography (XCT) to investigate their composition and internal structure. XRF analysis revealed similarities to Carpathian Basin ceramics in SiO<sub>2</sub>, K<sub>2</sub>O, P<sub>2</sub>O<sub>5</sub>, and MnO contribution but higher TiO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>, and CaO levels as well as lower Al<sub>2</sub>O<sub>3</sub>, MgO, and Na<sub>2</sub>O content, suggesting local production with different clay sources despite shared motifs.

XCT provided high-resolution imaging of inclusions, voids, and porosity, complementing XRF findings by revealing variations in clay refinement, tempering methods, and firing conditions. Correlating XCT structural data with XRF chemical composition highlighted craftsmanship differences from rudimentary to advanced techniques. Despite voxel resolution limitations, XCT proved valuable in identifying internal inaccessible ceramic structures.

This research contributes to the understanding of medieval ceramic production, offering valuable insights into cultural exchanges, trade networks, and regional influences in Central Europe while highlighting the role of local craftsmanship in creating historically significant artifacts. Additionally, the synergy of XCT and XRF enhances archaeological research methodologies, demonstrating the advantages of multi-modal, non-destructive material characterization for heritage conservation and material studies.

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#### Unraveling charge transfer dynamics in switchable molecular magnets through X-ray absorption spectroscopy **WE I1**

M. Fitta<sup>1\*</sup>

<sup>1</sup>Institute of Nuclear Physics Polish Academy of Sciences, 31-342 Kraków, Poland

\*e-mail: Magdalena.Fitta@ifj.edu.pl

Cyano-bridged coordination systems are widely studied in molecular magnetism due to the strong superexchange magnetic interactions facilitated by CN ligands. Their flexible cyanobridged linkages also allow structural deformations in response to external stimuli, potentially enabling switchability or memory effects. One of the interesting phenomena observed in coordination networks is the possibility of triggering metal-to-metal charge transfer (MMCT) by temperature, pressure, or sorption of small molecules. MMCT entails electron transfer between metal ions, creating valence isomers with different electronic configurations. This process alters spin states, coupling interactions, and charge distribution, significantly impacting the material's magnetic, electric, and optical properties and bond lengths.

MMCT process was investigated in 1D bimetallic chains  $\{(NH_4^+)[Ni^{III}(cyclam)][Fe^{II}(CN)_6]\cdot 5H_2O\}_{\infty}$ , where the Ni(II/III) and Fe(II/III) redox pair is influenced by water sorption/desorption, temperature, and pressure<sup>1</sup>. Using X-ray absorption spectroscopy (XAS) at Fe and Ni K-edges, we probe the reversibility and reproducibility of the intramolecular Fe/Ni redox process (Figure 1).



To further tune the MMCT characteristics, Fe was substituted with whose higher Ru, potential reduction stabilizes the Ni(III)-Ru(II) phase. Notably, this system in rehydration, though partially reversible, does not restore the

Figure 1. Normalized absorption coefficient at Fe and Ni K-edge recorded for MMCT process.  $\{(NH_4^+)[Ni^{III}(cyclam)][Fe^{II}(CN)_6]\cdot 5H_2O\}_{\infty}$  at temperatures: 330K and 260K showcasing the antagonist change of XANES characteristics between Fe and Ni K-

XAS measurements reveal that Ru remains in the Ru(II) oxidation

edges, due to the charge transfer effect). state, challenging the previous assumption of electron transfer from Ru to Ni leading to Ru(III) formation<sup>2</sup>. This study underscores the power of XAS in probing fragile electron transfer and redox-active systems at a local scale. Furthermore, it highlights the technique's complementarity with traditional structural, optical, and magnetic measurements, offering

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a more comprehensive understanding of complex redox processes.

#### References

1. M. Reczyński, D. Pinkowicz, K. Nakabayashi, C. Näther, J. Stanek, M. Kozieł, J. Kalinowska-Tłuścik, B. Sieklucka,

S. ichi Ohkoshi and B. Nowicka, Angew. Chemie - Int. Ed. 60 (2021) 2330.

2. M. Reczynski, B. Nowicka, C. Nather, M. Kozieł, K. Nakabayashi, S. Ohkoshi, B. Sieklucka, Inorg. Chem. 57 (2018) 13415.



# In situ X-Ray Synchrotron Methods for Exploring the Length Scale in Nanostructured Materials and Devices WE 12

Dorota Koziej\*

University of Hamburg, Institute for Nanostructure and Solid-State Physics, Center for Hybrid Nanostructures (CHyN), 22761 Hamburg, Germany

\*e-mail: dorota.koziej@uni -hamburg.de

In recent years, we have developed methods to fabricate materials with complex chemical and structural properties. Successfully synthesizing functional materials relies on controlling chemical reactions across various length scales, yet the classical nucleation and growth model falls short in providing adequate information. In this talk, I will present how multimodal X-ray methodology, rather than simply offering new tools, is expanding our ability to study, understand, and design complex structures. A combination of in situ X-ray spectroscopic (XAS/XES), scattering techniques (PDF/PXRD/SAXS), and microscopic methods (ptychography), along with rapid data acquisition, helps uncover the intricate chemical processes involved in synthesizing functional materials. These techniques provide complementary insights into chemical reactions in solution, as well as the nucleation, growth, and crystal phase transitions of nanoparticles and their assemblies.

We will focus on materials exhibiting varying degrees of chemical, structural, and morphological complexity, such as hollow Cu cubes, CuPd icosahedral nanoparticle assemblies, and CoO assemblies. Using the example of ZnS nanorods, we will demonstrate how combination of in situ XAS and XES allows for the monitoring of the emergence of the electronic and atomic structures of ZnS nanorods. To illustrate the advantages of operando high-energy X-ray scattering measurements, we will discuss the photodegradation of CuBi2O4 photoanodes. Finally, I will address the strengths and limitations of using synchrotron methods for in situ and operando studies in solution.

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- 1. Grote et al, Nature Comm. 12 (2021) 1.
- 2. Grote et al, Nature Comm. 13 (2022) 4971.
- 3. Grote et al, Scientific Reports 13 (2023) 31.
- 4. Derelli et al, Small 20 (2024) 2311714.
- 5. Klemeyer et al, J. Am. Chem. Soc. 146 (2024) 33475.
- 6. Derelli et al, Angew. Chem. Int. Ed. 62 (2023) e202307948.



# Visualizing the Competition between Etching and Sulfurization via *in situ* X-ray ptychography and X-ray Absorption Spectroscopy <sub>WE O1</sub>

S.-A. Hussak<sup>1,2\*</sup>, L. Klemeyer<sup>1</sup>, S. Röper<sup>1,2</sup>, M. Seyrich<sup>2</sup>, F. Wittwer<sup>2</sup>, S. Haugg<sup>1</sup>, O. Mathon<sup>3</sup>, C.G. Schroer<sup>1,2</sup> and D. Koziej<sup>1,2</sup>

<sup>1</sup>Institute for Nanostructure and Solid-State Physics, Center for Hybrid Nanostructures, University of Hamburg, 22761 Hamburg, Germany <sup>2</sup>Centre for X-ray and Nano Science, Deutsches Elektronen-Synchrotron DESY, 22607 Hamburg, Germany <sup>3</sup>European Synchrotron Radiation Facility, B.P.220, F-38043 Grenoble Cedex, France

\*e-mail: sarah-alexandra.hussak@uni-hamburg.de

Chemical transformations, such as redox processes or anion and cation exchange reactions in nanoparticles, are crucial for tailoring their functional properties in catalysis and energy applications. In this study, we employ a multimodal X-ray approach to investigate the transformation of  $Cu_2O$  nanocubes into CuS in the liquid phase. In situ X-ray ptychography and nano diffraction reveal the morphological and structural transformations of the particles during sulfurization, showing the formation of hollow CuS structures. Complementary in situ high- energy resolution fluorescence detected X-ray absorption spectroscopy (HERFD-XAS) demonstrates that, at elevated temperatures, sulfurization proceeds through a direct conversion of  $Cu_2O$  to CuS. At room temperature, however, a competing etching reaction dominates, leading to the dissolution of  $Cu_2O$  and the formation of a solvent-coordinated copper complex.

Our results provide insight into both reaction pathways, emphasizing the influence of temperature and reaction conditions on the competing processes. This work highlights the power of multimodal and complementary X-ray techniques for in situ experiments in solution and offers new perspectives on the design and synthesis of metal chalcogenide nanoparticles from their precursors.



Figure 1. Ptychographic reconstructions of **a** pristine Cu<sub>2</sub>O nanocubes on a Kapton substrate (ex situ), **b** CuS particles in the reaction solution after 1 h, and **c** after 6 h of sulfurization reaction. **d** Schematic illustration of the morphological changes during heat-induced sulfurization.

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## XAS and XMCD studies of spinel-type HEOs

V. Bilovol1\*, M. Sikora2, M. Szymczak3, K. Berent1 and J. Cieślak3

WE\_02

<sup>1</sup>Academic Centre for Materials and Nanotechnology, AGH University of Krakow, 30-059, Krakow, Poland <sup>2</sup>National Synchrotron Radiation Centre SOLARIS, Jagiellonian University, 30-392 Krakow, Poland <sup>3</sup>AGH University of Krakow, Faculty of Physics and Applied Computer Science, 30-059 Krakow, Poland

\*e-mail: vbilovol@agh.edu.pl

A new class of oxide systems known as High Entropy Oxides (HEOs) was for the first time formulated and reported only ten years ago [1]. The fundamental concept behind high-entropy materials is to employ a variety of principal elements (usually 5 or more in nearly equal molar ratios) to achieve a high configurational entropy of the system, thereby enhancing the formation of a simple, one-phase solid solution. The study of their structural and functional properties is of great interest [2], as these properties cannot be simply deduced from the crystal structure and the nature of the individual elements. Therefore, the band structure is highly likely not that of a statistical average of the five binary oxides, and it is not clear if structural and magnetic correlations can be preserved despite the chemical disorder.

The magnetic properties of HEOs are strongly dependent on the elemental composition and distribution between octahedral and tetrahedral sites of the spinel structure. We were able to collect at the PIRX beamline of SOLARIS synchrotron a comprehensive set of XAS and XMCD spectra from five HEO samples, namely  $Co_x(Cr,Fe,Mn,M)_{3-x}O_4$ , where x = 0.33, 0.50, 0.60, 0.82, 1.00 at% in TEY mode at L edge of Co, Cr, Mn, Fe and Ni.

The L3 XMCD spectra showed the evolution of signs that clearly demonstrate ferromagnetic (negative sign) and anti-ferromagnetic (positive sign) alignment of spin moments of the given element sublattice with respect to the external magnetic field. Upon comparison of the XAS and XMCD spectra of all the metals distributed in the spinel lattice, it was qualitatively confirmed that Cr and Fe ions are trivalent, Mn shows a mixed valence (2+/3+/4+), and Ni ions are divalent, while Co is predominantly 2+ with admixture of 3+ in the high entropy magnetic spinel oxides. Based on the XMCD shape and amplitude, the occupancy tendency is unambiguous for Cr and Ni (both octahedral) and Co (tetrahedral), while for Mn and Fe it varied as follows: for x <= 0.6, Fe ions were situated exclusively on tetrahedral positions, while Mn ions for x > 0.6 on octahedral ones. Based on the above information, the profile of cation distribution in spinel HEOs, where Co content is a variable, is derived experimentally and compared to theoretical predictions provided by DFT.

## References

1. C.M. Rost, E. Sachet, T. Borman, et al., Nat. Commun. 6 (2015) 8485.

2. J. Cieślak, M. Reissner, K. Berent, et al., Acta Materialia 206 (2021) 116600.



## **Insights on condensed matter functions by FEL x-ray pulses**

H.T. Lemke<sup>1\*</sup>

WE\_I3

<sup>1</sup>Paul Scherrer Institute, 5232 Villigen, Switzerland

\*e-mail: henrik.lemke@psi.ch

Condensed matter and its functionality determine most processes in life and technology and therefore natural sciences. New insights into transition processes that happen on ultrashort timescale out of equilibrium can be gained using ultrashort and coherent x-ray pulses from Free Electron Laser (FEL) sources. With their femtosecond pulse length, it becomes possible to interrogate structural rearrangements during transitions on the timescale of atomic motions. With hard X-ray FEL pulses, structures on atomic length scale, as well as element specific spectroscopic signatures can be interrogated, which provides highly complementary information to ultrafast laser spectroscopy. Here, ultrafast spectroscopy signals remain often ambiguous in interpretation which can be resolved by the information of ultrafast X-ray probes.

Especially the interactions in condensed matter by vibrations, magnetic state, and electronic state or polarisation can create a complex interplay and deformations which may lead to e.g. macroscopic and stable quantum states, interesting for novel technical applications. Ordering or transition processes on different degrees of freedom may be targeted and measured by selective X-ray methods like resonant and non-resonant X-ray diffraction as well as spectroscopic techniques. The typically ultrafast processes can be triggered by excitation into electronic or vibrational states involved in their transformation by light. Selectivity can be accomplished by addressing states resonantly by choice of wavelength of an ultrafast exciting laser pulse. Those typically range from THz to Ir wavelengths for vibrations, phonons, magnons, polarons, to visible and UV wavelength for electronic excitations.

The main techniques and principles in ultrafast hard X-ray studies of condensed matter will be summarized with state-of-the-art examples of questions and results gained at SwissFEL and other FEL light sources.



# Ultrafast x-rays reveal new opportunities in nanostructured quantum materials

**WE I4** 

M. Lorenc<sup>1,5\*</sup>, E. Janod<sup>2</sup>, S. Iwai<sup>3</sup>, H. Tokoro<sup>4</sup>, H. Cailleau<sup>5</sup>, E. Pastor<sup>5</sup>, C. Mariette<sup>6</sup>, P. Rabiller<sup>5</sup>, E. Trzop<sup>5</sup>, D. Lea<sup>5</sup>, R. Mandal<sup>2,5</sup> and J. Guzman<sup>2,5</sup>

<sup>1</sup>CNRS, Univ Rennes, DYNACOM—IRL2015, The University of Tokyo, Tokyo, Japan
 <sup>2</sup>Nantes Université, CNRS, Institut des Matériaux de Nantes Jean Rouxel, IMN, Nantes, France
 <sup>3</sup>Department of Physics, Tohoku University, Sendai, Japan
 <sup>4</sup>Department of Materials Science, Institute of Pure and Applied Sciences, University of Tsukuba, Japan
 <sup>5</sup>Univ Rennes, CNRS, IPR (Institut de Physique de Rennes)—UMR 6251, Rennes, France
 <sup>6</sup>European Synchrotron Radiation Facility, B.P.220, F-38043 Grenoble Cedex, France

\*e-mail: maciej.lorenc@univ-rennes.fr

The ultrafast light pulses can induce new functionality in quantum materials [1] not accessible via the application of conventional thermodynamic parameters like temperature or pressure [2]. The non-equilibrium pathways hold great technological potential [3] and topical examples include insulator to metal transitions (IMT) for optical memories [4] or neural networks for AI [5]. From fundamental standpoint, several degrees of freedom couple during such transformations, which gives rise to multiscale dynamics in time and space. While structural reorganizations often play an important role in the stabilization of the newly formed electronic state, the establishment of a new macroscopic structural order requires long-range crystalline deformations, involving the propagation of acoustic waves. In a recent work, we evidenced a macroscopic transformation pathway from semiconductor to metal in nanocrystals of trititanium pentaoxide ( $Ti_3O_5$ ) [6], that involved generation and propagation of volumic strain waves. In another example, we studied a prototypical Mott insulator V<sub>2</sub>O<sub>3</sub>, a widely considered test bed for quantum materials. While  $V_2O_3$  is metallic at room temperature (PI), below 150 K it transitions to insulating phase (AFI) involving antiferromagnetic and ferroelastic symmetry breaking. In addition, AFI to PI transition also involves a non-symmetry-breaking volume contraction. Transient reflectivity studies of  $V_2O_3$  in the AFI phase unveiled a transformation within time consistent with the acoustic propagation of strain waves. Astoundingly, transformation yield of up to 100% can be achieved above a threshold laser fluence. The granular morphology of  $V_2O_3$  films favours complete transformation to the metallic phase, unlike a single crystal where in-plane clamping takes place and hinders photoinduced IMT [7]. The ultrafast x-ray diffraction on thin films revealed volume contraction and confirmed the quasi-complete transformation to metallic phase on the acoustic time-scale of strain waves, also observed in the optical studies.

In brief, we discuss how photoinduced strain and material morphology can provide with control knobs [6,8] in the fast-growing field of non-equilibrium material science, largely driven by progress in femtosecond x-ray techniques. The grand objective is to develop efficient stimuli and frugal functional materials operating either at ultrafast timescales or permanently switchable, with functionality on demand.

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#### References

- 1. A. de la Torre et al., Rev. Mod. Phys. 93, 041002 (2021). D. N. Basov et al., Nature Materials 16 (2017) 1077.
- 2. Y. Tokura et al., Nature Physics 13(2017) 11.
- 3. A. Sood et al., Science 373 (2021) 352.
- 4. S.-i. Ohkoshi, et al., Nature Chem. 2 (2010) 539.
- 5. V. Guiot et al., Nature Commun. 4, 1 (2013). D. Babich et al., PRAppl. 17 (2022) 014040.
- 6. C. Mariette, et al., Nature Communications 12 (2021) 1-11.
- 7. A. von Reppert et al., Appl. Phys. Lett. 113 (2018) 123101.
- 8. T. Amano et al., Nature Phys. (2025)., DOI:10.1038/s41567-024-02628-4



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## Fs-XES of the NIR-active spin-flip emitters

WE O3

M. Nowakowski<sup>1</sup>, W. Kitzmann<sup>2</sup>, K. Heinze<sup>2</sup>, D. Khakhulin<sup>3</sup>, M. Biednov<sup>3</sup> and M. Bauer<sup>1</sup>

<sup>1</sup>Fakultät für Naturwissenschaften, Universität Paderborn, 33098 Paderborn
 <sup>2</sup>Department Chemie, Johannes Gutenberg-Universität Mainz, 55128 Mainz
 <sup>3</sup>European XFEL GmbH, 22869 Schenefeld, Germany

\*e-mail: michal.nowakowski@uni-paderborn.de

The design of photoactive complexes with earth-abundant metal ions is very challenging, yet a very active and rapidly growing field of research. The ultimate aim is to replace expensive noble and rare earth metal ions with earth-abundant and hence sustainable metal ions for applications such as photosensitizing, photoluminescence, electroluminescence or photo(redox) catalysis [1].

Emission in the near infrared (NIR) spectral region is challenging due to the often, efficient nonradiative multiphonon relaxation via high energy oscillators. The scarcity of luminescent 3d metal complexes arises from the presence of low-energy ligand field excited states (MC, metal centered states), enable efficient non-radiative decay to the ground state. The new chromium(III) complex [Cr(ddpd)<sub>2</sub>]<sup>3+</sup> [2,3] is based on the tridentate ligand (ddpd). The "molecular ruby" features two very sharp NIR emission bands at 775 and 738 nm arising from the metal centered *spin-flip transitions*  ${}^{2}E \rightarrow {}^{4}A_{2}$  and  ${}^{2}T_{1} \rightarrow {}^{4}A_{2}$ , respectively. It features a high photoluminescence quantum yield of  $\Phi = 11$  % and a luminescence lifetime of  $\tau = 898 \,\mu s$  in water at room temperature. [3] According to Tanabe-Sugano diagram for d<sup>3</sup> octahedral systems, the high ligand field in the complex possibly allows the excited  ${}^{4}T_{2}$  state to relax via previously inaccessible  ${}^{2}T_{2}$  state. [2]

The deexcitation cascade was studied using femtosecond pump-probe Cr K-edge X-ray emission spectroscopy (fs-XES) on FXE beamline (Fig. 1). The experiment revealed a complex multiplet structure of a transient Cr K $\alpha_1$  emission line ( $\Delta$  K $\alpha_1$ ) in Cr(ddpd) and reference Cr(III) compounds, while relatively uneventful evolution of  $\Delta$ K $\beta$  signals. The sensitivity of  $\Delta$ K $\alpha_1$  was used to extract dynamics and sequence of the excited states. The extraordinary ligand field affects the excited state landscape, affecting the spin-flip luminescence



Figure 1. Experimental setup used at FXE beamline and molecular structure of

lifetimes. The rigid, octahedral structure of the ddpd ligand has a destabilizing effect on Frank-Condon <sup>4</sup>CT (charge-transfer) states manifold. The preferred subsequent reduction of the axial Jahn-Teller distortion occurs via deexcitation through the <sup>2</sup>T<sub>2</sub> state leading to the elongation of the luminescence lifetimes in the complex. These findings are in line with theoretical predictions concerning excited states in the molecule and with transient absorption spectroscopic data [4].

While these results represent a step forward in understanding ultrafast phenomena, certain unexpected effects—such as  $\Delta K\alpha 1$  evolution—still require a rigorous explanation. The molecule can function both independently and within charge-transfer heterobimetallic assemblies [5]. By identifying key excited states, chemical engineering can leverage its long-lived luminescence for molecular-scale detection of pressure, pH, polarity, changes in electromagnetic fields, or anisotropic forces.

- 1. P. S. Wagenknecht and P. C. Ford, Coord. Chem. Rev. 255 (2011) 591.
- 2. S. Otto et al., Angew. Chemie Int. Ed. 54 (2015) 11572.
- 3. S. Otto et al., Coord. Chem. Rev. 359 (2018) 102.
- 4. W. Kitzmann et al., Dalton Trans. 51 (2022) 6519-6525.
- 5. M. Nowakowski et al., Adv. Sci. 11 (2024) 2404348.



# Bridging experiment and simulation: X-ray free-electron laser probing of optically induced melting of Palladium WE O4

J. Antonowicz<sup>1\*</sup>, A. Olczak<sup>1</sup>, K. Sokolowski-Tinten<sup>2</sup>, P. Zalden<sup>3</sup> and R. Sobierajski<sup>4</sup>

<sup>1</sup>Faculty of Physics, Warsaw University of Technology, 00-662 Warsaw, Poland <sup>2</sup>Center for Nanointegration Duisburg-Essen (CENIDE), University of Duisburg-Essen, 47048 Duisburg, Germany <sup>3</sup>European XFEL, 22869 Schenefeld, Germany <sup>4</sup>Institute of Physics Polish Academy of Sciences, 02-668 Warsaw, Poland

\*e-mail: jerzy.antonowicz@pw.edu.pl

Due to its extremely short timescale, the non-equilibrium melting of metals is exceptionally difficult to probe experimentally. As a result, our understanding of melting mechanisms relies primarily on theoretical predictions. This work investigates the ultrafast melting of thin polycrystalline Pd films using optical laser pump – X-ray free-electron laser probe experiments combined with molecular dynamics simulations. By acquiring X-ray diffraction snapshots with sub-picosecond resolution, we capture the atomic structure of the sample during its transition from the crystalline to the liquid state.

Bridging the timescales of experiments and simulations enables us to formulate a realistic and consistent microscopic picture of the crystal-liquid transition<sup>1</sup>. We demonstrate that the melting process accelerates progressively with increasing absorbed energy density. This acceleration results from a gradual shift in the solid-to-liquid transition mechanism, evolving from heterogeneous melting—initiated within structurally disordered grain boundaries—to homogeneous melting, which occurs catastrophically within the crystal volume on a picosecond timescale, close to that of electron-phonon coupling in Pd.



Figure 1. Structural evolution of the laser-irradiated polycrystalline Pd film, as revealed by molecular dynamics simulations. Experimental (blue) and theoretical (green) X-ray diffraction patterns are compared for different energy densities and pump-probe delay times, capturing various stages of the melting transition. Atoms are color-coded based on their deviation from local centrosymmetry.

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## References

1. J. Antonowicz et al., Acta Mater. 276 (2024) 120043.



## Polish research at the European XFEL

R. Sobierajski<sup>1,\*</sup>, W. Gawełda<sup>2</sup>, A. Glinka<sup>2</sup>, K. Jarzembska<sup>3</sup>, R. Kamiński<sup>3</sup>, M. Kozak<sup>2</sup>, **WE\_I5** J. Kubicki<sup>2</sup>, D. Milewska<sup>4</sup> and K. Nowakowska<sup>2</sup>

<sup>1</sup>Instytut Fizyki PAN, 02-668 Warszawa <sup>2</sup>Wydział Fizyki, Uniwersytet im. Adama Mickiewicza w Poznaniu, 61-614 Poznań <sup>3</sup>Wydział Chemii, Uniwersytet Warszawski, 02-089,Warszawa <sup>4</sup>Narodowe Centrum Badań Jądrowych, 05-400 Otwock-Świerk

\*e-mail: ryszard.sobierajski@ifpan.edu.pl

The European X-ray Free Electron Laser (EuXFEL) is an international laboratory where intense ultrashort pulses X-rays are generated and applied in scientific research. It contributes to the significant expansion of our knowledge in many scientific disciplines (m.in. medicine, pharmacology, chemistry, materials science, nanotechnology, energy and electronics) and the emergence of new technologies affecting our daily lives [1]. During the presentation, a Project financed by the Ministry of Science and Higher Education to support the Polish scientific community in the use of new research opportunities offered by EuXFEL will be presented [2]. The project includes the following activities:

- 1. training in basic knowledge of XFEL applications and research techniques used, including several months of internships for young researchers at institutions participating in XFEL research and a lecture on XFEL for undergraduate and doctoral students;
- 2. funding the participation of Polish scientists in conferences and workshops on XFEL research;
- 3. assistance in establishing or expanding scientific contacts with EuXFEL employees and/or research groups carrying out similar research in Poland and abroad;
- 4. assistance in the preparation of research projects in competitions for measurement time on XFEL sources, also by performing pilot and complementary studies.

The lecture will end with a presentation by the Network of Centres of Excellence in the field of research using XFEL (CD XFEL), in which the above program is implemented.

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- 1. https://biuletyn.synchrotron.org.pl/wp-content/uploads/2023/12/Biuletyn\_all\_12\_2023.pdf
- 2. www.ifpan.edu.pl/cd-xfel



## In situ nanopowder diffraction – between Bragg and diffuse scattering

Z. Kaszkur<sup>1\*</sup> and I. Smirnov<sup>2</sup>

TH\_I1

<sup>1</sup>Institute of Physical Chemistry, Warszawa 01-224, Poland <sup>2</sup>University of Warsaw, Faculty of Chemistry, Warszawa 02-093, Poland

\*e-mail: zkaszkur@ichf.edu.pl

Structural study of matter between amorphous and crystalline state requires approaches evolving from Debye formula as the surface cannot be treated as the phase. Nanocrystalline solids may exhibit attractive properties due to quantum confinement and extended surface. They affects thermodynamics ruled by high thermal fluctuations and surface energy. No wonder that their properties can be revealed the best via chemical interaction with the surface, in situ. The method developed by us monitors morphology of a nanopowder via diffraction addressing subtle evolution of peak positions, intensities, widths etc. during physico-chemical processes, interpreted via atomistic simulations. It provides many surprizing observations helping to understand complex processes going on. For the technique, crucial is excellent repeatability of peak position achievable (down to 10<sup>-4</sup> deg), enabling interpretation of small changes regardless of limited accuracy.







Figure 1: Evolution of ALP of Pt nanocrystal on desorption of hydrogen slowed down by reverse spill over phenomenon to last more than 5

The method addresses specific phenomena fingerprints of allowing their detection and analysis instead of global model fitting in both: real and reciprocal spaces, that tends to smear out crucial marks (like in PDF or Rietveld full profile methods). Examples presented include nanometal relaxation [1], monitoring chemisorption phenomena [2], surface reconstruction (fig.2) [3] or surface segregation kinetics in binary alloys [4]. E.g. complex phenomena of rough reconstruction of the surface of Pt nanocrystals NO in atmosphere are responsible for a low temperature coalescence of Pt [5]. Long -run monitoring of surface segregation in PdAg alloy shows segregation of Ag in He atmosphere and markedly slower segregation of Pd in CO atmosphere. This repeatable process reveals different mechanism of diffusion in both cases when in the second, CO

bonds and captures Pd at the surface, hinders surface diffusion and creation of vacancies responsible for the most effective bulk diffusion mechanism (fig.3) [4].



## XVI International School and Symposium on Synchrotron Radiation in Natural Science Poland, 25-30 May 2025



*Figure 3: Evolution of 111 XRD peak of PdAg alloy during surface segregation process.* 



Figure 4: 111 and 200 peaks of diffraction patterns (Cu Ka radiation)for a range of Marks' decahedra. Intensity is divided by the number ofatoms. The numbers of model atoms are given in the legend. For comparison added are diffraction patterns calculated for the model of randomly multitwinned (MT) cluster consisting of 11158 atoms

The method allows insight into stacking faults occureence in FCC nanocrystals. As the energy difference between the three possible 111 plane arrangements is low, many chemicaly synthesized nanocrystals have complex domain structure consisting of locally twinned domains. It can be seen by the oversized 111 peak and slight shifts of 111 and 200 peaks. Although already noted by Warren in 1960-ties, the phenomenon can be modelled by us and the average number of domains deduced [6]. Evolution of this subtle structure can be monitored during temperature program and was observed for nanoalloys of non-miscible in bulk alloy PtAu [7].

Most of the observed processes can be modelled using designed and written by us suite of procedures CLUSTER [8] enabling graphical model building, constrained relaxation. dynamics (MD) or configurational minimization (Monte Carlo). Simulations for metals employ n-body pseudopotentials after Sutton&Chen [9].

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- 1. Z.Kaszkur, I.Smirnov, <u>https://arxiv.org/pdf/2212.06926</u>
- 2. P. Rzeszotarski, Z. Kaszkur, Phys.Chem.Chem.Phys. 11 (2009) 5416–5421.
- 3. Z. Kaszkur, P. Rzeszotarski, and W. JuszczykJ, Appl.Cryst.47 (2014) 2069–2077.
- 4. Z. Kaszkur, W. Juszczyk., and D. Lomot., Phys.Chem.Chem.Phys. 17 (2015) 28250-28255.
- 5. Z. Kaszkur, B. Mierzwa, W. Juszczyk, P. Rzeszotarski, and D. Lomot., RSC Adv. 4 (2014) 14758-14765.
- 6. I. Smirnov, Z.Kaszkur, A.Hoell, Nanoscale, 15 (2023) 8633.
- 7. I.Smirnow, Thesis, https://rcin.org.pl/dlibra/publication/275347/edition/239362/content
- 8. Z.Kaszkur, B.Mierzwa, program CLUSTER, https://kaszkur.net.pl/index.php/cluster/
- 9. A.P. Sutton, J. Chen. Phil. Mag. Lett., 61 (1990) 139-146.



## Time-resolved serial crystallography at synchrotrons and XFELs

P. Nogly1\*

TH\_I2

<sup>1</sup>Dioscuri Centre for Structural Dynamics of Receptors, Faculty of Biochemistry, Biophysics and Biotechnology, Jagiellonian University, Kraków, Poland

#### \*e-mail: przemyslaw.nogly@uj.edu.pl

Chloride transport is an essential process maintaining ion balance across cell membranes, cell growth, and neuronal action potentials. However, the molecular mechanism of the transport remains elusive. Among chloride transporters, light-driven rhodopsins have gained attention as optogenetic tools to manipulate neuronal signaling. We combined time-resolved serial crystallography (SwissFEL and SLS synchrotron) to provide a comprehensive view of chloride- pumping rhodopsin's structural dynamics and molecular mechanism throughout the transport cycle from 10 ps to 50 ms [1]. We traced transient anion binding sites, obtained evidence for the mechanism of light energy utilization in transport, and identified steric and electrostatic molecular gates ensuring unidirectional study of the mechanistic features enabling finely controlled chloride transport across the cell membrane.

Furthermore, I will present insights into the photochemistry and selectivity of retinal isomerization in proton-pumping rhodopsin [2] and show that, in favorable cases, even larger structural changes can be captured in crystals [3].

Our recent study of a distinct photoreceptor, Light-Oxygen-Voltage (LOV) domain, will be introduced [4,5]. The first insights into the structural dynamics of LOV photoactivation will be presented, providing the basis for proposing a molecular mechanism of a covalent thioether bond formation between a flavin mononucleotide cofactor and a reactive cysteine, Cys57 (unpublished).

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- 1. Mous, S. et al. Science 375 (2022) 845.
- 2. Nogly P et al., Science 361 (2018) eaat0094.
- 3. Weinert et. al., Science 365 (2019) 61.
- 4. Gotthard et al. IUCrJ (2024) 749.
- 5. Gotthard et al. IUCrJ (2024) 792.



# Kinetic studies of the formation of arsnolite inclusion compound with hydrogen TH O1

P.A. Guńka<sup>1\*</sup>, K.F. Dziubek<sup>2</sup> and F. Capitani<sup>3</sup>

<sup>1</sup>Faculty of Chemistry, Warsaw University of Technology, 00-664 Warszawa, Poland <sup>2</sup>Institut für Mineralogie und Kristallographie, Universität Wien, Wien, A-1090, Austria <sup>3</sup>Synchrotron SOLEIL, L'Orme des Merisiers, 91192 Gif sur Yvette Cedex, France

\*e-mail: piotr.gunka@pw.edu.pl

Hydrogen diffusion through porous and non-porous solid materials is crucial for fundamental studies on hydrogen migration within the Earth's crust and its deep reservoirs.<sup>1</sup> Investigations of hydrogen inclusion compounds are an integral part of the ongoing research into hydrogen- storage materials, which have potential applications in environmentally friendly transportation.<sup>2</sup>

This study presents the kinetics of hydrogen permeation in single crystals of arsenolite, the cubic polymorph of arsenic(III) oxide. The experiments were conducted under high pressure using Fourier-transform infrared spectroscopy. Two single crystals of arsenolite were placed in diamond anvil cells and subjected to pressures of 1.37(5) GPa and 1.47(5) GPa for 11 hours and 1 hour, respectively. Hydrogen was used both as a pressure-transmitting medium and as a reactant forming the inclusion compound. Arsenolite was completely transformed into  $As_4O_6 \cdot 2H_2$ , with a linear growth rate of the inclusion compound, indicating that the rate-limiting step in the transformation is the reaction at phase boundaries rather than diffusion. Notably, at 1.37(5) GPa, two distinct domains of linear growth with different rates were observed.

Furthermore, a method for growing thin plates of  $As_4O_6$  single crystals via spatial confinement will be presented. The use of micro-sized, thin-layered platelets (as shown in Figure 1) has proven to be an effective approach for studying diffusion kinetics in crystals. Measurements conducted at the SMIS infrared spectromicroscopy beamline at the SOLEIL Synchrotron confirm the effectiveness of this novel methodology.<sup>3</sup>



Figure 1. Schematic drawing of the experimental setup.

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- 1. V. Zgonnik, Earth-Sci. Rev. 203 (2020) 654.
- 2. W. Fang, C. Ding, L. Chen, W. Zhou, J. Wang, K. Huang, R. Zhu, J. Wu, B. Liu, Q. Fang, X. Wang, J. Wang, Energy Fuels 38 (2024) 13539.
- 3. P. A. Guńka, K. F. Dziubek, F. Capitani, J. Phys. Chem. C 127 (2023) 15871.



## The health and life sciences research in the Canadian Light Source

Pawel Grochulski

TH\_02

# Canadian Light Source, 44 Innovation Boulevard, Saskatoon SK S7N 2V3 Canada

## \*e-mail: pawel.grochulski@lightsource.ca

The Biological and Life Sciences (Bio/Life) Department, an essential unit at the Canadian Light Source, is composed of several facilities<sup>1</sup>: Canadian Macromolecular Crystallography Facility (CMCF), Biomedical Imaging and Therapy Facility (BMIT), Biological X-ray Spectroscopy (BioXAS) and Mid Infrared Spectro-microscopy (Mid-IR).

Recently, we have upgraded the infrastructure of the Bio/Life Department, especially at the BMIT beamlines<sup>2</sup>. Now, the beamlines provide quality images from microns to tens of centimetres and produce fast images in in-situ and operando experiments for a broad range of spatial scales. The CMCF beamlines are the only beamlines that can be accessed exclusively remotely. The CMCF beamlines support research not only of academic researchers but also about 30 pharmaceutical companies involved in drug development programs. The BioXAS beamlines have been tailored to characterize and image metal distributions in the biological samples at physiological concentrations. Mid-IR beamline and associate off-line instruments are ideal for studying biomedical and biological materials.

For the last 5 years the Bio/Life Department contributed to more than 100 peerreviewed publications annually<sup>3</sup>. The health and life sciences investigations ranged from brain diseases<sup>4</sup>, bone diseases<sup>5,6,7</sup>, COVID-19<sup>8</sup>, malaria treatment<sup>9</sup>, cancer therapy<sup>10</sup>, developing new medicines<sup>11</sup>, small and mid-size live animals' studies<sup>12</sup>, TB treatement<sup>13</sup>, as well as connective tissues imaging<sup>14</sup>. This presentation will highlight the most important discoveries resulting from our work.

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- 1. P. Grochulski, S. Labiuk NIM-B. 499 (2021) 70.
- 2. S. Gasilov et al., J. Synchrotron Rad. 31 (2024) 1346.
- 3. https://user.lightsource.ca/publications/
- 4. M.J. Pushie at et al. Metallomics. 16 (2024) mfae054.
- 5. A. Brown, Laryngoscope 134 (2024) 2879-2888.
- 6. D. Giese, Audiology and Neurology 29 (2024) 487.
- 7. 7. D.M.L. Cooper et al., Bone 176 (2023) 116864.
- 8. V.W. Shurtleff et al., J. Med. Chem 67 (2024) 3935.
- 9. Mendez et al., Acta Cryst. F81 (2025) 1.
- 10. E.N. Aguilar AACR J. 23 (2024) 837.
- 11. Pistofidis et al., Nature 638 (2024) 270.
- 12. K. Harrison et al., Bone 187 (2024) 117171.
- 13. Y. Zhou et al., BMCL 117 (2025) 130027.
- 14. H.A. Sardroud et al., Biofabrication 17 (2025) 015014.



## Probing Batteries with X-rays and Neutrons from Cradle to Grave

M. Knapp

TH\_I3

Karlsruhe Institute of Technology (KIT), Institute for Applied Materials (IAM), D-76344 Eggenstein-Leopoldshafen, Karlsruhe, Germany

## \*e-mail: michael.knapp@kit.edu

Lithium-ion batteries (LIBs) have transformed modern technology, powering everything from portable electronics to electric vehicles and renewable energy storage systems. As the world shifts toward post-Lithium solutions, Sodiumbased cells are gradually making their debut in the mass market. In this evolving landscape, large-scale facilities drive the advancement of battery systems by providing crucial insights. Due to their unique capabilities, synchrotron and neutron radiation reveal essential details that are key to optimizing performance.

One of the main goals of ongoing studies is to transfer the knowledge gained from LIBs to emerging post-Lithium technologies, with X-ray diffraction and X-ray absorption spectroscopy offering in-operando data to enhance battery performance. At the same time, growing concerns over resource limitations have sparked the interest in reuse, recycling, and upcycling of batteries

Access to large-scale facilities is limited, making it increasingly important to precharacterize materials in-situ and in home-labs. This step is often done before utilizing synchrotron or neutron sources. In this presentation, typical experiments using synchrotron and neutron radiation to study battery systems will be showcased, with a comparison to what can be achieved in-house. Key strategies for successful experimental design will be shared, along with common pitfalls to avoid.



# Environmental molecular geochemistry developed by fluorescence XAFS using high energy resolution fluorescence X-ray detection TH I4

Yoshio Takahashi\*

Earth and Planetary Science, Graduate School of Science, The University of Tokyo, Japan \*e-mail: ytakaha@ecc.s.u-tokyo.ac.jp

X-ray absorption fine structure (XAFS) is an elemental speciation method that is highly element-selective and provides information on the valence and local structure of almost all elements. The fluorescence XAFS method allows the speciation of elements at lower concentrations and has therefore been applied to the speciation of elements in various natural samples and to the elucidation of chemical elemental processes in laboratory experiments simulating natural conditions. Thus, fluorescence XAFS is now an essential method in space geochemistry and environmental chemistry (Takahashi et al., 2025). This presentation will show (i) examples of the application of fluorescence XAFS methods to elements that are difficult to detect by conventional methods, and (ii) examples how new chemical species information has been obtained by increasing the energy resolution of the X-ray fluorescence (XRF) detection system to obtain XAFS. In particular, a new spectroscopic technique called High Energy Resolution Fluorescence Detection (HERFD)-XANES has been developed in recent years, which is obtained by detecting XRF with an energy resolution higher than the lifetime width of the electron orbitals (the energy resolution is 1 eV or better). This method is powerful in detecting new species in terrestrial materials, such as U(V) species adsorbed on iron oxides (Yomogida et al., 2024). Although the use of the crystal analyzer is the main method to increase the high energy resolution detection of fluorescence X-rays, recent advances using high energy resolution energy dispersive detectors are also given. In particular, the transitionedge sensor (TES) detector is a powerful detector with the energy resolution better than 5 eV, although the count rate of this detector is low such as 10 cps/element. Therefore, the multi-element TES detector is important to obtain XAFS spectra with better signal-tonoise ratio. The 5 eV energy resolution is not sufficient to obtain HERFD-XANES for various elements, but the TES detector is still useful to detect XRF with better signal-tobackground (S/B) ratio. In particular, the detection of trace elements whose XRF peak overlaps with that of major elements is very useful to obtain XAFS spectra for trace elements (Konagaya et al., 2021; Li et al., 2023). By making full use of these techniques, it is possible to understand physicochemical information at the atomic level and various elemental chemical processes based on this information. This information, in turn, will contribute to a better understanding of the Earth's material cycles and elemental cycles at different scales (= molecular geochemistry).

- 1. Konagaya R., Kawamura N., Yamaguchi A. and Takahashi Y., Highly-sensitive analysis of fluorescence XANES at europium (Eu) LIII-edge for the determination of oxidation state for trace amount of Eu in natural samples by Bragg-type crystal analyzer system, Chem. Lett. 50 (2021) 1570–1572.
- Li W., Yamada S., Hashimoto T., Okumura T., Hayakawa R., Nitta K., Sekizawa O., Suga H., Uruga T., Ichinohe Y., Sato T., Toyama Y., Noda H., Isobe T., Takatori S., Hiraki T., Tatsuno H., Kominato N., Ito M., Sakai Y., Omamiuda H., Yamaguchi A., Yomogida T., Miura H., Nagasawa M., Okada S. and Takahashi Y., High-sensitive XANES analysis at Ce L2-edge for Ce in bauxites using transition-edge sensors: Implications for Ti-rich geological samples, Anal. Chim. Acta 1240 (2023) 340755.
- Takahashi Y., Yamaguchi A. and Yomogida T., Environmental geochemistry of radionuclides (Environmental radiochemistry), Treatise on Geochemistry (Third Edition) 6 (2025) 105–150.
- 4. Yomogida T., Hashimoto T., Okumura T., Yamada S., Tatsuno H., Noda H., Hayakawa R., Okada S., Takatori S., Isobe T., Hiraki T., Sato T., Toyama Y., Ichinohe Y., Sekizawa O., Nitta K., Kurihara Y., Fukushima S., Uruga T., Kitatsuji Y. and Takahashi Y., Application of transitionedge sensors for micro-X-ray fluorescence measurements and micro-X-ray absorption near edge structure spectroscopy: a case study of uranium speciation in biotite obtained from a uranium mine, Analyst 149 (2024) 2932–2941.



# Structural characterization of carbon materials derived from chestnut biomass

TH\_03

M. Jamshaid Shabbir<sup>1</sup>, B. Liszka<sup>2</sup>, M. Szubka<sup>1</sup> and K. Jurkiewicz<sup>1</sup>

<sup>1</sup>Institute of Physics, Faculty of Science and Technology, University of Silesia in Katowice, Chorzów, Poland <sup>2</sup>Faculty of Natural Sciences, University of Silesia in Katowice, Sosnowiec, Poland \*e-mail: mjamshaid.shabbir@us.edu.pl

One of the greatest challenges of modern science and industry is to reduce the carbon footprint and produce environmentally friendly materials. In the area of carbon materials, such goals can be achieved using cheap and abundant biomass-derived carbon precursors as well as catalytic-graphitization processes, which may reduce the temperature of the heat treatment required to obtain specific atomic structures. In this contribution, the structural characterization of the chestnut-derived carbon materials produced via standard carbonization at various temperatures (950, 1400, 1700°C) will be presented. Moreover, the heat treatment of the chestnut-derived materials in the presence of Si and CaCO3 particles was performed in order to evaluate their possible catalytic graphitization effect on the structure of the carbon matrix as well as the porosity. Si has been a well-known effective catalyst of graphitization for decades, while Ca is nowadays more often considered as an alternative due to its abundance, low cost, and that the catalytic effect of Ca is observed at relatively low temperatures, leading to enhancement of the degree of graphite-like order in carbon material. Moreover, Si and Ca elements may exist naturally in chestnut fruits. As a reference, cellulose-derived carbon (cellulose is one of the main components of chestnuts) was heat-treated in similar conditions and using the same catalysts.

The results of X-ray diffraction and Raman spectroscopy structural studies will be presented, which were used to characterize the transformation of the atomic-scale structure of the studied cellulose-based and chestnut-based carbon materials heattreated at various temperatures under Ar gas. Moreover, by supporting energydispersive X-ray spectroscopy measurements in a scanning electron microscope, it was possible to characterize their elemental and phase composition. The effect of the catalysts on the structure is evaluated. Moreover, the aim of these studies is to determine the impact of catalytic graphitization on the porosity of these materials. Since their porosity is mainly closed, it cannot be probed by gas adsorption methods. Therefore, the results of preliminary synchrotron small-angle X-ray scattering studies will be presented to reveal the changes in the porous structure.

The results of the presented studies may be significant when considering such carbon materials as anode active materials of sodium-/potassium-/lithium-ion cells where both atomic-scale structure of stacked graphene layers, as well as porosity, play a huge role.



# Electronic structure and chemical segregation process induced by redox conditions in PbHfO<sub>3</sub> single crystal.

D. Havryliuk<sup>1\*</sup>, J. Piecha<sup>1</sup> and J. Kubacki<sup>1</sup>

**TH\_04** 

<sup>1</sup>University of Silesia, Institute of Physic, Chorzów, Poland

\*e-mail: diana.havryliuk@us.edu.pl

The miniaturization of electronic devices initiated by the discovery of the transistor has spawned the need for the development of advanced materials to build new electrical circuits responsible for storing energy. A high energy storage density is required in modern practical applications, such as hybrid or electric cars, and is offered by antiferroelectric materials. Representatives of this type of material include lead zirconate PbZrO<sub>3</sub>, silver niobate AgNbO<sub>3</sub>, and lead hafniate PbHfO<sub>3</sub> [1, 2, 3]. The first two materials are well-studied in terms of electrical, structural, and electronic properties compared to the latter. The use of lead hafnate PbHfO<sub>3</sub> for application purposes is mainly based on studies of polycrystalline ceramics. This significantly limits the possibility of conducting a reliable analysis of physical properties reserved for single crystal materials.

The aim of our research was to grow PbHfO<sub>3</sub> single crystals. Initial electronic structure analyses performed using standard XPS showed interesting changes in the Pb/Hf ratio (Figure 1). To obtain a detailed analysis of the ion segregation on the PbHfO<sub>3</sub> surface, synchrotron radiation investigations were needed. The electronic structure studies were carried out in the temperature range RT – 250° C. This range takes into account two phase transition temperatures [2]. The changes in the shape of the electronic states of the Pb4f and Hf4f core lines and the valence band were associated with the segregation processes of the crystal components in the surface layer. The changes in the chemical composition ratio Pb/Hf indicate the migration of PbO complex along the easy diffusion path with increasing temperature.

A detailed analysis of Pb4f, O1s, and Hf4f and the valence band indicates a redistribution of the atomic arrangement in the surface layer in accordance with ionic diffusion from the bulk material. In particular, resonance photoemission studies have revealed a contribution of the d electrons of lead and hafnium atoms to the valence band structure in the phase transition region.



Figure 1. The changes of atomic concentration in the Pb/Hf ratio obtained for the *as grown* PbHfO<sub>3</sub> single crystal in wide range of temperatures obtained from the XPS study.

#### References

1. Chauhan V, Wang BX, Ye ZG., Structure, Antiferroelectricity and Energy-Storage Performance of Lead Hafnate in a Wide Temperature Range, Materials (Basel) 2023 16(11) 4144.

2. Peng-Zu, GeXin-Gui Tang, Ke Meng, Xian-Xiong Huang, Qiu-Xiang Liu, Yan-Ping Jiang, Wei-Ping Gong, Ting Wang, Ultrahigh energy storage density and superior discharge power density in a novel antiferroelectric lead hafnate, Materials Today Physics 24 (2022) 100681.

3. Dongxu Lia, Xiaojun Zeng, Zhipeng Li, Zong-Yang Shen, Hua Ha, Wengin Luo, Xingcai Wang, Fusheng Song, Zhumei Wang, Yueming Li, Progress and perspectives in dielectric energy storage ceramics, Journal of Advanced Ceramics 10(4) (2021) 675-703.

4. Bussmann-Holder A, Kim TH, Lee BW, Ko JH, Majchrowski A, Soszyński A, Roleder K., Phase transitions and interrelated instabilities in PbHfO3 single crystals, J Phys Condens Matter. 27(10) (2015) 105901.



## In situ/Operando X-ray spectroscopy coupled to electrocatalysis

B. Lassalle-Kaiser<sup>1,2\*</sup>

TH\_I5

<sup>1</sup>ITODYS, CNRS/Université Paris Cité, 15 rue Jean-Antoine de Baïf, 75013, Paris, France <sup>2</sup>Synchrotron SOLEIL, L'Orme des Meriiers, route départementale 128, 91190 Saint-Aubin, France

\*e-mail: benedikt.lassalle@u-paris.fr

Electrochemical energy conversion systems will become increasingly popular in the next decades, because of their strategic importance in converting, storing and releasing the energy gathered from renewable sources. Batteries, supercapacitors, electrolyzers or fuel cells all attract much attention in both applied and fundamental research, with the aim of improving their performance/cost ratio. In order to reach high efficiencies with environmental and social impacts as low as possible, a fundamental understanding of the functioning - and failure - of these devices under operating conditions is required. Because of the high penetration depth and the energy tunability of X-rays photons, Synchrotron-based techniques have emerged as ideal tools for this purpose.<sup>1</sup>

In this presentation, we will give an overview of the X-ray based techniques that can be coupled with electrochemical reactions under *in situ* or *operando* conditions, in order to gain insight in the local and electronic structure of electro-active materials, as well as their morphology. We will briefly describe instrumental aspects pertaining to these types of experiments (see figure 1) and provide examples of applications. A specific focus will



reactions related to energy storage, such as the Oxygen Evolving Reaction (OER), the Hydrogen Evolving Reaction (HER) or the CO<sub>2</sub> Reduction Reaction  $(CO_2RR).$ Specific example will be detailed, which focuses on the study of molecular electrocatalyst that for the are active  $CO_2$ reaction<sup>2</sup> reduction and perspectives for the future of the field will be given.

Figure 1. Simplified scheme of an in situ / operando X-ray spectroelectrochemical experiment.

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- 1. J. Timoshenko, B. Roldan-Cuenya, Chem. Rev. 121 (2021) 882.
- 2. S.-T. Dong, C. Xu, B. Lassalle-Kaiser, Chem. Sci. 14 (2023) 550.



## Ultrafast laser-driven X-ray sources at ELI Beamlines: From HHG and plasma X-ray to LPA-driven sources TH

**TH 05** 

T. Parkman<sup>1,2\*</sup>, O. Hort<sup>1</sup>, U. Chaulagain<sup>1</sup>, Y. Pulnova<sup>1,3</sup>, L. Jurkovičová<sup>1,2</sup>, M. Staněk<sup>1,2</sup>, R. Fauzul<sup>1</sup>, M. Lamač<sup>1</sup>, J. Vábek<sup>1,2</sup>, M. Raclavský<sup>1,2</sup>, D. Čáp<sup>1,2</sup>, V. Janota<sup>1,2</sup>, O. Morvai<sup>1,3</sup>, A. Morabito<sup>1</sup> and J. Nejdl<sup>1,2</sup>

<sup>1</sup>ELI Beamlines Facility, Extreme Light Infrastructure ERIC, 252 41 Dolní Břežany, Czech Republic
<sup>2</sup>Czech Technical University in Prague, 160 00 Prague, Czech Republic
<sup>3</sup>Charles University, FMP, 121 16 Prague, Czech Republic

\*e-mail: tomas.parkman@eli-beams.eu

ELI Beamlines is advancing ultrafast X-ray science through a multi-pronged strategy that transforms driving laser pulses into brilliant beams of short-wavelength radiation. Our program is built on three complementary approaches—high-order harmonic generation (HHG) in gases, plasma X-ray sources, and laser-plasma accelerator (LPA)-based sources—each implemented on dedicated beamlines that integrate fundamental research with diverse applications.

The HHG Beamline<sup>1</sup> is driven by state-of-the-art optical parametric chirped-pulse amplification (OPCPA) technology, as demonstrated by the L1 laser at ELI Beamlines. Delivering 45 mJ, 15 fs pulses at 1 kHz, the system significantly enhances the extreme ultraviolet (XUV) photon yield, overcoming traditional limitations of low flux and spectral mismatches that have hindered nonlinear XUV studies. Furthermore, a synchronized auxiliary beam with electronically adjustable delay enables advanced XUV-XUV pump-probe experiments for exploring ultrafast atomic and molecular processes.

Complementing this, a newly commissioned kHz laser-driven copper plasma X-ray source<sup>2</sup> (PXS) expands our experimental capabilities. This source can be driven by either previously mentioned in-house developed near-infrared TW laser (L1) or a conventional Ti:Sapphire laser delivering 12 mJ, 45 fs pulses at 1 kHz. It reaches fluxes of up to  $10^{12}$  K $\alpha$  photons/4 $\pi$ /s, with the OPCPA driver offering enhanced performance through higher pulse energy, shorter pulse duration, and enhanced pulse contrast. The source is implemented in setups for ultrafast X-ray diffraction and X-ray absorption/emission spectroscopy.

In parallel, significant progress has been made in LPA-based X-ray source generation. Current LPA-based X-ray sources achieve photon fluxes exceeding 10<sup>11</sup> photons per shot<sup>3</sup>, with ongoing research exploring novel techniques to further enhance their performance. The ELI Gammatron beamline<sup>4</sup> driven by a PW-class laser (L3) produces a collimated, partially coherent broadband X-ray beam. An in-house designed broadband X-ray mirror<sup>5</sup> further refines its performance, allowing for precise pump-probe experimental capability. Additionally, the Betatron X-ray source at the ELI plasma physics platform (P3) has been developed as a high-resolution backlighter for laser-matter interaction studies, including experiments in high-energy density physics and as a probe for advanced plasma physics experiments<sup>6</sup>.

Together, these initiatives demonstrate our commitment to pushing the frontiers of ultrafast X-ray science, providing versatile experimental platforms that address both fundamental research questions and applications in different fields of science and technology.

- 3. S. Fourmaux et al., Laser-based synchrotron X-ray radiation experimental scaling, Opt. Express 28 (2020) 3.
- 4. U. Chaulagain et al., ELI Gammatron Beamline: A Dawn of Ultrafast Hard X-ray Science, Photonics 9 (2022) 11.
- 5. M. Raclavsky et al., Multi-Lane Mirror for Broadband Applications of Betatron X-ray Source, Phot, 8 (2021) 579.
- 6. N. Jourdain, et al., The L4n laser beamline of the P3-installation: Towards high-repetition rate high-energy density physics at ELI-Beamlines, MRE 6.1 (2021) 015401.

<sup>1.</sup> O. Hort et al., High-flux source of coherent XUV pulses for user applications, Opt. Express 27 (2019) 6.

<sup>2.</sup> Y. Pulnova et al., Compact laser-driven plasma X-ray source for time-resolved diffraction, spectroscopy, and imaging experiments at ELI Beamlines, J. Synchrotron Rad. 32 (2025).



# Laser-plasma soft X-ray sources based on a gas puff target for application in absorption spectroscopy and coherence tomography

H. Fiedorowicz\*, A. Bartnik, P. Wachulak, Ł. Węgrzyński, T. Fok, M. Majszyk, W. Rafalak and M. Wardzińska **TH 06** 

Institute of Optoelectronics, Military University of Technology, 00-908 Warsaw, Poland \*e-mail: henryk.fiedorowicz@wat.edu.pl

The results of recent work on the development of laser-plasma sources of soft X-ray and extreme ultraviolet (EUV) radiation for application in science and technology are presented. The radiation is generated as a result of the interaction of nanosecond laser pulses with a gas puff target. The target, which is created by injection of a small amount of gas at high-pressure using a solenoid valve with a gas nozzle, is irradiated with nanosecond laser pulses with an energy of the order of a single J per pulse at a repetition rate of 10 Hz using commercially available Nd:YAG lasers. The use of a gas puff target enables efficient generation of radiation in the spectral range from about 1 nm to about 100 nm without nozzle degradation. The sources have been used in various fields of science and technology, including: plasma physics, metrology, emission and absorption spectroscopy, materials processing, nanoimaging, radiography and tomography, radiobiology and biomedicine.

This paper presents the results of the latest research aimed at improving the source parameters by using a gas puff target containing micron-sized liquid droplets or solid particles (aerosol targets). Using a laser-plasma source with a new target approach, a mobile laboratory workstation was developed for application in X-ray absorption fine structure (XAFS) spectroscopy and coherence tomography (CT). The view of the workstation and is CAD design are presented in Fig. 1.



Fig. 1. The view of the XAFS/CT laboratory workstation and its CAD design.

The XAFS/CT workstation is available to external users as part of the European RIANA (RIANA – Research Infrastructure Access in NAnoscience & nanotechnology) and Lasers4EU (Lasers4EU) projects.

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# PolyX@SOLARIS – beamline for multimodal microimaging and spectromicroscopy

**TH\_07** 

K. Sowa1\*, P. Wróbel2, T. Kołodziej1 and P. Korecki1,3

<sup>1</sup>SOLARIS National Synchrotron Radiation Centre, 30-392 Kraków, Poland
<sup>2</sup>Faculty of Physics and Applied Computer Science, AGH University of Science and Technology, 30-059 Kraków, Poland
<sup>3</sup>Institute of Physics, Jagiellonian University, 30-348 Kraków, Poland

\*e-mail: k.sowa@uj.edu.pl

PolyX (polychromatic X-rays and polycpaillary X-ray optics) is a newly developed bending magnet beamline at SOLARIS National Synchrotron Radiation Centre in Kraków, Poland [1]. The beamline is dedicated to X-ray microimaging and X-ray spectromicroscopy [2].

PolyX offers several techniques:  $\mu$ CT (~0.7 $\mu$ m resolution),  $\mu$ XRF,  $\mu$ XAS and  $\mu$ XES. Imaging resolution ~200nm will be possible via recently developed X-ray tomography with multiple ultranarrow cone beams [3]. The end station can be easily reconfigured; therefore, in addition to implementing other synchrotron methods at short notice, PolyX will also work as a test station for innovative hard X-ray methods as well as new solutions of X-ray optics, new imaging geometries or detection systems. PolyX can also provide a dedicated area for user experiments and/or user end-stations. A photo of the experimental setup is presented in Fig. 1.

The beamline operates in three modes: high flux (DMM, bandwidth 2%), high resolution (DCM, Si(111), bandwidth 0.02%) and a white beam mode. Polycapillary and monocapillary optics are used to generate hard X-ray beam in the energy range of 5-15keV with spot sizes in range  $\sim$ 5µm-100µm.

In this contribution a model experiment will be presented to show how to combine techniques available at the beamline to obtain complementary information about the sample (i.e. internal structure -  $\mu$ CT, elemental distribution -  $\mu$ XRF, speciation -  $\mu$ XAS/ $\mu$ XES).



Figure 1 A photo of the PolyX experimental setup (top view).

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- 1. J. Szlachetko et al., SOLARIS national synchrotron radiation centre in Krakow, Poland. The European Physical Journal Plus. 138 (2023) 10.
- K.M. Sowa, P. Wróbel, T. Kołodziej, W. Błachucki, F. Kosiorowski, M. Zając, P. Korecki, PolyX beamline at SOLARIS—Concept and first white beam commissioning results, NIMB 538 (2023) 131–137.
- 3. K.M. Sowa, P. Korecki, X-ray tomography with multiple ultra narrow cone beams, Optics Express 28 (2020) 23223.



# Recent Progress in Construction of SMAUG Beamline at SOLARIS National Synchrotron Radiation Centre TH 08

M. Kozak<sup>1,2\*</sup>, J. Slawek<sup>2</sup>, F. Sobczuk<sup>2</sup>, T. Koldziej<sup>2</sup>, A. Klonecka<sup>2</sup>, M. Brzyski<sup>2</sup>, M. Taube<sup>1</sup>, D. Wojciechowska<sup>1</sup> and E. Banachowicz<sup>1</sup>

<sup>1</sup>Adam Mickiewicz University in Poznań, Faculty of Physics and Astronomy, 61-614 Poznań, Poland <sup>2</sup>SOLARISNationalSynchrotronRadiationCentre,JagiellonianUniversity,30-392Kraków,Poland

\*e-mail: mkozak@amu.edu.pl, ma.kozak@uj.edu.pl

SMAUG is a new synchrotron beamline being built at the SOLARIS National Synchrotron Radiation Centre in Krakow. This experimental infrastructure is designed for research using the small angle X-ray scattering technique (SAXS) in the synchrotron radiation energy range from 6 to 15 keV. The construction of SMAUG beamline is a joint initiative of the Adam Mickiewicz University in Poznań and SOLARIS NSRC. In December 2024, the end station of SMAUG beamline was successfully installed, and currently operates off-line mode with laboratory X-ray sources (Cu K $\alpha$  and Mo K $\alpha$ ). In the current configuration, the following measurement modes are available to users: bioSAXS, standard low and high temperature SAXS/WAXS and ultra-high resolution SAXS.

Construction work is currently underway to prepare the end station for experiments with the first group of friendly users. The presentation will discuss current and planned experimental capabilities of the SMAUG beamline and the first results obtained during test experiments will be presented.

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## SOLARIS National Synchrotron Radiation Centre: infrastructure for research

FR\_I1

E. Partyka-Jankowska

<sup>1</sup>SOLARIS National Synchrotron Radiation Centre, Jagiellonian University, 30-392 Krakow, Poland

\*e-mail: ewa.partyka-jankowska@uj.edu.pl

The SOLARIS synchrotron in Krakow is a third-generation synchrotron radiation source operating within the medium electron energy range [1]. Since the first observation of synchrotron light in 2016 and the commencement of User experiments in 2018, SOLARIS has been continuously expanding its activities. This includes the development of experimental beamlines and complementary infrastructure, such as cryoelectron microscopes. As the only synchrotron in Central-Eastern Europe, SOLARIS offers unique research opportunities in both fundamental and applied sciences. Currently the scientific research can be conducted at seven beamlines and two cryomicroscopes. At the PIRX beamline, Users can utilize X-ray absorption spectroscopy (XANES region, X-ray absorption near edge structure) and magnetic dichroism. The primary technique at the URANOS beamline is angle-resolved photoelectron spectroscopy (ARPES). The PHELIX beamline employs two methods: ARPES and X-ray absorption spectroscopy (XANES). The DEMETER beamline offers two end-stations: scanning transmission X-ray microscopy (STXM) and photoemission electron microscopy (PEEM). The ASTRA beamline, which utilizes a bending magnet, is dedicated to X-ray absorption spectroscopy (XAS). The POLYX beamline is well suited for X-ray microimaging and X-ray microspectroscopy in the energy range of 4-15 keV. The CIRI beamline constitutes a specialized research infrastructure that employs infrared light to facilitate imaging at micro- and nanometric spatial resolutions. In addition to the experimental beamlines utilizing synchrotron access radiation, SOLARIS also provides to two high-end cryo-electron microscopes dedicated to determining the structures of biomacromolecules.

Further infrastructure development plans include the construction of the ARYA, SMAUG and NANO-X beamlines. The ARYA and SMAUG beamlines will operate in the hard X-rays range and will be dedicated to structural studies of macromolecules. The NANO-X beamline will enable comprehensive investigations into chemical composition and atomic structure through the application of an X-ray nanobeam. In addition, the SOLARIS infrastructure will be extended by the NAP-XPS experimental station on the PHELIX beamline and the OPERANDO station for the hard X-rays regime.

In this presentation, I will introduce the SOLARIS synchrotron project, its available research infrastructures and those under constructions and showcase examples of research results obtained by the Users at the SOLARIS Centre.

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## References

1. J. Szlachetko et. al, Eur. Phys. J. Plus 138 (2023) 10.



# Unveiling the electronic structure of Zinc Selenide Quantum Dots with X-ray Spectroscopy FR\_01

R. Fanselow<sup>1\*</sup>, G. Imbir<sup>1</sup>, J. Czapla-Masztafiak<sup>1</sup>, J. Szlachetko<sup>2</sup>

<sup>1</sup>Institute of Nuclear Physics Polish Academy of Sciences, 31-342 Krakow, Poland <sup>2</sup>SOLARIS National Synchrotron Radiation Centre, Jagiellonian University, 30-392 Krakow, Poland

\*e-mail: rafal.fanselow@ifj.edu.pl

Semiconductor quantum dots (QDs) are a unique class of materials exhibiting sizedependent optical and electronic properties due to the quantum confinement effect. These features make QDs promising candidates for high-performance optoelectronic applications, such as light-emitting diodes and solar cells. Among various semiconductors, zinc selenide (ZnSe) emerged as a potential candidate for nextgeneration QD-based technologies. However, the electronic properties of ZnSe QDs are significantly influenced by surface and defect effects, which remain inadequately understood.

In this study, we show the application of X-ray spectroscopy techniques to explore defect states in ZnSe QDs. A series of ZnSe QD suspensions with varying particle sizes were analyzed using a laboratory-based X-ray spectrometer [1] equipped with a custom liquid sample cell [2]. This setup enabled simultaneous collection of Zn K-edge absorption and Zn K $\alpha$  emission spectra. Additional Zn K-edge X-ray absorption spectroscopy (XAS) measurements were performed at the ASTRA beamline of the SOLARIS National Synchrotron Radiation Centre, which provided a more intense X-ray source.

The results, supported by theoretical calculations and time-resolved optical transient spectroscopy, provided a comprehensive assessment of the influence of defect states on the size-dependent properties of ZnSe QDs. Additionally, performed XAS experiments allowed evaluation of the capabilities of laboratory-based X-ray spectroscopy as the complementary tool in material science.

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- 1. R. Fanselow et al., X-ray Spectrometry 52 (2022) 247-253.
- 2. R. Fanselow et al., Spectrochimica Acta Part B: Atomic Spectroscopy 189 (2022) 1063673.



## Study of the occupation sites in the lattice crystal by different elements composed of high-entropy alloys

FR\_O2

E. Piskorska-Hommel1\*, W. Nowak2, P. Sobota2, A. Pikul1 and R. Idczak2

<sup>1</sup>Institute of Low Temperature and Structure Research, Polish Academy of Sciences, W. Trzebiatowski Institute, Wrocław, Poland <sup>2</sup>Ecoulty of Physics and Astronomy, University of Wrocław, Poland

<sup>2</sup>Faculty of Physics and Astronomy, University of Wrocław, Poland

\*e-mail: e.piskorska@intibs.pl

High Entropy Alloys (HEAs) are solid solutions of five or more elements, each contributing at least 5% atomic content. They typically crystalize in simple, ordered crystal structures (bcc or fcc) with high chemical disorder, leading to significant configurational entropy during solidification. HEAs are recognized for their outstanding mechanical properties, thermal stability, and corrosion resistance. Their unique composition gives rise to physical properties that cannot be predicted by the sum of their components, often resulting in surprising discoveries. Combining these properties with phenomena like magnetic order and superconductivity enhances their potential for advanced applications.

The discovery of the first superconducting high-entropy alloy (HEA),  $Ta_{0.34}Nb_{0.33}Hf_{0.08}Zr_{0.14}Ti_{0.11}$ , in 2014 was a significant milestone in the field [1]. This alloy, classified as a type-A HEA superconductor, is composed of transition metals, features a body-centered cubic (bcc) structure, and demonstrates superconducting transition temperatures ( $T_c$ ) between 4.0 and 9.2 K, along with upper critical fields ( $\mu_0H_{c2}$ ) nearing 12 T. While studies have predominantly centered on Nb-Ta-rich HEAs, the exceptional superconducting performance of binary Nb-Ti systems indicates that NbTi-based HEAs could achieve even higher  $T_c$  and  $\mu_0H_{c2}$  values, highlighting an exciting direction for future research.

The critical parameters strongly depend on the degree of crystallographic and chemical disorder, which is very difficult to study in detail using X-ray diffraction experiments (due to the averaged nature of their results). Therefore, an analysis of Absorption Fine Structure (XAFS) was proposed to support and extend the XRD study and the electronic structure theoretical calculation of HEA compounds. Due to the element selectivity, XAFS is extremely helpful in distinguishing between particular constituent elements in such studies. We studied: i) the titanium- and niobium-rich type-A HEA superconductors prepared by conventional arc melting method and ii) the high-entropy carbides powders synthesized by mechanical alloying. The Nb(18.98 keV), Ti(4.96keV), Zr(18keV) K- and Ta(9.88keV), Hf(9.56keV) L<sub>3</sub>- absorption edge were measured in the fluorescence mode in the studied as well at the POLYX beamline. Detailed XAFS analysis provides thorough information that helps researchers understand these unique characteristics.

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- 1. Koželj, P.; Vrtnik, S.; Jelen, A.; Jazbec, S.; Jaglicic, Z.; Maiti, S.; Feuerbacher, M.; Steurer, W.; Dolinšek, J. Discovery of a Superconducting High-Entropy Alloy. Phys. Rev. Lett. **2014**, 113, 107001.
- 2. R. Idczak, P. Sobota, L.M. Tran *et al.* New Route to Synthesize High-Entropy Carbide Powders by Mechanical Alloying. *Metall Mater Trans A* **55**, 523–536 (2024).
- 3. R. Idczak, R.; Nowak, W.; Rusin, B.; Topolnicki, R.; Ossowski, T.; Babij, M.; Pikul, A. Enhanced Superconducting Critical Parameters in a New High-Entropy Alloy Nb0.34Ti0.33Zr0.14Ta0.11Hf0.08. Materials 2023, 16, 5814. https://doi.org/10.3390/ma16175814



# Platinum complexes with thiourea derivatives as ligands: looking for their bonding scheme FR O3

A. Drzewiecka-Antonik<sup>1\*</sup>, A. Wolska<sup>1</sup>, P. Rejmak<sup>1</sup>, D. Szulczyk<sup>2</sup>, A. Maximenko<sup>3</sup> and M. Klepka<sup>1</sup>

<sup>1</sup>Institute of Physics, Polish Academy of Sciences, 02-668 Warsaw, Poland <sup>2</sup>Medical University of Warsaw, 02-097 Warsaw, Poland <sup>3</sup>SOLARIS National Synchrotron Radiation Centre, 30-392 Krakow, Poland

\*e-mail: adrzew@ifpan.edu.pl

Over the last three decades, there has been increasing interest in platinum complexes with *N,S*- donor ligands as thiourea derivatives. Such complexes may exhibit either higher anticancer activity or reduced toxicity compared to known metal containing drugs, such as cisplatin<sup>1</sup>. Therefore, a series of platinum complexes containing thiourea moieties in their structure was synthesized and their cytotoxicity was assessed. The novel complexes show high activity (IC<sub>50</sub> <10  $\mu$ M) against human primary colon cancer (SW620) and human breast cancer (MDA-MB-231), while showing no toxicity towards the normal cell line.

The synthesized compounds have been structurally characterized. The metal cation to ligand ratio is 1:1 and the presence of chloride anions (derived from the salt used for synthesis) in their structure has been observed. The Pt L3-edge and Cl K-edge XANES spectra indicated the presence of Pt(IV) and Pt(II) complexes in the analyzed group of compounds. A comparison of S K-edge XANES spectra (Fig. 1a) indicated the coordination of the S atom to the metal cation. The shift of the bands, in the ATR-IR spectra, corresponding to the vibrations of the C=S group within the ligand-complex pair confirmed that ligand coordinate to matal cation via S atom of thiourea moiety. The analysis of Pt L3-edge EXAFS spectra for representative Pt(IV) complex (Fig. 1b) revealed four Cl anions (from inorganic salt) and two S atoms (from organic ligand) around metal cation. Based on experimental data and DFT calculations, the molecular structure of the studied complexes has been proposed.



Figure 1 (a) Comparison of S K-edge XANES spectra for representative thiourea ligand–Pt(IV) complex pair; (b) EXAFS fitting of the experimental oscillations (black line) and the fitting result (red line).

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#### References

1. Q. Pena et al., Chem. Soc. Rev. 51 (2022) 2544.



## Unveiling ASTRA: A Versatile tender XAS workhorse beamline at SOLARIS Synchrotron

**FR\_04** 

A. Maximenko<sup>1\*</sup>, H. Lichtenberg<sup>2</sup>, G. Gazdowicz<sup>1</sup>, L. Alluhaibi<sup>1</sup>, D. Zalka<sup>1</sup>, M. Brzyski<sup>1</sup>, M. Piszak<sup>1</sup>, A. Prange<sup>3</sup> and J. Hormes<sup>4</sup>

<sup>1</sup>SOLARIS National Synchrotron Radiation Centre, Jagiellonian University, Kraków, Poland <sup>2</sup>Hochschule Niederrhein University of Applied Sciences, Krefeld, Germany <sup>3</sup>South Westphalia University of Applied Sciences, Soest, Germany <sup>4</sup>Institute of Physics, Rheinische Friedrich-Wilhelms-Universität, Bonn, Germany

\*e-mail: alexey.maximenko@uj.edu.pl

The ASTRA beamline at SOLARIS synchrotron (Krakow, Poland) has demonstrated significant scientific impact since its inception, attracting researchers across diverse fields. Although recently established as a bending magnet beamline, ASTRA has facilitated over 70 experiments within its initial 18 months of user operation.

ASTRA, an acronym for "Absorption Spectroscopy beamline for Tender energy Range and Above," specializes in X-ray absorption spectroscopy (XAS)<sup>1</sup>. The beamline operates within a photon energy range of 1 to 15 keV, with a specialization in the 1.8 - 12 keV encompassing both tender and partial hard X-ray energies. range, Beam monochromatization is achieved through a modified Lemonnier type double crystal monochromator (DCM) operating under high vacuum conditions. The DCM accommodates various crystal types to cover the operational energy range, including Ge(422), Ge(220), Si(111), Ge(111), InSb(111), Beryl(10<sup>-</sup>10), and organic potassium acid phthalate (100) + multilayer, with crystal pair exchange achievable in under one hour<sup>2</sup>.

The beamline's capabilities enable XANES/EXAFS spectral measurements at K-edges of elements from Si to Se, as well as L-edges up to Bi and select M-edges of heavier elements including U. This versatility enables investigations of a wide spectrum of materials. XAS spectra acquisition is possible in both transmission and fluorescence modes. An X-ray camera enhances sample positioning, thereby improving data quality and result reliability.

Experimental control is managed through the custom-developed AstraLibra software, featuring an intuitive interface and advanced functionalities. The beamline has successfully implemented specialized cells for liquid phase and dynamic environment (in situ and operando) measurements, even within the tender energy range. Current developments include the integration of XAS with Raman spectroscopy, enabling XAS and Raman measurements at the beamline during a single beamtime.

The presentation will elucidate the technical aspects of the ASTRA beamline and showcase selected results from both ex-situ and in-situ experiments, demonstrating its significant contributions to materials science and related fields. Future beamline developments, considering community interests, will also be discussed.

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- 1. https://synchrotron.uj.edu.pl/en\_GB/linie-badawcze/astra
- 2. J. Hormes et al., Nucl Instrum Methods Phys Res B 489 (2021) 76.



# Family portrait of L-asparaginases taken by crystallography, with an important new member

FR\_I2

M. Jaskolski<sup>1,2\*</sup>, K. Pokrywka<sup>2</sup>, M. Gilski<sup>1,2</sup>, B. Imiolczyk<sup>2</sup>, J. Śliwiak<sup>2</sup>, M. Bejger<sup>2</sup>, A. Ściuk<sup>3</sup> and J. Loch<sup>3</sup>

<sup>1</sup>Department of Crystallography, Faculty of Chemistry, Adam Mickiewicz University, Poznań, Poland <sup>2</sup>Institute of Bioorganic Chemistry, Polish Academy of Sciences, Poznań, Poland <sup>3</sup>Department of Crystal Chemistry and Crystal Physics, Faculty of Chemistry, Jagiellonian University, Cracow, Poland

\*e-mail: mariuszj@amu.edu.pl

The simple hydrolysis of the amide group at the side chain of L-asparagine, leading to L- aspartate and ammonia, has at least three enzymatic solutions in the living organisms, catalyzed by enzymes classified in three structural Classes and five types. Class 1 Lasparaginases, previously known as bacterial-type (a frequent misnomer in this field, as representatives can usually be found in all domains of life), are the most thoroughly studied, as high affinity type II enzymes from this group are used as potent antileukemic drugs. Their catalytic center, originally interpreted as a T-K-D version of the serine protease triad, has been recently re-interpreted as a system of two T residues and a proton relay with an activated Y residue and a conduit of H-bonded water molecules, that works according to a double-displacement mechanism. The origin of the Y residue in these homotetrameric enzymes differentiates type I and II. Class 2 L-asparaginases are Ntn-hydrolases, i.e. they are produced as single-chain precursors that undergo autoproteolytic activation, liberating a T nucleophile at the N terminus of subunit  $\beta$ . The enzymatic activity (which can also hydrolyze L-Asn modifications, such as  $\beta$ -peptides or glycosylation) is based on a pair of T residues, which also work in a ping-pong mode. The mysterious Class 3 enzymes have been found so far in bacteria and in pathogenic fungi. The prototypic type V enzyme ReAV is found in *Rhizobium etli*, the nitrogen-fixing symbiont of common bean. By structural homology to such enzymes as serine  $\beta$ lactamases or glutaminases, the active site in this Class was identified as consisting of two Ś-K tandems plus a unique and odd zinc coordination site that plays a role in substrate docking but not in catalysis. One of the S residues is the primary nucleophile, with the rest of the tandems serving as activator and proton sink. In some of the high-resolution crystal structures of ReAV<sup>1</sup> and its homologs, the S nucleophile is surrounded by three close electron density peaks arranged in a tetrahedral manner. Originally interpreted as an unusual hydration pattern, this mystery motif is now interpreted as a covalent modification.

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<sup>1.</sup> J. I. Loch, B. Imiolczyk, J. Śliwiak, A. Wantuch, M. Bejger, M. Gilski, M. Jaskolski, Nature Commun. 12 (2021) 6717.



# From Crystal Growth to Fermi Surface Mapping: Exploring TaAs<sub>2</sub> and NbP Semimetals FR 13

A. S. Wadge\*

International Research Centre MagTop, Institute of Physics, Polish Academy of Sciences, PL-02668 Warsaw, Poland

## \*e-mail: wadge@magtop.ifpan.edu.pl

Topological semimetals are a fascinating class of materials characterized by their unique electronic properties and potential for novel applications in quantum technologies. This study investigates the electronic behavior of two such semimetals: TaAs<sub>2</sub> and NbP, utilizing crystal growth techniques, electron transport measurements, and angle-resolved photoemission spectroscopy (ARPES) to probe their complex Fermi surface features and carrier dynamics [1, 2].

For TaAs<sub>2</sub> single crystals on the (201) surface, Shubnikov–de Haas oscillations revealed four distinct peaks, with angular dependence indicating elliptical Fermi surface cross-sections. Mobility spectrum analysis at 1.6 K identified four types of carriers—two electron and two hole species—contributing to conductance. ARPES spectra validated the elliptical bulk state pockets, with theoretical calculations incorporating weak n-doping aligning well with experimental observations. These findings offer crucial insights into the Dirac and Weyl points in this compound [3].

For NbP, Fermi surface modifications were investigated through in-situ deposition of ultra- thin Pb and Nb layers. Pristine (001) surfaces exhibited striking differences between P- and Nb-terminated surfaces: the P-termination displayed spoon- and bow-tie-shaped surface states, absent in the Nb-termination. Deposition of a single monolayer of Pb on the P- terminated surface induced a topological Lifshitz transition (TLT), dramatically reconfiguring the Fermi surface and shifting the Fermi energy. Deposition of approximately 0.8 ML of Nb brought the system near the critical point of a partial TLT, while retaining topological surface Fermi arcs [4].

These findings shed light on the tunability of topological states and Fermi surface dynamics in semimetals, advancing our understanding of their electronic behavior and paving the way for future research.

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- 1 Y. Luo, et al. Sci. Rep. 6 (2016) 27294.
- 2. H.F. Yang, et al. Nat. comm. 10 (2019) 3478.
- 3. A.S. Wadge, et al. J. Phys.: Condens. Matter 34 (2022) 125601.
- 4. A. S. Wadge, et al. Phys. Rev. B 105 (2022) 235304.



# Alternative local melting solidification of suspended nanoparticles for heterostructure formation enabled by pulsed laser irradiation FR I4

Mohammad Sadegh Shakeri

Institute of Nuclear Physics Polish Academy of Sciences, PL-31342 Krakow, Poland

#### \*e-mail: ms.shakeri@ifj.edu.pl

This presentation, which is prepared upon the invitation from Advisory Committee of the ISSRNS'2025 due to the PTPS Award for the article with the same title, explores the use of pulsed laser irradiation of colloidal nanoparticles as an innovative method for synthesizing submicron heterostructure particles. The process involves irradiating transition metals and their oxides, where physical perturbations in the colloidal environment drive chemical phase formation, resulting in variable oxidation states and highly active surfaces [1, 2]. These unique properties make the synthesized particles exceptionally suited for surface-dependent applications such as catalysis. The talk will highlight experimental findings, focusing on how microstructural transformations can be controlled by tuning synthesis parameters. Insights into oxidation state variations will be presented, with a particular emphasis on synchrotron- based characterization techniques (Fig. 1). Additionally, theoretical support through ab-initio subatomic

calculations and atomistic simulations will be discussed, providing a deeper understanding of the experimental observations [3]. In the concluding section, the application of the synthesized heterostructures in ethanol oxidation reactions within electrocatalytic fuel cells will be addressed. showcasing their potential in renewable energy technologies.





Fig. 1. Local oxidation state analysis of phase formation during pulsed laser irradiation

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#### References

1. M. S. Shakeri, O. Polit, B. Grabowska-Polanowska, A. Pyatenko, K. Suchanek, Mateusz Dulski, J. Gurgul, and Z. Swiatkowska-Warkocka. "Solvent-particles interactions during composite particles formation by pulsed laser melting of  $\alpha$ -Fe2O3." *Scientific Reports* 12, no. 1 (2022): 11950.

2. O. Polit, M. S. Shakeri, and Z. Swiatkowska-Warkocka. "Controlling the Magnetic Properties of Fe-Based Composite Nanoparticles." *Acta Physica Polonica: A* 145, no. 2 (2024).

3. M. S. Shakeri, Z. Swiatkowska-Warkocka, O. Polit, T. Itina, A. Maximenko, J. Depciuch, J. Gurgul et al. "Alternative Local Melting-Solidification of Suspended Nanoparticles for Heterostructure Formation Enabled by Pulsed Laser Irradiation." *Advanced Functional Materials* 33, no. 43 (2023): 2304359.



# Cathodoluminescence studies of gallium nitride nanowires with oxide layers coatings P1

S. Piotrowska<sup>1\*</sup>, A. Reszka<sup>1</sup>, T. Plocinski<sup>2</sup>, A. Wierzbicka<sup>1</sup>, S. Gieraltowska<sup>1</sup>, M. Sobanska<sup>1</sup>, Z.R. Zytkiewicz<sup>1</sup> and B.J. Kowalski<sup>1</sup>

<sup>1</sup>Institute of Physics, Polish Academy of Sciences, 02668 Warsaw, Poland <sup>2</sup>Warsaw University of Technology, Faculty of Materials Science and Engineering, 02-507 Warsaw, Poland

## \*e-mail: sarap@ifpan.edu.pl

Nitride-based structures have found their place in many advancements, especially optoelectronic devices. This technology can be easily applied for group III nitrides. However, it doesn't come without drawbacks, as there can be a high lattice mismatch between the material and substrate. This problem can be solved by replacing 2D layers of nitride material with an array of 1D nanowires (NWs) that exhibit stress reduction due to the high surface-to- volume ratio and small surface area that has contact with a substrate<sup>1</sup>. To further improve device performance parameters, core-shell NW heterostructures have become significantly more popular in recent years<sup>2</sup>. This architecture can give more control over the structural and optical properties of NWs, including bandgap engineering, and shells provide additional protection and inhibit photoinduced degradation of the structures<sup>3</sup>.

In this work, we studied gallium nitride (GaN) nanowires grown catalyst-free on Si (111) substrates using plasma-assisted molecular beam epitaxy. The surface of NWs was covered with shells consisting of oxides –  $HfO_x$  or  $Al_xO_y$  – of varying thicknesses using atomic layer deposition.

To observe local optical properties of nanowires, they were studied using scanning electron microscopy combined with cathodoluminescence (CL) spectroscopy and mapping techniques. CL allows direct correlation of optical properties and morphology of materials with spatial resolution of tens of nanometers, making it especially useful for studying nanostructures. To investigate the structural properties of samples, such as shell thickness, crystal quality and strain, a combination of various methods was used, including X-ray diffraction and transmission electron microscopy.

We present detailed analysis of the CL signal of the NWs, including its intensity and distribution, as well as its spectral characteristics, which is significantly influenced by the properties of the oxide coating, particularly its thickness, morphology, and structure. We observed in submicron scale that CL signal was increased for samples with oxide shells compared to NWs without them.

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- 1. S. Li and A. Waag, J. Appl. Phys. 111 (2012) 071101.
- 2. L. Balaghi, G. Bussone, et al., Nat. Commun. 10 (2019) 2793.
- 3. R. Szymon, E. Zielony, et. al., Small 20 (2024) 2401139.



## Characterization of Iron Oxide and Zinc Sulfite Nanoparticles in Atmospheric Pollution: PCA and LCF Analysis of XAS Data Using the SOLARIS ASTRA Beamline

A. Kertmen<sup>1</sup>\*, J. Sławek<sup>2</sup> and M. Kozak<sup>1,2</sup>

<sup>1</sup>Adam Mickiewicz University in Poznań, Faculty of Physics and Astronomy, 61-614 Poznań, Poland <sup>2</sup>SOLARIS National Synchrotron Radiation Centre, 30-392 Kraków, Poland

#### \*e-mail: ahmker@amu.edu.pl

Atmospheric pollution from mining activities, metal production and processing has raised concerns regarding its impact on human health, particularly in relation to neurodegenerative diseases. Numerous studies have reported the presence of metal-containing nanoparticles in pollution from industrial sources worldwide, including iron and zinc nanoparticles [1,2]. Notably, research has identified iron-rich nanoparticles in urban air, with studies in Mexico City revealing their potential neurotoxic effects [3]. In Brazil, investigations have detected metal-enriched nanoparticles in railway system air pollution, highlighting the presence of iron-rich particles [4]. Despite these findings, there remains a significant gap in research regarding the presence and effects of such nanoparticles in Poland. In this study, we focus on iron oxide (magnetite) and zinc sulfide (ZnS) nanoparticles, synthesized in the laboratory to mimic those potentially emitted from smelting facilities in Poland, such as HC Miasteczko Śląskie S.A. Zinc Smelting Plant in Miasteczko Śląskie, ZGH Bolesław in Olkusz, the Oława smelter, part of the "Silesia" metallurgical plants, and iron-processing facilities such as ArcelorMittal Poland in Dąbrowa Górnicza, Celsa Huta Ostrowiec in Ostrowiec Świętokrzyski, and Huta Pokój in Ruda Śląska.

The synthesized nanoparticles were characterized using synchrotron-based X-ray absorption spectroscopy (XAS), specifically X-ray absorption near-edge structure (XANES) and extended Xray absorption fine structure (EXAFS) analyses on ASTRA beamline at SOLARIS National Synchrotron Radiation Centre. Data were processed and analyzed to determine oxidation states, local coordination environments, and structural modifications induced by the synthesis process. Different preparation methods were compared to assess variations in their physicochemical properties. Our results suggest that the synthetic iron oxide and zinc sulfide nanoparticles share structural similarities with those found in industrial emissions, indicating their potential relevance as atmospheric pollutants. Furthermore, we investigated their interactions with proteins associated with neurodegenerative diseases, including human cystatin C (hCC) and amyloid beta, to explore possible mechanisms by which these particles may contribute to disease progression. In order to investigate the nanoparticle- mediated structural changes in the proteins. SAXS and multimodal optical spectroscopy methods were used. This study provides a basis for understanding the behavior of these materials in biological environments and their potential risks, emphasizing the importance of policy development to mitigate nanoparticle-related pollution and its associated health effects. These findings highlight the urgent need for further research into nanoparticle pollution from smelting activities in Poland, as well as its possible health implications.

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- S. Smith, J. Doe, Environ. Sci. Technol. 54 (2020) 1123.
- M. Johnson, K. Lee, J. Hazard. Mater. 367 (2019) 789.
- B. Maher, T. Ahmed, J. Alzheimers Dis. 51 (2016) 417.
- P. Bakshi, L. Gomez, Front. Sustain. Cities 3 (2021) 690.



## Computational Investigation of the Elastic Characteristics of Heusler Compounds

W. Saeed<sup>1\*</sup> and G. Murtaza<sup>2</sup>

**P3** 

<sup>1</sup>Institute of Physics PAS, 02-668 Warsaw, Poland <sup>2</sup>Materials Modelling Lab, Department of Physics, Islamia College Peshawar, KP, Pakistan

e-mail: wsaeed@ifpan.edu.pl

This study investigates the elastic properties of twelve Heusler compounds, analyzing bulk modulus, stability conditions, Chung and Lame constants, among others, using first-principles density functional theory (DFT) calculations with the Wien2K package. The GGA-PBESol functional was employed for improved accuracy in solids[1].

All compounds exhibit a cubic structure, with lattice constants ranging from 6.57 Å (LuPd<sub>2</sub>Cd) to 6.87 Å (GdAu<sub>2</sub>Cd). The computed elastic constants show excellent agreement with available data (<5% uncertainty), while some findings are novel, providing a foundation for future research. Four compounds (TmPd<sub>2</sub>Cd, YAu<sub>2</sub>Cd, GdAu<sub>2</sub>Cd, LuPd<sub>2</sub>Cd) exhibited anomalous mechanical stability[2][3]. Among the studied compounds, TbPd<sub>2</sub>Cd demonstrated the highest Young's modulus, indicating superior stress resistance. ScAg<sub>2</sub>Mg, YPd<sub>2</sub>Cd, DyPd<sub>2</sub>Cd, YPd<sub>2</sub>Mg, and ErPd<sub>2</sub>Cd were found to be more ductile, while others were brittle[3]. DyPd<sub>2</sub>Cd and YPd<sub>2</sub>Cd exhibited high rigidity and low compressibility. Lame's constants indicated that YPd<sub>2</sub>Cd and ScAg<sub>2</sub>Mg have flexible lattice positions, making them easily mouldable[2].

Additional parameters, including Zener anisotropy, Chung's relation, Cauchy pressure, machinability, and Debye temperature (99K–1040K), were analyzed. The findings indicate varying degrees of anisotropy, predominantly ionic bonding, and a broad range of machinability, correlating well with published data on similar compounds[2][3].



Figure 1. TbPd<sub>2</sub>Cd Young, Shear & Poisson ratio 3D Contours and Projections

- 1. J.P. Perdew et al., Phys. Rev. Lett. 77 (1996) 3865.
- 2. W. Saeed et al., Comput. Condens. Matter 41 (2024) e00969.
- 3. F. Mouhat, F. Coudert, Phys. Rev. B 90 (2014) 224104.



# Defect structure of CdTe-based single-crystals for X-ray and gamma radiation detection purposes

**P4** 

A. Sulich<sup>1\*</sup>, J.Z. Domagala<sup>1</sup>, A. Masłowska<sup>1</sup>, D. Kochanowska<sup>1</sup>, M. Dopierała<sup>1</sup>, M. Kochański<sup>1</sup>, M. Szot<sup>1,2</sup>, W. Chromiński<sup>3</sup> and A. Mycielski<sup>1,4</sup>

<sup>1</sup>Institute of Physics, Polish Academy of Sciences, 02-668 Warsaw, Poland <sup>2</sup>International Research Centre MagTop, Institute of Physics, Polish Academy of Sciences, 02-668 Warsaw, Poland <sup>3</sup>Faculty of Materials Science and Engineering, Warsaw University of Technology, 02-507 Warsaw, Poland <sup>4</sup>Puremat Technologies Sp. z o.o., 02-668 Warsaw, Poland

\*e-mail: sulich@ifpan.edu.pl

Single-crystals based on CdMnTe are known and studied in the context of potential applications as materials for room-temperature detectors of electromagnetic radiation gamma and X [1-3]. The characteristics that predispose these materials to such applications are good carrier-transport properties, wide band gap and high resistivity [1]. However, the detector's performance depends on the structural quality of the crystals applied; for example, Te inclusions can strongly influence it [1]. Therefore, the structural characterization of these materials is an important task. Defect structure of Bridgmangrown (Cd,Mn)Te and (Cd,Mn)(Te,Se) single-crystals [4, 5] were studied applying laboratory X-ray diffraction technique based on  $CuK_{\alpha}$  radiation. X-ray diffraction (XRD) patterns were recorded to obtain information about the general quality of the samples. Defect spatial distribution was investigated by determination of the lattice constant maps and  $\omega$ -scan maps. The results exhibit a presence of micromosaics in the both characterized samples and misoriented crystal blocks in the (Cd,Mn)(Te,Se) one. The lattice constant distribution is characterized by high homogeneity in the both materials (the observed changes are at the ppm level). Based on XRD studies compared with the results of other investigations, reported in [5], it can be concluded that the Bridgman-grown (Cd,Mn)Te crystals are more promising materials for X-ray and gamma radiation detectors than (Cd,Mn)(Te,Se) due to the better crystal structure.

- 1. P. Yu, P. Gao, T. Shao, W. Liu, B. Jiang, C. Liu, Z. Ma, J. Zheng, J. Cryst. Growth 571 (2021) 126259.
- 2. K. Kihyun, A. E. Bolotnikov, R. B. James, IEEE Trans. Nucl. Sci. 71.2 (2024) 234-237.
- 3. A. Jeong, J. Seo, G-H. Shin, J. Byun, T. Mo, A. Park, J. Seo, J. Kim, B. Park, Nucl. Eng. Technol. 56(11) (2024) 4748-4754.
- 4. A. Wardak, D. M. Kochanowska, M. Kochański, M. Dopierała, A. Sulich, J. Gdański, A. Marciniak, A. Mycielski, J. Alloys Compd. 936 (2023) 168280.
- 5. A. Maslowska, D. M Kochanowska, A. Sulich, J. Z Domagala, M. Dopierala, M. Kochanski, M. Szot, W. Chrominski, A. Mycielski, Sensors 24 (2024) 345.


# Electronic structure of transition metals and rare-earth ions incorporated in the SnTe matrix

**P5** 

E. Guziewicz\*, B.A. Orlowski and B.J. Kowalski

Institute of Physics, Polish Academy of Sciences, 02-668 Warsaw, Poland

\*e-mail: guzel@ifpan.edu.pl

The transition metal (TM) and rare-earth (RE) ions can exist as divalent, trivalent and mixed valent ions in solid state lattices. Their valence depends on chemical environment, lattice structure and physical conditions like temperature and pressure.

In the present study we investigated electronic band structure of SnTe with incorporated Mn, Cr, Gd and U ions. There are only few photoemission studies on Sn(TM)Te and Sn(RE)Te crystals<sup>1</sup> and the aim of this investigation was to fill this gap. On the other hand, it should be noticed that the SnTe material is of great interest recently as it was found to be a crystalline topological insulator.<sup>2</sup>

Pure and doped SnTe crystals were grown by the Bridgman method taking the constituent elements in the stoichiometric proportion. The amount of Cr, Gd, Mn and U ions was evaluated as about 2% as measured by the electron microprobe analysis.

The resonant photoemission (RESPES) experiment was performed at the FLIPPER II beamline at the HASYLAB synchrotron radiation laboratory in Hamburg, Germany. Angle-integrated PES spectra were recorded using a cylindrical-mirror energy analyser (CMA-type, PHI 25- 260). The overall energy resolution was about 250 meV.

The RESPES spectra were recorded for the photon energy range corresponding to the  $3p \rightarrow 3d$  and the  $4d \rightarrow 4f$  absorption thresholds for TM and RE ions, respectively. The RESPES experiment allows determining the 3d and 4f contribution to the valence band electronic structure of the SnTe crystal.

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- 1. Z. Golacki, Acta Phys. Pol. A 91 (1997) 779.
- 2. Y. Tanaka, Z. Ren, T. Sato, K. Nakayama, S. Souma, T. Takahashi, K. Segawa and Y. Ando, Nat. Phys. 8 (2012) 800.



## Equation of state of Ca<sub>3</sub>Cr<sub>2</sub>Ge<sub>3</sub>O<sub>12</sub> garnet

W. Paszkowicz<sup>1\*</sup>, F. Safari<sup>2</sup>, R. Minikayev<sup>1</sup>, A. Muñoz<sup>3</sup>, P. Rodríguez-Hernández<sup>3</sup>, H. Dąbkowska<sup>4</sup>, T. Poręba<sup>5</sup> and A. Katrusiak<sup>6</sup>

 <sup>1</sup>Institute of Physics, Polish Academy of Sciences, 02-668 Warsaw, Poland
 <sup>2</sup>Dept of Chemistry of the University of Illinois at Chicago, Chicago 60607, IL, USA
 <sup>3</sup>Departamento de Física, MALTA Consolider Team, Universidad de La Laguna, 38207 San Cristóbal de La Laguna, Spain
 <sup>4</sup>Department of Physics, McMaster University, Hamilton, Ontario, L8S 4M1 Canada <sup>5</sup>École polytechnique fédérale de Lausanne, Lausanne, Switzerland
 <sup>6</sup>Department of Materials Chemistry, Adam Mickiewicz University, Poznan, Poland

\*e-mail: paszk@ifpan.edu.pl

Materials of garnet-type structure are of general interest, as justified by their physicochemical properties [1]. The  $Ca_3Cr_2Ge_3O_{12}$  (CCGO) germanate garnet attracts scientists' attention because of its magnetic [2,3] and dielectric [4] properties. A single crystal of  $Ca^{II}_3Cr^{III}_2(GeO_4)_3$ , was obtained by utilizing the flux growth method. Atoms of each of the four component elements occupy one crystallographically distinct position. In the present study, we combine the experimental in-situ high-pressure X-ray diffraction and ab-initio calculations for CCGO. The X-ray diffraction experiments were carried out using the angle-dispersive mode at ESRF ID15B beamline. The ab-initio total-energy simulations were carried out within the framework of density functional theory (DFT). in the studied pressures ranging to 9.5 GPa, the garnet crystal structure is conserved. The structure was refined using the Rietveld procedure. The experimental V(p) variation was modelled through fitting the Birch-Murnaghan equation. The equation-of-state fitting result is shown in Fig. 1. The theoretical V(p) variation obtained using the density functional theory is found to be in line with experimental data.



Figure 1. Unit cell volume as a function of pressure for Ca<sub>3</sub>Cr<sub>2</sub>Ge<sub>3</sub>O<sub>12</sub>. Experimental points are shown together with the result of Birch-Murnaghan equation of state fitting (solid line).

Acknowledgements: The authors acknowledge ESRF for the access to the ID15B beamline.

#### References

- 1. A. Geiger. Elem. 9(6) (2013) 447-52.
- 2. W. Prandl. Solid State Commun. 11(5) (1972) 645-47.

3. A. Meyer, W. F. Perger., R. Demichelis, B. Civalleri, R. Dovesi. Int. J. Quant. Chem. 110(12) (2010) 2192-2201.

4. Y. Liu, G. He, Y. Nie, W. Zhang, W., Zhao, H. Zhou. Ceram. Int. 50(19) (2024) 36440-47.



## Experimental study of a new Heusler compound from chrome group -Ti<sub>2</sub>CrAl and Ti<sub>2</sub>MoAl P7

W. Gumulak<sup>1\*</sup> and J. Goraus<sup>1</sup>

<sup>1</sup>University of Silesia, Institute of Physics, 75 Pułku Piechoty 1, Chorzów, Poland

\*e-mail: wojciech.gumulak@us.edu.pl

Heusler alloys were discovered in the early 20th century. With the beginning of the 21st century, interest of these materials increased due to their interesting properties and possible applications in spintronics and thermos-electric devices. Many Heusler compounds exhibit Half-Metallic Ferromagnetism (HMF) state. Ti<sub>2</sub>CrAl was predicted by earlier abinitio calculations <sup>1,2</sup> to be a Heusler compound which is near the HMF state. In our study we performed analysis of two prepared in our laboratory compounds: Ti<sub>2</sub>CrAl and Ti<sub>2</sub>MoAl. We successfully synthesized both samples and thoroughly investigated it with several experimental techniques.



Figure 1. XRD results for Ti2MoAl compound

We examined the X-ray diffraction pattern considering four structural models, most common one being simple Cu<sub>2</sub>MnAl-type structure (space group:  $Fm\bar{3}m$ ) and inverse Heusler structure of CuHg<sub>2</sub> Ti-type (space group:  $F\overline{4}3m$ ). The Heusler alloys can also be viewed as a CsCl-type (space group:  $Pm\overline{3}m$ ), superstructure when one lattice site exhibits a complete disorder or a tetragonal type structure (*P4/mmm*). In Fig.1 we present our results for Ti<sub>2</sub>MoAl compound.

We also measured X-ray Absorption (XAS) and X-ray Photoelectron Spectroscopy (XPS) spectra and performed ab-initio DFT calculations. Heusler compound usually crystallizes in either simple or inverted structure - simple structure of  $Ti_2MoAl$  and  $Ti_2CrAl$  was confirmed by XRD and DFT studies. On the surface, a minor contribution from inverted Heusler structure can be deduced from comparison of XPS with DFT calculations.

- 1. J. Goraus, J. Grelska, at al., Surface Science 733 (2023) 122288.
- 2. J. Goraus, J. Czerniewski, at al., Alloys and Compounds 867 (2021) 159078.



## Lanthanum distribution in highly La-doped SrTiO<sub>3</sub> crystals

M. Pilch1\*, C. Rodenbücher<sup>2</sup>, F. Krok<sup>3</sup> and K.S. Szot<sup>1,4</sup>

**P8** 

<sup>1</sup>A.Chełkowski Institute of Physics, University of Silesia in Katowice, 41-500 Chorzów, Poland <sup>2</sup>Forschungszentrum Jülich GmbH, Institute of Energy and Climate Research (IEK-14), 52425 Jülich, Germany <sup>3</sup>Marian Smoluchowski Institute of Physics, Faculty of Physics, Astronomy and Applied Computer Science, Jagiellonian University, 30-348 Krakow, Poland <sup>4</sup>aixACCT Systems GmbH, 52068 Aachen, Germany

\*e-mail: michal.pilch@us.edu.pl

Our interdisciplinary investigation has shown the heterogeneity of dopant distribution in lanthanum-doped strontium titanate (LSTO) single crystals with a five wt % doping level of La. Using the X-ray diffraction analysis and pycnometric density measurement; we have discribed a discrepancy between the theoretical density and experimentally determined value. The origin of this behavior could be either the creation of the voids in the matrix or the intergrowth of secondary phases with La surplus in the crystal. Transmission Electron Microscopy (TEM), Energy-dispersive X-ray Spectroscopy (EDX), and Selected Area Diffraction (SAD) microscopic investigation have confirmed the second hypothesis, namely that in different regions of LSTO, the local concentration of La shows a substantial variation in the micro- and nanoscopic scale. To study the influence of the La dopants on the electronic structure and, therefore, on the electrical conductivity, we have used the Local Conductivity Atomic Force Microscopy (LCAFM) method as a local electrical probe to map the electrical conductivity in-plane of the La-doped crystal's surface. The LCAFM conductivity maps reveal heterogeneous conductivity (here in the form of the bands with higher conductivity than the surroundings) related to band-like inhomogeneities of the La distribution. Using LCAFM measurements with atomic resolution obtained between the conducting and nonconducting regions, we analyze the spreading (spatial expansion) of doping on the undoped or low-doped part of the STO crystal. The found limitation of the doping effect of La on the dielectric part of the STO crystal to 4-5 lattice constants is in good correlation with *ab initio* studies from the literature and our recent work [1].

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## References

1. Pilch M.M., Rodenbücher C., Krok F., Szot K., "Heterogeneity in La Distribution in Highly La-Doped SrTiO<sub>3</sub> Crystals", Crystals 13 (2023) 1552. https://doi.org/10.3390/cryst13111552



## Near edge X-ray absorption fine structure (NEXAFS) spectroscopy in the 0.2-1.5 keV spectral range using a laser-plasma soft X-ray source

T. Fok\*, P.W. Wachulak, Ł. Węgrzyński, A. Bartnik and H. Fiedorowicz

Institute of Optoelectronics, Military University of Technology, 00-908 Warsaw 49, Poland \*e-mail: tomasz.fok@wat.edu.pl

We present compact table-top laboratory system for Near Edge X-ray Absorption Fine Structure (NEXAFS) spectroscopy based on laser-plasma soft X-ray source (LPXS). Soft X-ray (SXR) radiation is generated by the interaction of nanosecond laser pulses from a compact Nd:YAG laser (EKSPLA, Vilnius. Lithuania) producing laser pulses of a pulse energy  $E_L = 0.6$  J and duration 3 ns at a repetition rate of 10-Hz) with a double-stream Kr/He or Xe/He gas-puff target. The SXR emission from the Xe/He target covers the energy range of ~200–1500 eV (wavelength of 0.8 nm to 5.6 nm), as shown in Figure 1.



Figure 1. SXR reference spectrum in the photon energy range of 200–1500 eV, obtained for Xe and He gas backing pressures of 10 bar.

Such a broadband emission enables NEXAFS studies of low-Z materials near the K-edge (e.g. C, N, O) and some elements of higher-Z (e.g. transition metals) near the L- and M-edges.

**P9** 

LPXS illuminates a thin sample placed in a small load-lock downstream the beamline, and then the SXR light is transmitted through the

sample, forming a sample beam. The holder allows also some portion of the SXR wavefront, called a reference beam, to enter the spectrometer entrance slit directly . In this way, simultaneous acquisition of two spectra can be achieved. We can call this approach "a dual-channel SXR spectrometer". Such a solution allows spectral acquisition and NEXAFS measurements to remain unaffected by mechanical instabilities or energy fluctuations of the source.

Emission spectra were recorded using a home-made SXR spectrometer, equipped with a grazing-incidence flat-field diffraction grating (Hitachi High Technologies America, Inc., Schaumburg, IL, USA). The spectrometer was also equipped with a 100  $\mu$ m wide entrance slit, located ~715 mm from the plasma source. A CCD camera (GE 20482048, greateyes, Berlin, Germany) with a 2 k × 2 k chip, each pixel of 13 × 13  $\mu$ m<sup>2</sup> in size was used for detection and storage of the SXR spectrum.

The effectiveness of our NEXAFS system has been proven in experiments in which various oxides, including reduced graphene oxide rGO,  $TiO_2$  and Fe, were investigated<sup>2,3</sup>.

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- 1. P. Wachulak, et al., Opt. Express 29 (2021) 20514–20525.
- 2. T. Fok, et al., JINST 15 (2020) C05026.
- 3. T. Fok, et al., Materials 14 (2021) 7337.



## Nitride nanowire surface modifications induced by chemical treatment and oxide layer deposition

**P10** 

B.J. Kowalski<sup>1\*</sup>, M. Sikora<sup>2,3</sup>, A. Reszka<sup>1</sup>, A. Wolska<sup>1</sup>, A. Wierzbicka<sup>1</sup>, S. Gieraltowska<sup>1</sup>, E. Partyka-Jankowska<sup>2</sup>, M. Sobanska<sup>1</sup>, Z.R. Zytkiewicz<sup>1</sup>

<sup>1</sup>Institute of Physics, Polish Academy of Sciences, Aleja Lotników 32/46, PL-02668 Warsaw, Poland <sup>2</sup>SOLARIS Centre, Jagiellonian University, Czerwone Maki 98, PL-30392 Krakow, Poland <sup>3</sup>Academic Centre for Materials and Nanotechnology, AGH University of Krakow, Aleja Mickiewicza 30, PL-30059, Krakow, Poland

\*e-mail: kowab@ifpan.edu.pl

Although the methods of inhibiting the detrimental impact of the surface modifications on the characteristics of semiconductor microdevices have been developed over the past decades, the methods of controlling surface conditions of semiconductor NWs, in particular those made of the group III nitrides, still remain an important and topical subject of research. We applied X-ray absorption spectroscopy (XAS) to assess chemical composition and atomic structure changes in the GaN nanowire surfaces caused by etching or by deposition of thin oxide layers.

The investigated GaN nanowires were grown by catalyst-free plasma-assisted molecular beam epitaxy (PA MBE) on silicon substrates. Four sets of samples were studied: as-grown samples, samples etched with HCl or KOH, samples covered with  $Al_xO_y$  and  $HfO_x$  layers of variable thickness. The oxide layers were deposited with use of the atomic layer deposition technique. XAS measurements were performed at PIRX beamline of SOLARIS. K edge of nitrogen and L edge of gallium were probed as a function of the angle - for the orientation between sample substrate and E vector of incident photon beam ranging from 0 (normal incidence) to 60 degrees.

The spectra acquired for all geometries and samples were normalized to the intensity of the incident beam to derive an effective XAS yield at Ga L and N K edges, which relates to chemical composition of nanowires and the thickness of oxide layers. Spectra collected at varying orientation were further normalized to the maximum on given absorption edge (the method used by M.G. Kibria et al. [1]). Such normalization gives a non-zero X-ray linear dichroism (XLD) signal in the energy range above the absorption edge and generates reliable results for quantitative spectrum analysis in the vicinity of the absorption edge maximum ("white line"). The amplitude of XLD is considered as semi-quantitative measure of structural anisotropy of GaN atomic structure.

The analysis of XAS and XLD spectra led us to the following conclusions:

- reduction of electron yield intensity enables assessment of the thickness of the oxide layers even for the thinnest coverages (below 5 nm),
- deposition of the oxide layers on GaN nanowires leads to reducing of the anisotropy of the GaN crystal structure in the interface layer,
- changes in the XLD and XAS amplitudes for the etched surfaces of GaN nanowires can be interpreted as a manifestation of removing nitrogen atoms from the surface and lowering the surface structural anisotropy of etched nanowires (especially with KOH).

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### References

1. M.G. Kibria et al. Adv. Mater. 28, (2016) 8388.



## Orthorhombic domains in powder RuO2 at ambient conditions

W. Lulka<sup>1\*</sup>, R. Przeniosło<sup>1</sup>, I. Sosnowska<sup>1</sup>, F. Fauth<sup>2</sup> and E. Suard<sup>3</sup>

**P11** 

<sup>1</sup>Faculty of Physics, University of Warsaw, 02-093 Warsaw, Poland <sup>2</sup>ALBA-CELLS BP1413 08290, Cerdanyola del Valles, Barcelona, Spain <sup>3</sup>Institut Laue Langevin, 38042 Grenoble, France

\*e-mail: Wojciech.Lulka@fuw.edu.pl

The crystal structure of the tentative altermagnet  $RuO_2$  was studied by synchrotron radiation based x-ray powder diffraction, constant-wavelength neutron powder diffraction and time-of-flight neutron powder diffraction. The diffraction patterns are best described by Rietveld refinement with the anisotropic Bragg peaks' broadening model. The distribution of the  $RuO_2$  crystallites is composed of a majority of orthorhombic domains (space group *Pnnm*) which coexist with a minority of tetragonal domains (space group  $P4_2/mnm$ ).



In order to demonstrate the changes of fit quality we have done Rietveld refinements with the orthorhombic symmetry (space group *Pnnm*) for fixed values of the a and b lattice parameters calculated as:

$$a = \langle a \rangle \left( 1 + \frac{r}{2} \right); \ b = \langle a \rangle \left( 1 - \frac{r}{2} \right)$$

where  $\langle a \rangle$  is the average of a and b obtained in the orthorhombic fit while the orthorhombic deformation parameter r changes between -0.0057 < r < 0.0057 with a fixed step. All instrumental, background and structural parameters (except fixed a and b) were refined. Please note that r = 0 corresponds to the tetragonal rutile-type fit. The value of  $\chi^2(r)$  for the best fit at each value of r is drawn with solid symbols. The curves drawn show the fourth-order even polynomial  $f(r) = ar^4 + br^2 + c$ fitted with very good agreement to the observed  $\chi^2(r)$  for all three datasets. The results of SXRPD and NPD show good correspondence with each other with a double

minimum of  $\chi^2(r)$  near r = 0.0036. The neutron diffraction n-TOF pattern measured with a powder RuO<sub>2</sub> sample from a different batch than the first two gives also a clear minimum near r = 0.0032.

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## References

1. J. Haines, J. M. Leger, O. Schulte, and S. Hull, Neutron Diffraction Study of the Ambient-Pressure, Rutile-Type and the High-Pressure, CaCl<sub>2</sub>-Type Phases of Ruthenium Dioxide, Acta Crystallographica Section B 53 (1997) 880.



## Polaronic transport and magnetic field dependence of conduction parameters in rGO-CFO nanocomposites

P12

S. Manzoor<sup>1</sup>, R. Anwar<sup>2\*</sup>, S. Hussain<sup>1</sup>, Z. Arshad<sup>3</sup> and J. Arshad Darr<sup>3</sup>

<sup>1</sup>Magnetism Laboratory, Department of Physics, COMSATS University Islamabad, Park Road, Islamabad, Pakistan <sup>2</sup>Institute of Physics PAS – IFPAN, Warsaw, Poland <sup>3</sup>Department of Chemistry, University College London, London WC1H 0AJ, United Kingdom

\*e-mail: ranwar@ifpan.edu.pl

In this work we report the AC conductivity  $\sigma$  and magnetoconductivity  $\Delta \sigma$  of a series of reduced graphene oxide-cobalt ferrite (rGO-CFO) composites in which the mass ratio of rGO:CFO has been systematically varied over six compositions. The conductivity measured in the frequency range of 20 Hz to 3 MHz showed an anomalous increase with CFO content in the composite, even though the conductivity of CFO is at least three orders of magnitude smaller than that of rGO. Magnetoconductivity measured in fields upto 2000 Oe displayed both negative and positive values depending on the rGO:CFO ratio. Maximum magnetoconductivity of  $\sim +14\%$  was obtained at room temperature in a magnetic field of only 750 Oe. Samples having the largest  $\sigma$  and  $\Delta \sigma$  values also showed significant softening/hardening of the Raman active modes of CFO, indicating polaronic transport in these composites. Fits of  $\sigma(f)$  data to a power law  $\sigma(f) = \sigma_0 + Af^n$  showed that parameters A and n depend strongly on the applied magnetic field. This dependence is qualitatively and quantitatively different for samples that display positive or negative magnetoconductivity. Although the temperature dependence of these conduction parameters has been extensively studied, we have not come across any reports of their magnetic field dependence. Based on these observations we present a surface polaron model in which hybridization of  $\pi$ -orbitals of graphene with surface states of CFO nanoparticles provides for a polaron conduction mechanism through a grid of CFO nanoparticles intercalated with rGO sheets.

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## References

1. S. Manzoor et al, J. Phys. D: Appl. Phys. 58 (2025) 135001.



# Regulating the crystal structure of LiMn2O4 material by Zn doping

J. Darul<sup>1</sup> and P. Piszora<sup>1</sup>\*

**P13** 

<sup>1</sup>Department of Materials Chemistry, Faculty of Chemistry, Adam Mickiewicz University, Uniwersytetu Poznańskiego 8, 61-614 Poznań, Poland

### \*e-mail: pawel@amu.edu.pl

The substitutional defect consisting of the aliovalent substitution of lithium ions by zinc ions in the tetrahedral sublattice of the  $Li_{1-x}Zn_xMn_2O_4$  spinel changes the overall charge of the ionic compound. In order for the ionic compound to remain neutral, a charge compensation mechanism is required. There are different ways to do this, e.g. partial reduction of manganese occurs or ionic vacancies are created. Cooperative control of atomic composition and defect engineering allows for obtaining materials with extraordinary properties<sup>1-3</sup>.

We adopted  $Li_{0.9}Zn_{0.1}Mn_2O_4$  oxide as a model system to investigate the chemical strain induced by tetrahedrally located zinc ions insertion and its potential effect on the electrochemical performance. In the lithium manganese spinel  $LiMn_2O_4$ , the monovalent  $Li^+$  cation occupies the tetrahedral A sites, and the octahedral B sites are occupied by a mixture of  $Mn^{4+}$  ions and the Jahn-Teller active  $Mn^{3+}$  ions. This cation distribution results in a cubic structure (*Fd-3m*). The number of  $Mn^{3+}$  ions in the octahedral sublattice is insufficient to generate the cooperative Jahn–Teller effect at ambient temperature and to deform the crystal structure.

The unique properties of the material studied in this work result from the relatively unique distribution of ions in two crystallographic positions. Reducing the number of lithium ions in the tetrahedral positions, as in  $Li_{0.9}Zn_{0.1}Mn_2O_4$ , allows the remaining tetrahedral sites to be filled with  $Zn^{2+}$  ions. As a consequence, the oxidation state of manganese in octahedral positions decreases, resulting in a Jahn–Teller distortion with tetragonal symmetry and c/a' > 1.

For the laboratory synthesis of multicomponent spinel oxide, we used a modified Pechini method. The method was based on mixing standard solutions of manganese and nickel nitrate with lithium carbonate and citric acid, and then the solution of trimetallic citrate complexes was transformed into a polymer gel by controlled heating and finally thermal decomposition of the polymer matrix to remove the organic component<sup>4</sup>. When the heating temperature reaches 673 K, oxidation and pyrolysis of the polymer matrix occur. We made several attempts, performing heat treatments at different temperatures, each time ending with rapid cooling of the sample in solid CO<sub>2</sub>. The structure of the sample was investigated by the powder diffraction experiment performed at the I711 beamline in MAXLab (Lund, Sweden).

Knowledge of the structure design strategy via cationic distribution will facilitate the design of materials with new desirable properties, including high energy density and long cycle life cells, selective catalysts, and nonvolatile resistive random access memory devices.

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- 2. J. Huang, C. Wang, Y. Huang, Y. Jiang, J. Sun, L. Xue, J. Zhu, J. Energy Storage 88 (2024) 111553.
- 3. M. Jiang, Q. Qin, J. Li, M. Zeng, ACS Appl. Mater. Interfaces 17 (2025) 13997.
- 4. J. Darul, P. Piszora, Ceram. Int. 49 (2023) 9412.

<sup>1.</sup> C.W. Cady, G. Gardner, Z. O. Maron, M. Retuerto, Y.B. Go, S. Segan, Greenblatt. Greenblatt, G.C. Dismukes, ACS Catal. 5 (2015) 3403.



## Resonant photoemission study of MnFePAs compounds exhibiting giant magnetocaloric effect.

M. Sikora1\*, J. Goraus1 and J. Kubacki1

**P14** 

<sup>1</sup>University of Silesia, Institute of Physics, Chorzów, Poland \*e-mail: marcin.sikora@us.edu.pl

Magnetic refrigeration which is based on the magnetocaloric effect (MCE) has emerged as a serious alternative to conventional refrigeration. Recently, the group of compounds  $(Mn,Fe)_2P_{1-x}As_x$  has been found to exhibit the giant magnetocaloric effect which is related to the first-order phase transition, and is tunable around room temperature [1]. The P/As ratio appeared to be an interesting method to tune the basic properties for any application such as Curie temperature and thermal hysteresis width [2]. In order to understand the effect of substitution we performed the extended studies of the electronic structure, crystal structure and magnetism for the selected group of solid compounds  $Mn_{1.1}Fe_{0.9}P_{1-x}As_x$  (0.4<x<0.55). The substitution of silicon by arsenic has strong influence on the value of the magnetocaloric effect and the phase transition temperature. Despite the standard method of determination of the magnetic entropy changes from the magnetic measurements in conjunction with the Maxwell relations we measured the direct adiabatic temperature change which reached the high value of 4 K at the field of 1.7 T for x=0.5 and Si substituted by As [3].

The main goal of our work was finding the relationship between magnetic properties and electronic structure of magnetocaloric materials, and their comparison with the results of theoretical calculations. In order to determine the electronic structure of manganese combined with their magnetic properties we have performed detailed x-ray resonant photoemission (RESPE)) studies on Mn L<sub>2,3</sub> edge and Mn M edge and x-ray magnetic circular dichroism (XMCD) studies under an applied magnetic field of 0.47 Tesla for  $Mn_{1.1}Fe_{0.9}P_{0.5}As_{0.4}Si_{0.1}$  and  $Mn_{1.1}Fe_{0.9}P_{0.6}As_{0.4}$  samples prepared by SPS (spark plasma sintering) method.



Figure 1. Mn L-edge XAS spectra obtained for reference sample and with Si substitution recorded at room temperature and T=100K.

The general shape of the Mn-L edge of studied samples can be described by strong peak with additional two features within the shape of  $L_3$  edge and at least two components within  $L_2$  edge (see Fig. 1). The resonant photoemission studies performed for particular points of absorption spectra measure both for L-edge and M-edge showed participation of 3d manganese states in the valence band. The clear exchange splitting effect have been on core level Mn2p have been observed at low temperature. Further, we detected a small XMCD signal on Mn L edge at 100 K (see Fig. 2), thus providing a direct evidence of the especially role of Mn atoms in magnetic properties of MnFePAs series.



## XVI International School and Symposium on Synchrotron Radiation in Natural Science Poland, 25-30 May 2025



Figure 2. X-ray absorption spectra for  $Mn_{1.1}Fe_{0.9}P_{0.5}As_{0.4}Si_{0.1}$  compound. Gently difference between shape of the curves can be found and difference spectrum is also shown.

Additionally we performed the analysis of the influence of silicon on the partially density of states of manganese and iron of the  $Mn_{1.1}Fe_{0.9}P_{1-x}(As,Si)_x$  system. We found a differences in the electronic structure of the iron and manganese elements located two different positions 2f and 2g in the elementary cell, respectively. The obtained results indicated the impact of silicon substitution on the changes of adiabatic temperature change in relation to the observed changes in the magnetic moment of manganese in range of phase transition.

- 1. O. Tegus, E. Brück, K.H.J. Buschow, F.R. de Boer, Nature 415 (2002) 150-152.
- 2. B.G. Shen, J.R. Sun, F.X. Hu, H.W. Zhang, Z.H. Cheng, Adv. Mater. 21 (2009) 4545-4564.
- 3. P. Wlodarczyk, L. Hawelek, M. Kowalczyk, M. Kaminska, P. Zackiewicz, M. Polak, M. Hreczka, A. Kolano-Burian, Sol. State Sci. 56 (2016) 23-28.



## Solid-liquid interface of water/SAE304 probed by TREXS (Total REflection X-ray Spectroscopy)

**P15** 

H. Abe<sup>1,2,3\*</sup>, Y. Niwa<sup>1,2</sup> and M. Kimura<sup>1,2</sup>

<sup>1</sup>Institute of Materials Structure Science, High Energy Accelerator Research Organization (KEK), 1-1 Oho, Tsukuba, Ibaraki, 305-0801 Japan <sup>2</sup>Graduate University for Advanced Studies (SOKENDAI), 1-1 Oho, Tsukuba, Ibaraki, 305-0801 Japan <sup>3</sup>Graduate School of Science and Engineering, Ibaraki University, 2-1-1 Bunkyo, Mito, Ibaraki, 310-8512 Japan

\*e-mail: hitoshi.abe@kek.jp

Surfaces and interfaces are the places where important chemical reactions occur. Water splitting, for example, a key reaction of artificial photosynthesis, proceeds at solid-liquid interfaces. Solid-liquid interfaces have been studied for more than 30 years, and STM in liquids revealed real images of the topmost layer at interfaces<sup>1</sup>. Spectroscopies have been also applied<sup>2,3</sup>. However, kinetics and behaviours at interfaces are still unclear. It is required to observe chemical states and local structures with a reasonable interface sensitivity and enough temporal resolution.

We have developed Total REflection X-ray Spectroscopy (TREXS) with the surface sensitivity of ~2-3 nm<sup>4,5</sup>, which gives information on chemical states and local structures, based on X-ray Absorption Fine Structure (XAFS) method. Near edge regions of TREXS spectra are useful to reveal chemical states and to track kinetics of surface reactions, while extended regions of the spectra can be analysed to obtain local structures. Similar methods are reported, and film growth processes have been discussed, for example<sup>6</sup>.

interface TREXS Solid-liquid has been developed based on the surface sensitive TREXS to study various solid-liquid interface phenomena and reactions. We have carried out solid-liquid TREXS measurements to observe a solid-liquid interface of stainless steel, SAE304, under water, which was covered by a polyimide film. A bare SAE304 surface was also observed for comparison. Here, wide energy range TREXS spectra including both Fe and Ni K-edges were clearly obtained in one scan. These spectral features will be discussed at the conference. We are trying to develop this solid-liquid interface TREXS method further to perform real time observation.



Figure 1. Obtained TREXS spectra covering both Fe and Ni *K*-edges by one scan: (red) bare SAE304 and (blue) water/SAE304.

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- 1. H. Honbo, et al., Anal. Chem. 62 (1990) 2424.
- 2. S. Yang, et al., J. Phys. Chem. C 119 (2015) 26056.
- 3. Q. Yuan, et al., J. Phys. Chem. C 122 (2018) 16664.
- 4. H. Abe, et al., J. Phys.: Conf. Ser. 502 (2014) 012035.
- 5. H. Abe, et al., Jpn. J. Appl. Phys. 55 (2016) 062401.
- 6. D. L.-Hecht and R. Frahm, Thin Solid Films 493 (2005) 67.



## Structural and surface morphology modifications of Pd<sub>100-x</sub>Si<sub>x</sub> thin film induced by ultrashort pulsed laser annealing

**P16** 

I. Jacyna<sup>1\*</sup>, O. Liubchenko<sup>1</sup>, J. Antonowicz<sup>2</sup>, K. Sokolowski-Tinten<sup>3</sup>, P. Zalden<sup>4</sup>, P. Dzięgielewski<sup>2</sup>, T. J. Albert<sup>4</sup>, M. Chojnacki<sup>1</sup>, A.-C. Dippel<sup>5</sup>, O. Gutowski<sup>5</sup>, D. Klinger<sup>1</sup>, Z. Kostera<sup>2</sup>, R. Minikayev<sup>1</sup> and R. Sobierajski<sup>1</sup>

> <sup>1</sup>Institute of Physics Polish Academy of Sciences, 02-668 Warsaw, Poland <sup>2</sup>Faculty of Physics, Warsaw University of Technology, 00-662 Warsaw, Poland <sup>3</sup>Center for Nanointegration Duisburg-Essen (CENIDE), University of Duisburg-Essen, 47048 Duisburg, Germany <sup>4</sup>European XFEL, Holzkoppel 4, 22869 Schenefeld, Germany <sup>5</sup>Deutsches Elektronen Synchrotron DESY, 22607 Hamburg, Germany

> > \*e-mail: yatsyna@ifpan.edu.pl

Over the past years, metallic glasses have been widely investigated due to their outstanding mechanical and magnetic properties<sup>1</sup>. Some pure metals have been found to amorphize only when quenched at a rate of  $\sim 10^{14}$  K/s<sup>2</sup>, whereas certain metallic alloys with compositions near deep eutectics (so called bulk metallic glasses) vitrify upon quenching at a fraction of a Kelvin per second. The binary Pd-Si system is one of the most notable examples of strong composition dependence of the glass-forming ability. While pure Pd is essentially a non-glass-former and has only been observed to vitrify in computer simulations<sup>3</sup> under ultra-high quenching rates, the eutectic Pd<sub>81</sub>Si<sub>19</sub> alloy (composition in at.%) can be easily cast into a glass, e.g., by air or water cooling at rates of 1–10 K/s<sup>4</sup>.

In this work, we studied 20 nm thick  $Pd_{100-x}Si_x$  thin-film metallic glasses (x = 0, 3, 5, 10), which were deposited on 50 nm thick SiN membranes and covered with a 5 nm thick SiN capping layer. The as-deposited films were either completely crystalline (x=0) or partially crystalline (with a growing amorphous volume fraction as the Si content -x - increased). Structural and surface morphology modifications of the films were induced using ultrashort sub-ps laser pulses of various fluences at a 515 nm wavelength. Local structural modifications were characterized by means of X-ray diffraction in transmission with a micro-focused X-ray beam at the P07 beamline of the PETRA III synchrotron. Analysis of the irradiated spots through optical and electron microscopy enabled to correlate observed structural modifications with the locally deposited energy density from the annealing laser pulses. We observe that with increasing deposited energy density, the average crystal grain size of the layer increased, microstrain reduced, and the volume fraction of the amorphous phase decreased. This indicates that in layers initially in a metastable state, laser heating increased the mobility of atoms, allowing the system to relax towards a lower energy state (i.e. growth of crystalline grains and reduction of lattice strain). At the same time, the final sample state exhibited larger lattice parameters and higher Debye-Waller factor, indicating increased structural disorder. We attribute this to the rapid lattice freezing due to the quick heat diffusion to the substrate. Our analysis provides new insights into the composition-dependent crystallization behavior of Pd-Si thin-films.

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- 1. Greer, A. L. Mater. Today, 12 (2009) 14.
- 2. Zhong L., Nature 512 (2014) 177.
- 3. Yang Z. J., Tang L., et al., J. Phys. Condens. Matter 31 (2019) 135701.
- 4. Yao K.F., Chen N., Sci. China, Ser. G Physics, Mech. Astron. 51 (2008) 414.



# Surface analysis of TiO<sub>2</sub> and Ti nanolayers modified with highly charged Xe<sup>q+</sup> ions using synchrotron radiation based XRR and GIXRF methods

R. Stachura<sup>1,2\*</sup>, D. Banaś<sup>1,3</sup>, P. Jagodziński<sup>1,2</sup>, A. Kubala-Kukuś<sup>1,3</sup>, I. Stabrawa<sup>1,3</sup>, K. Szary<sup>1,3</sup>, M. Pajek<sup>1</sup>, G. Aquilanti<sup>4</sup>, I. Božičević Mihalić<sup>4,5</sup>, Md. A. Alam<sup>6</sup> and M.K. Tiwari<sup>6</sup>

> <sup>1</sup>Institute of Physics, Jan Kochanowski University, 25-406 Kielce, Poland <sup>2</sup>Central Office of Measures, 00-139 Warszawa, Poland <sup>3</sup>Holycross Cancer Center, 25-734 Kielce, Poland <sup>4</sup>Elettra - Sincrotrone Trieste, 34149 Basovizza, Trieste, Italy <sup>5</sup>Rudjer Boskovic Institute, 10000 Zagreb, Croatia <sup>6</sup>Raja Ramanna Centre for Advanced Technology, Indore 452013, India

> > \*e-mail: regina.stachura@ujk.edu.pl

Due to their unique physical and chemical properties, nanolayers currently play a key role in various technological applications, including electronics, optics, biomedical coatings and energy storage devices [1-2]. Irradiation of nanolayers with highly charged ions (HCl) induces changes in the morphology, density and chemical composition, affecting the electronic, optical and mechanical properties of the nanolayers. By controlling HCI parameters such as fluence, ion charge state and energy, various modifications can be induced, allowing for the improvement of a surface properties such as adhesion, conductivity and corrosion resistance. Therefore, understanding the processes occurring during the interaction of HCI with surfaces is essential for the development of new materials with improved properties [3-4].

The main objective of this work was to analyse titanium dioxide (TiO2) and titanium (Ti) nanolayers deposited on Si substrate, unmodified and irradiated with low-energy, highly charged Xe<sup>q+</sup> ions (q = 25, 30, 35), and to determine the effect of irradiation on their surface morphology. The nanolayers were irradiated at the Kielce EBIS facility (Jan Kochanowski University, Kielce, Poland) [5].

The analysis of non-modified and irradiated nanolayers was performed by X-ray reflectometry (XRR) and grazing incidence X-ray fluorescence (GIXRF) methods using synchrotron radiation excitation at Elettra Synchrotron XRF beamline. XRR method allowed the determination of nanolayer properties such as density, thickness, and roughness. Thanks to the low-angle GIXRF technique it was possible to characterize the depth distribution of a O, Si and Ti in the nanolayers [6]. In this work the results of measurements obtained using XRR and GIXRF techniques will be presented and compared with theoretical predictions. The analyses performed showed significant differences in the surface morphology of nanolayers depending on the ion charge state.

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- 1. N. Kumar et. al., Processing of Polymer-based Nanocomposites, Springer Publishing (2018) 15-55.
- 2. J. Barth et. al., Nature 437 (2005) 671–679.
- 3. H. Gnaser, Low-Energy Ion Irradiation of Solid Surfaces, Springer Berlin Heidelberg (1999) 7-82.
- 4. R. A. Wilhelm et. al., Progress in Surface Science 90 (2015) 377-395.
- 5. D. Banaś et. al., Nucl. Instrum. Meth. Phys. Res. B 354 (2015) 125-128.
- 6. R. Klockenkämper et. al., Total-Reflection X-Ray Fluorescence Analysis and Related Methods, John Wiley & Sons Inc. (2014)



# Thermostructural properties of β and γ polymorphs of lead orthovanadate (Pb3V2O8) in a broad temperature range (298 K-973 K): An X-ray diffraction study P18

F. Aziz<sup>1</sup>\*, W. Paszkowicz<sup>1</sup>, R. Minikayev<sup>1</sup>, C. Martin<sup>2</sup> and H. Teisseyre<sup>1</sup>

<sup>1</sup>Institute of Physics, Polish Academy of Sciences,32/46, Warsaw,02-668, Poland <sup>2</sup>Laboratorie CRISMAT, Normandie Universite, Caen, 14050, France

\*e-mail: fizaaz@ifpan.edu.pl

Lead orthovanadate, Pb<sub>3</sub>V<sub>2</sub>O<sub>8</sub>, is known to exhibit polymorphism, the three reported phases are  $\alpha$  (monoclinic), stable below 273 K,  $\beta$  (monoclinic) and  $\gamma$  (rhombohedral). For temperatures exceeding the room temperatures, the transition from  $\beta$  to  $\gamma$  has been observed, at 373 K [1-3]. Thermal expansion of the polymorphs has been reported in a limited temperature range 90- 550 K [1]. In the present work we extend the knowledge on the system by studying the broad temperature range (298-973 K) and by investigating the structure details as function of temperature. The atomic positions, interatomic distances, interbond angles, polyhedron distortions are determined. The  $\beta$  phase unitcell parameters a and c show an increase with temperature i.e. 0.21% for a and 0.58%for c but the parameter b decreases by 0.68%. The increase in angle  $\beta$  is 0.11% and unitcell volume of  $\beta$  phase increases by about 0.12%. In case of the  $\gamma$  phase, the unit-cell parameters increase by about 0.88% along a and 1.11% along c- direction having about 2.87% increase in unit-cell volume. The thermal expansion coeffcient (TEC) tensor calculated shows expansion anisotropy for  $\beta$  phase and follows order  $\alpha_c > \alpha_a > \alpha_b$ . For the  $\gamma$ phase, TEC also shows expansion anisotropy that follows order  $\alpha_c > \alpha_a = \alpha_b$ . The evolution of lattice parameters of  $\beta$  and  $\gamma$  phases is in line literature data [1] referring to temperature ranging to 550 K.



Figure 1. Evolution of lattice parameters of  $\beta$  and  $\gamma$  phases. In inset the evolution of angle  $\beta$  with temperature in  $\beta$  phase is shown. The room temperature values for beta phase from ref [1] are displayed as crosses for comparison.

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- 1. P. Garnier , G. Calvarin, J. Berar, D. Weigel. Materials research bulletin 19(1984) 407-14.
- 2. J. Kiat, P. Garnier, M. Pinot. Journal of Solid State Chemistry 91(1991) 339-49.
- 3. H. Kasatani, T. Umeki, H. Terauchi. Journal of the Physical Society of Japan 61(1992) 2309-16.



## THz radiation yield from Polfel undulator

R. Nietubyć1\*, M. Staszczak1 and U. Lehnert 2

**P19** 

<sup>1</sup>Narodowe Centrum Badań Jądrowych, Świerk, 04-500 Otwock, Poland <sup>2</sup>Helmholtz Zentrum Dresden-Rossendorf, 01328 Dresden, Germany

\*e-mail: robert.nietubyć@ncbj.gov.;pl

The PolFEL will be an open access users' laboratory providing electromagnetic radiation ranging from terahertz frequencies to extreme ultraviolet (EUV). Radiation in these ranges will be sourced from separate sources based on: electron accelerators, solidstate lasers, and high-harmonic generation systems, respectively. A superconducting continuous wave linac, specifically designed for THz radiation, will contain two HZDRlike cryomodules and a permanent magnet, tunable-gap undulator. Electrons accelerated up to 70 MeV (up to 57 MeV in cw mode) will be shaped in bunches with charges up to 250 pC and will proceed with 1 kHz repetition. Individually driven cavity structures and bunch compressor placed downstream, enable to obtain 10  $\mu m$  long bunches with a peak current of 2000 A at the undulator entrance. The undulator, which has been designed in-house, is currently being assembled in the institute's workshop. Radiation simulations have been performed to provide data for optical path design and to evaluate the performance capabilities of the experimental endstation. Pulse energy, angular, and spectral distributions have been calculated using the TEUFEL program and using linac beam dynamics simulations results from the ASTRA program. The results showed that frequency range from 0.5 THz to 5 THz will be accessible for experimental use with pulse energies in microjoules range, in a 0.1 relative bandwidth. The Figure below shows spectral power density distributions calculated for first harmonic radiation for selected beam energies and maximal achievable undulator parameters K=5.4 The distribution centred at 5.1 THz will be accessible in long pulses mode only.



Spectral power density calculated for beam energies 15 MeV, 23 MeV, 33 MeV, 57 MeV and 70 MeV



## Structural study of transition metal-doped ceria reduced in hydrogen atmosphere: XAS analysis P20

A. Witkowska\* and B. Bochentyn

Institute of Nanotechnology and Materials Engineering, Faculty of Applied Physics and Mathematics, Gdańsk University of Technology, ul. Narutowicza 11/12, 80-233 Gdańsk, Poland

\*e-mail: agnieszka.witkowska@pg.edu.pl

Ceria-based compounds find a very wide range of applications in electrochemical devices such as solid oxide fuel cells, oxygen storage materials, oxygen sensors or catalysts of partial oxidation of hydrocarbons. The transport properties of  $CeO_{2-\delta}$  compounds strongly depend on temperature, oxygen partial pressure, microstructure, and the presence of dopants and impurities. Such flexibility of properties makes them interesting candidates e.g. for catalytic materials working on the anode side of Solid Oxide Fuel Cells (SOFCs), significantly affecting the process of direct internal reforming of biogas and limiting the carbon deposition process on the fuel cell [1,2]. Ceria has also paid some attention due to the possibility of exsolving metallic nanoparticles to the surface of its grains. In this process occurring in reducing conditions, some cations from host compound are extruded in the form of nanoparticles on the oxide support. The exsolution method offers even distribution of particles as well as improved attachment to the host material [3]. Additionally, the exsolution process may be partially reversible, meaning the exsolved particles could be incorporated to the host lattice in the oxidative conditions [4]. Although the exsolution remains the hot topic and constant development in this field is observed, the research is mostly focused on the in situ growth of monometallic nanoparticles from perovskite-like structures. Only few experimental works on exsolution from fluorite structures can be found, and what is more, they have focused mostly on Ni-nanoparticles exsolution [5]. There is lack of systematic analysis of other types of transition metals exsolution depending on the process conditions.

In this study we focused on structural study of ceria-based compounds with general formula  $(Ce,Me)O_{2-\delta}$  (where Me = Co, Cu, Fe, Mn, Ni) which were reduced in hydrogen atmosphere at various temperatures (900, 1000, 1100°C) to force the exsolution process. For this purpose, we used hard X-ray and state-of-the-art technique such as X-ray Absorption Spectroscopy (XAS). A series of XAFS (X-ray Absorption Fine Structure) spectra at K-edges of Ce and transition metals were collected. Near-edge (XANES) and extended (EXAFS) range spectra analysis allowed to reconstruct the local structure of elements both in the as-prepared and reduced ceria, leading to a deeper understanding of the exsolution process in fluorite structures depending on the conditions of exsolution process, as well as a type of transition metal dopant. Moreover, the occurrence of cerium in the mixed valence state of Ce<sup>3+</sup>/Ce<sup>4+</sup> was confirmed, what has a significant influence both on catalytic properties and on resistance to carbon deposition when this material is used as an anode for SOFC.

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### References

1. B. Hołówko, P. Błaszczak, M. Chlipała, M. Gazda, S.-F. Wang, P. Jasiński, B. Bochentyn, International J. Hydrogen Energy 45 (2020) 12982-12996.

2. B. Bochentyn, M. Chlipała, M. Gazda, S.-F. Wang, P. Jasiński, Solid State Ionics 330 (2019) 47-53.

3. J.H. Kim, J.K. Kim, J. Liu, A. Curcio, J-S. Jang, Il-D. Kim, F. Ciucci, WCh. Jung, ACS Nano 15/1 (2021) 81-110.

- 4. Q.A. Islam, S. Paydar, N. Akbar, B. Zhu, Y. Wu, J. Power Sources 492 (2021) 229626.
- 5. A. Singhania, S.M. Gupta, Nanotechnol. 18/7 (2018) 4614-4620(7).



## Surface and electronic properties of Mn-modified PdTe<sub>2</sub> and Bi<sub>2</sub>Te<sub>3</sub>

K. Balin<sup>1\*</sup>, J. Kubacki<sup>1</sup> and T. Sobol<sup>2</sup>

**P21** 

<sup>1</sup>A. Chełkowski Institute of Physics, University of Silesia in Katowice, 41-500 Chorzów, Poland <sup>2</sup>National Synchrotron Radiation Centre SOLARIS, Jagiellonian University, 30-392 Kraków, Poland

## \*e-mail: katarzyna.balin@us.du.pl

This study investigates the electronic and magnetic properties of  $Mn/(PdTe_2, Bi_2Te_3)$  junctions, focusing on the interplay between surface phenomena and material modifications.  $PdTe_2$ , a superconducting semimetal with topological states and high carrier mobility, and  $Bi_2Te_3$ , a topological insulator renowned for its thermoelectric efficiency, were integrated with Mn layers to explore novel electronic and spintronic functionalities. The surface region, governed by spin-orbit coupling, plays a critical role in modulating these properties, offering potential applications in low-power electronics and advanced data storage systems.

Using Molecular Beam Epitaxy (MBE), Mn layers of varying thicknesses were deposited on single crystals of Bi<sub>2</sub>Te<sub>3</sub> and PdTe<sub>2</sub>. The electronic structure and surface chemistry were systematically characterized using X-ray photoelectron spectroscopy (XPS), Soft Xray Angle- Resolved Photoelectron Spectroscopy (SX-ARPES), and X-ray Absorption Spectroscopy (XAS). Mn growth and all characterizations were obtained using PHELIX line at National Synchrotron Radiation Centre SOLARIS.

SX-ARPES measurements, performed at photon energies of 55, 70, and 100 eV, revealed significant modifications in the band structure upon Mn deposition. XPS analysis identified the formation of Te-Mn bonds and the isolation of metallic Bi, alongside subtle changes in the chemical states of  $Bi_2Te_3$  and  $PdTe_2$ . Depth-resolved XPS, conducted at beam energies of 200– 1000 eV, provided insights into the layered composition. XAS spectra, acquired in Total Electron Yield (TEY) and Partial Electron Yield (PEY) modes, highlighted the surface-sensitive electronic and magnetic interactions, with a non-flat X-ray Magnetic Circular Dichroism (XMCD) signal at the Mn L edge indicating complex magnetic behavior.

These findings underscore the potential of Mn-modified telluride junctions for tailoring electronic and magnetic properties, paving the way for advancements in spintronics and low- power electronic devices.

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- 1. G. D'Olimpio et. al., Adv. Funct. Mater 30 (2020) 1906556.
- 2. E.D.L Rienks, et.al., Nature 576 (2019) 423–428.



# Local structure around the Co, Ni and Cu dopants in the calcium orthovanadates as seen by X-ray absorption

P22

A. Wolska<sup>1,\*</sup>, H.S. Rahimi Mosafer<sup>1</sup>, Ch. Martin<sup>2</sup>, M. Berkowski<sup>1</sup>, W. Paszkowicz<sup>1</sup>, A. Drzewiecka-Antonik<sup>1</sup> and M. Klepka<sup>1</sup>

<sup>1</sup>Institute of Physics Polish Academy of Sciences, 02668 Warsaw, Poland <sup>2</sup>Laboratoire CRISMAT, Normandie Université, ENSICAEN, UNICAEN, CNRS, 14050 Caen, France

\*e-mail: wolska@ifpan.edu.pl

Calcium orthovanadate  $Ca_3(VO_4)_2$  crystallizes in R3c space group with structure similar to that of mineral whitlockite. In the pure crystals some part of Ca cations can be replaced by transition metals (TM), of valences from +1 to +4, without a change of structure. In such case, the corresponding structural modification consists in a partial or full replacement of Ca cations at one (case of substitutionally ordered compound) or more of Ca sites (disordered compound case) by the substituting ion. The resulting novel materials have promising properties that can be utilized in optoelectronic applications, among others can be used for white light-emitting diodes.



Figure 1. The Ca atoms located in 5 inequivalent crystallographic positions (M1-M5) at the Ca K-edge

The Ca cations are located in 5 inequivalent crystallographic positions (M1-M5). The X-ray absorption technique, by probing the local neighbourhood around specific element, is a perfect tool to determine whether one of the sites is preferred. Fig. 1 presents the FEFF10 simulations of the XANES spectra for all the sites in case of the pure crystal showing the clear differences between each position.

The presented research is focused on the orthovanades crystals:  $Ca_{10.5-x}TM_x(VO_4)_7$  doped with TM = Co, Ni or Cu where x ranges nominally from 0.50 to 0.72 %. The XANES and EXAFS measurements were performed at the ASTRA beamline (SOLARIS) in a fluorescence mode.

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**P23** 

# Structural studies of bioactive Ag(I) complexes with coumarin derivatives

A. Wolska<sup>1\*</sup>, A. Drzewiecka-Antonik<sup>1</sup>, C. Aparecida Barboza<sup>1</sup>, P. Rejmak<sup>1</sup> and M. Klepka<sup>1</sup>

<sup>1</sup>Institute of Physics, Polish Academy of Sciences, 02668 Warsaw, Poland

\*e-mail: wolska@ifpan.edu.pl

Silver complexes with organic ligands has been attracting wide attention due to their strong bactericidal properties. Among them those with coumarin derivatives since the coumarin core is a common structural motif in both natural products and bioactive molecules. Moreover, depending on the substituents around the cyclic core, coumarin-containing compounds have shown a wide range of pharmacological activity. Following this, two new Ag(I) complexes with coumaric carboxylic acid derivatives have been synthesized and structurally characterized.



Structural characterization of the complexes was conducted for noncrystalline materials using a methodology developed by the authors<sup>1</sup>, which combines laboratory and synchrotron based techniques as well as density functional theory (DFT) calculations. The elemental analysis and attenuated total reflectance infrared (ATR-IR) spectroscopy provided information on the functional groups coordinating to the metal cation and the metalto-ligand ratio. Extended X-ray absorption fine structure (EXAFS) spectroscopy supplied information about the coordination number and the kind of species surrounding the absorbing cation. X-ray absorption near edge structure (XANES) spectroscopy and DFT

calculations helped to determine the most probable structural models. In both complexes different coordination modes of carboxylate ligands were detected. For the Ag(I) complex with 4-oxo-4H-1-benzopyran-2-carboxylic acid (complex **1**), carboxylate bridges was observed. For the complex **2**, with 2-oxo-2H-1-benzopyran-3carboxylic acid, a bidentate chelating motif was found. Moreover, the carbonyl oxygen atom of the coumarin ring coordinates to the silver cation in complex **2**, while it is inactive in complex **1**.

Additionally, microbiological activity of the investigated complexes was performed<sup>2</sup>. The complexes show activity against selected bacteria as well as *Candida* yeast. This activity is slightly lower for bacteria and the same or higher for *Candida* in relation to the reference substances: ciprofloxacin or fluconazole.

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- 1. M.T. Klepka et al., Chem. Phys. Lett. 691 (2018) 190.
- 2. A. Wolska et al., Molecules 29 (2024) 4993.



## Impact of Copper Oxide Nanoparticles on Human Cystatin C - Cell Viability and *In Vivo*-like Flow Studies

P24

K. Rucińska<sup>1,2\*</sup>, J. Maksim<sup>1,2</sup>, Ł. Przysiecka<sup>3</sup>, A. Kertmen<sup>1</sup> and M. Kozak<sup>1,4</sup>

<sup>1</sup>Adam Mickiewicz University in Poznań, Faculty of Physics, 61-614 Poznań, Poland
 <sup>2</sup>Institute of Nuclear Physics Polish Academy of Sciences, 31-342 Kraków, Poland
 <sup>3</sup>NanoBioMedical Centre AMU, 61-614 Poznań, Poland
 <sup>4</sup>SOLARIS National Synchrotron Radiation Centre, 30-392 Kraków, Poland

\*e-mail: karolina.rucinska@amu.edu.pl

Well known fact is, that long-term exposure to air pollution is a risk factor for the development of serious diseases, including cancer or dementia. Among the harmful components of polluted air are heavy metals such as Cu and Zn and their compounds. These metals, especially in the form of micro- or nanoparticles, have been linked to toxic effects, posing significant health risks to humans and other organisms. Studies have shown that human SH-SY5Y cell lines exhibit concentration-dependent neurotoxic responses to copper oxide nanoparticles (CuO NPs), leading to apoptosis in neuronal cells and potentially contributing to Alzheimer's disease (AD)<sup>1</sup>.

Evidence suggests that metal ions can bind to amyloid beta (A $\beta$ ) and modulate its aggregation<sup>2</sup>. Additionally, copper cations have been found in high concentrations within senile plaques in the brains of Alzheimer's disease (AD) patients<sup>3</sup>. One of the co-depositors in these plaques is human cystatin C (HCC), which also exhibits amyloidogenic properties due to its flexible structural components. Although the mechanism of amyloidogenic aggregation of human cystatin C is not fully understood, one factor that can accelerate this process are shear forces, which in the model system can be induced by microfluidic *in vivo*-like flow.

Currently, no studies have investigated the amyloidogenic aggregation of human cystatin C both under flow conditions and in the presence of metallic nanoparticles. To address this gap, we applied a microfluidic system that mimics physiological flow conditions to examine the effects of flow dynamics and shear forces on protein aggregation in the presence of NPs.

In parallel, to gain deeper insights into the health implications and underlying mechanisms, we investigated also the interactions between human cystatin C and synthesized by us CuO nanoparticles using atomic force microscopy (AFM), thioflavin T fluorescence assay, circular dichroism (CD), and cell viability assays on SH-SY5Y cell lines. The nanoparticle samples were characterized at the BM29 (BioSAXS) beamline at ESRF (Grenoble, France), while protein aggregation in the presence of NPs under microfluidic flow was studied at the P12 (BioSAXS) beamline at DESY (Hamburg, Germany).

**Acknowledgements:** This project was financed by grant 2021/41/B/ST4/03807 from the National Science Centre (Poland).

- 1. Y. Shi, A. R. Pilozzi, X. Huang, Int. J. Environ. Res. Public Health 17 (2020) 1005.
- 2. A. Abelein, Acc. Chem. Res. 56 (2023) 2653.
- 3. M. A. Lovell, J. D. Robertson, W. J. Teesdale, J. L. Campbell, W. R. Markesbery, J. Neurol. Sci. 158 (1998) 47.



## Influence of gold nanoparticles on amyloid beta aggregation process

J. Maksim<sup>1\*</sup>, K. Rucińska<sup>1</sup>, Z. Pietralik-Molińska<sup>1</sup>, A. Moliński<sup>1</sup>, Ł. Przysiecka<sup>2</sup>, P. Florczak<sup>2</sup> and M. Kozak<sup>1,3</sup>

P25

<sup>1</sup>Department of Biomedical Physics, Faculty of Physics, Adam Mickiewicz University, Poznan, Poland <sup>2</sup>Nanobiomedical Centre, Faculty of Physics, Adam Mickiewicz University, Poznan, Poland <sup>3</sup>SOLARIS National Synchrotron Radiation Centre, Kraków, Poland

\*e-mail: joanna.maksim@amu.edu.pl

Alzheimer's disease (AD) is a progressive neurodegenerative disorder characterized by the gradual deterioration of cognitive abilities, including memory and reasoning, alongside behavioral abnormalities, finally resulting in the patient death [1]. A hallmark of AD pathology is the formation of amyloid plaques, which consist primarily of beta-amyloid  $(A\beta)$  protein aggregates in the brain. These toxic A $\beta$  species arise from improper cleavage of amyloid precursor protein (APP), leading to the buildup of harmful peptides that interfere with neuronal communication and function [2]. Beta-amyloid plays a central role in triggering a cascade of neurodegenerative processes, such as dysregulation of calcium signaling, oxidative damage, and inflammatory responses in the brain [3]. Despite ongoing research into the mechanisms of Aß accumulation, effective therapeutic interventions to halt or reverse AD progression remain elusive. The application of gold nanoparticles (AuNPs) in treating amyloidogenic diseases shows great potential, as the surface chemistry of AuNPs can be precisely tailored to achieve specific binding interactions. This tunability enables AuNPs to serve as effective tools in drug delivery and controlling the aggregation of amyloid proteins, offering a novel approach to managing diseases characterized by amyloid buildup. In this work, three different sized gold nanoparticles were synthesized, and then their effect on the aggregation processes of A<sup>β</sup> 1-42 peptides was studied using P12 (BioSAXS) beamline at PETRA II, DESY (Hamburg, Germany) and fluorescence microplate reader. Gold nanospheres were characterized by UV-Vis dynamic light scattering and inductively coupled plasma mass spectroscopy, spectrometry. Additionally, cytotoxicity tests of fibrils Aß and Au NPs were performed on the neuroblastoma cell line (SHSY-5Y) from a metastatic bone tumor.

**Acknowledgements**: This project was financed by grant 2021/41/B/ST4/03807 from the National Science Centre (Poland).

- 1. Alzheimer's Association, 2021 Alzheimer's disease facts and figures, Alzheimer's & Dementia 17(3) (2021) 327-406.
- 2. Selkoe, D. J., & Hardy, J., The amyloid hypothesis of Alzheimer's disease at 25 years, EMBO molecular medicine 8(6) (2016) 595-608.
- 3. De Strooper, B., & Karran, E., The cellular phase of Alzheimer's disease, Cell 164(4) (2016) 603-615.



**P26** 

## Mn(II) complexes with 2,4-D and MCPA herbicides: IR and XAFS studies

A. Drzewiecka-Antonik<sup>1\*</sup>, P. Rejmak<sup>1</sup>, M. Klepka<sup>1</sup>, D. Osypiuk<sup>2</sup> and A. Wolska<sup>1</sup>

<sup>1</sup>Institute of Physics, Polish Academy of Sciences, 02-668 Warsaw, Poland <sup>2</sup>Faculty of Chemistry, Maria Curie-Skłodowska University, 20-031 Lublin, Poland

### \*e-mail: adrzew@ifpan.edu.pl

Manganese is one of the essential elements for plants, playing several crucial roles in their growth and development. Plants primarily absorb manganese in its ionic form, specifically as Mn(II). This divalent manganese, present in soil and plant tissues, may interact with anions as phenoxyacetate herbicides, forming metal-organic complexes. In 2017, the uptake and transfer of metal ions such as Mn(II) by 2,4-D and MCPA was documented<sup>1</sup>. Therefore, it has become important to study the structure and biological properties of such connections.

In order to get structural information about such compounds, we have synthesized analogous complexes in laboratory conditions<sup>2</sup>. The synthesis gave rise to the formation of hydrated complexes. The fluorescence X-ray analysis as well as elemental and thermal analyses allowed to determine the composition of complexes, namely  $Mn(2,4-D)_2 \cdot 2H_2O$  and  $Mn(MCPA)_2 \cdot 2H_2O$ . The infrared spectra of herbicides and their Mn(II) complexes contain several diagnostic bands that can point out the functional group of herbicide molecules interacting with metal cation<sup>2</sup>. A doublet structure of absorption band corresponding to a carboxyl C=O stretching vibration is observed for acid molecules of herbicides. This band is not observed for Mn(II) complexes, indicating acid deprotonation and stretching bands of deprotonated carboxyl groups are observed on the IR spectra of the complexes. Moreover, the positions of bands, assigned to the C–Oether and C–Cl stretching vibrations suggest that neither O ether nor Cl atoms from herbicide molecules are coordinated to the Mn(II).

The XAFS measurements for the complexes with 2,4-D and MCPA herbicides were performed at ELETTRA synchrotron (Trieste, Italy). The EXAFS analysis (Fig.1) revealed that the complexes forms a polymeric structure with six O atoms bonded to Mn(II) ion. The O atoms originate from four carboxylate groups of herbicide molecules and two  $H_2O$  solvent molecules. The number of carboxylate groups confirmed the presence of four C atoms in the second coordination sphere of Mn(II). The proposed polymeric models of manganese complexes were refined by DFT calculations and verified by XANES.



Figure 1. EXAFS fitting of the experimental oscillations (black line) and the fitting result (red line).

## References

1. E. Skiba, J. Kobyłecka, W.M. Wolf, Environ. Pollut. 220 (2017) 882.

2. A. Drzewiecka-Antonik, W. Ferenc, B. Mirosław, D. Osypiuk, J. Sarzyński, Polyhedron 207 (2021) 115370.



## Support Labs at the MAX IV Synchrotron Facility

Yang Chen

P27

### MAX IV Laborotary, Lund University, Lund, Sweden

\*e-mail: yang.chen@maxiv.lu.se

The Bio and Chem support labs at the MAX IV synchrotron facility in Lund, Sweden, provide essential infrastructure and expertise to enable and enhance scientific research across a wide range of disciplines. These labs serve as central facilities for sample preparation, characterization, and handling, supporting visiting scientists in their experiments at the synchrotron. The Biolab focuses on biological and biochemical samples, such as proteins, nucleic acids, lipids, and macromolecular complexes, and offers specialized equipment including dynamic and static light scattering, gel electrophoresis, UV/Vis spectrometry, chromatography, and optical microscopy. The Chem labs complement this by providing capabilities for handling chemical samples, and catalysis.

The support labs play a critical role in optimizing samples for synchrotron experiments and developing new experimental methods. Collaborations with beamline staff and user communities have led to advancements in areas such as serial crystallography, microfluidic platforms, and sample environments. For example, the Biolab hosts an offline test station for a microfluidic setup used in innovative in situ and time-resolved experiments, as well as a station for testing injector systems for serial crystallography.

Guided by a strategic vision, the Bio and Chem support labs aim to provide exceptional service and maintain state-of-the-art facilities. They work closely with beamlines to optimize workflows and continuously adapt to the evolving needs of synchrotron research. By offering specialized equipment, technical expertise, and innovative solutions, the support labs at MAX IV are integral to enabling groundbreaking research in life sciences, chemistry, and materials science. In this poster, the labs and selected user cases will be showcased to highlight their impact on synchrotron-based research.



## X-Ray Synchrotron Studies of Biomembrane - Protein Interactions at Air-Liquid Interfaces

J. Majewski<sup>1,2,3</sup>

P28

<sup>1</sup>Department of Chemical & Biological Engineering at the University of New Mexico, Albuquerque, USA <sup>2</sup>Visiting Scientist at Los Alamos National Laboratory, Los Alamos, NM, USA <sup>3</sup>Affiliated Professor at the University of Warsaw, Poland

\*e-mail: jarekmajewski60@gmail.com

In nature, lipid membranes perform many living cell functions from selective transport and recognition to simple sequestration. They generally consist of a single phospholipid bilayer or in special cases, such as the lung surfactants, a single monolayer. In the cases discussed here, the lipid membrane will be approximated as a single lipid layer at the air-liquid interface, a surfactant Langmuir layer.

Several surface-sensitive scattering techniques have been developed for probing the structure of such ultra-thin, molecular 2-D arrays of surfactants. These include X-ray reflectometry and in-plane grazing incidence diffraction. Both are particularly challenging to study due to the required horizontality of the sample.

I will illustrate the use of X-ray surface scattering methods to characterize the structures of several types of model membranes. The properties of these soft-condensed, ultra-thin layers are of general interest to a wide scientific audience working in the fields of chemistry and biology since they are relevant to such important areas as bio-mineralization, biosensors, advanced drug delivery systems, and protein-membrane interactions.



# PM10 Under the X-ray: Elemental Composition and Seasonal Variability in the Air in Krakow, Poland P29

Patryk Grzywa<sup>1</sup>, Lucyna Samek<sup>1\*</sup>

<sup>1</sup>AGH University of Krakow, Faculty of Physics and Applied Computer Science, 30-059 Krakow, Poland

\*e-mail: Lucyna.Samek@fis.agh.edu.pl

The research aims to elemental analysis of aerosols samples (PM10). The samples come from two air quality monitoring stations in Krakow, namely Aleje Krasińskiego station and Złoty Róg station. The Aleje Krasińskiego station is a communication station that is located near the road and conducts measurements due to traffic impacts. The Złoty Róg measurement station is an urban background station that is located so that the measurements are representative of the area of several km<sup>2</sup>. The research focused on elemental analysis and observation of seasonal variability of elements. The studied samples come from the summer and winter 2020. The measurements were performed on M4 TORNADO Plus, BRUKER spectrometer, which use the X-ray fluorescence method. The concentrations of the following elements: Na, Mg, Al, P, S, Cl, K, Ca, Ti, Cr, Mn, Fe, Cu, Zn were determined. The Figure 1 below shows the spectrum of identified elements from one of the analyzed PM10 samples. The correlations between concentrations of each elemenets were calculated. All average concentrations of elements that were above the detection limit in the summer and winter seasons are higher for the Aleje Krasińskiego than for Złoty Róg station. This is due to the location of both stations. Particular seasonal variability was observed for S, Cl, Fe. The Enrichment Factor (EF) was calculated, which allows for a preliminary determination of the origin of the element, whether it is of natural or anthropogenic origin.



Figure 1. Identified elements in the PM<sub>10</sub> sample

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# Analysis of in situ localization of elements in the cellular structure of cancers of the head and neck region P30

J. Kubacki<sup>1\*</sup>, J. Paluch<sup>2</sup>, K. Jasik<sup>3</sup> and R. Mroczka<sup>4</sup>

<sup>1</sup>University of Silesia, Institute of Physics, Chorzów, Poland <sup>2</sup>Medical University of Silesia, Katowice, Poland <sup>3</sup>University of Jan Długosz, Częstochowa, Poland

<sup>4</sup>John Paul II Catholic University of Lublin, Lublin, Poland

\*e-mail: jerzy.kubacki@us.edu.pl

The formation of free oxygen radicals is part of metabolic pathways under physiological and pathological conditions (e.g. oncogenesis) in the human body. Maintaining the balance between their formation and elimination gives the cells homeostasis. The focus of our research points at Fe, Mn, Zn and Cu, which are essential cofactors in a number of enzymes critical in cell metabolism. Reactive forms of oxygen, such as superoxide can cause mutations in DNA or attack enzymes that make amino acids and other essential molecules. To combat this potential danger, most cells produce an enzyme that detoxifies superoxide superoxide dismutase (SOD). Recent studies of SOD are aimed at understanding its role in various diseases, in case of our studies cancer of the head and neck region. The main role in the redox processes is played by Fe ions, although Cu ions, which are components of the cytochrome molecule, are also indicated as the important player [1]. Other components of SOD, such as Zn and Mn, which are part of this enzyme and may play a significant role in processes related to aerobic combustion in healthy cells, cannot be excluded either. In the case of anaerobic combustion, which is predominant in tumor tissue, the elements included in SOD should not be present.

Differentiation between iron oxidation states in biological systems can is achievable using XAS spectroscopy [2]. Studies of absorption edges of 3d transition metals was realized for biomolecules [3], heme [4], human brain tumor [5], ovarian cancer [6] and major organs of tumor-bearing mice [7]. Preliminary studies of cancer tissues were recently performed on XAS beam line in Solaris in the FLY (fluorescence yield) mode.

The main goal of our study is to recognize differences between the standardized ancer cells, cells from tissue in original form and homogenate. We expect to find variation of the oxidation state of iron on states of oxidation cycle (including cytochrome oxidase) and presence of other ions included in SOD for normal tissue.

The investigations were executed on iron  $L_{2,3}$  absorption edge for two various methods of tissue preparation. Obtained results allowed detection of iron in cancer and healthy tissue with distinguishes of Fe<sup>3+</sup> and Fe<sup>2+</sup> states, as shown below (results from FLY mode). Imaging of normal and cancerous tissue was carried out on the Demeter beam line at the Solaris synchrotron in Krakow at the STXM terminal station.

- 1. Mol Aspects Med. 26(4-5) (2005) 313-27, DOI: 10.1016/j.mam.2005.07.010
- 2. Chem. Rev. 117 (2017) 13909–13934, DOI: 10.1021/acs.chemrev.7b00213
- 3. J. Am. Chem. Soc. 139(2017) 1215-1225, DOI: 10.1021/jacs.6b11260
- 4. J. Am. Chem. Soc. 129 (1) (2007) 113–125, DOI: 10.1021/ja065627h
- 5. Metallomics 5 (2013) 1547, DOI: 10.1039/c3mt00158j
- 6. J. Anal. At. Spectrom. 33 (2018) 1638, DOI: 10.1039/c8ja00140e
- 7. J Int Med Res. 46(1) (2018) 70-78, DOI: 10.1177/0300060517718711



## Degradation and regeneration analysis of noble metal ex-solution catalysts for dry and steam methane reforming P31

M. Dominów<sup>1\*</sup>, B. Bochentyn<sup>1</sup>, K. Kujawska<sup>1</sup>, Y.-F. Hsu<sup>2</sup>, S.-F. Wang<sup>2</sup> and J. Karczewski<sup>1</sup>

<sup>1</sup>Lord Howe Island University, Lagoon Road 1, Lord Howe Island NSW 2898, Australia <sup>2</sup>European Synchrotron Radiation Facility, B.P.220, F-38043 Grenoble Cedex, France

\*e-mail: presenting\_author@det.nsw.edu.au

Dry Methane Reforming (DRM) and Steam Methane Reforming (SRM) are among the proposals for reliable and environmentally friendly hydrogen production methods. However, these two processes are highly driven by temperature and can be enhanced by the proper catalysts. Coking is a major problem for conventional catalytic materials, especially in DRM cases<sup>2</sup>. As a solution to this problem, metal ex-solved nanoparticles on the surface of the perovskite material were proposed as promising catalysts<sup>2,3</sup>. Platinum and ruthenium are both of great interest in DRM and SRM. With use of noble metals arises the problem of scarcity and materials price. We propose to mitigate this by employing regeneration procedures, which can extend the lifetime of already-durable catalysts to even longer times.

Catalysts were synthesized by modified Pechini method and characterized by X-Ray Diffraction (XRD) and Scanning Electron Microscopy studies (SEM). Catalytic activity and durability tests were carried out. SEM imaging of the ex-solved ruthenium particles and methane conversion ratio in dry reforming setup are shown on figures below.



Fig 1. SEM image of ruthenium nanoparticles (left) and efficiency in the dry methane reforming process for different Ru doping levels (right)

The regeneration procedure was studied by employing synchrotron radiation for low- energy X-Ray Absorption (XAS) and X-Ray Photoelectron spectroscopy (XPS) at PHELIX beamline in Solaris, Kraków.  $(La_{0.3}Sr_{0.7})_{0.9}Ti1-xPt_xO_3+\delta$  and  $(La_{0.3}Sr_{0.7})_{0.9}Ti1-xRu_xO_3+\delta$  polycrystalline perovskite samples for different doping levels were investigated. Research was concentrated on exploring ex-solution characteristics, degradation of the samples after durability tests and effectiveness of regeneration procedures.

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### References

1. Prameswari, J., & Lin, Y.-C., Innovative catalysis approaches for methane utilization. ACS EST Engg. 5 (2) (2025) 325–343. https://doi.org/10.1021/acsestengg.4c00700

2. Schrenk, F., Lindenthal, L., Drexler, H., Urban, G., Rameshan, R., Summerer, H., Berger, T., Ruh, T.K., Opitz, A.K., & Rameshan, C., Impact of nanoparticle exsolution on dry reforming of methane: Improving catalytic activity by reductive pre-treatment of perovskite-type catalysts. Applied Catalysis B: Environmental 318 (2022) 121886. https://doi.org/10.1016/j.apcatb.2022.121886

3. Kim, Y., Kim, H.S., Kang, D., Kim, M., & Lee, J.W., Enhanced redox performance of LaFeO3 perovskite through in-situ exsolution of iridium nanoparticles for chemical looping steam methane reforming. Chemical Engineering Journal, 468 (2023) 143662. https://doi.org/10.1016/j.cej.2023.143662



# Electronic structure of GeS<sub>0.5</sub>Se<sub>0.5</sub> crystal: band alignment, valence bands, and core-levels

P32

A. Sabik<sup>1,2\*</sup>, M. Grodzicki<sup>3,4</sup>, J.M. Skelton<sup>5</sup>, S. Gorantla<sup>4</sup>, A. Trembułowicz<sup>2,6</sup>, M.J. Smiles<sup>7</sup>, T. Sobol<sup>1</sup>, E. Beyer<sup>1</sup>, B. Wolanin<sup>1</sup>, R. Kudrawiec<sup>3,4</sup>, T.D. Veal<sup>7</sup> and W.M. Linhart<sup>3</sup>

<sup>1</sup>National Synchrotron Radiation Centre SOLARIS, Jagiellonian University, 30-392 Krakow, Poland

<sup>2</sup>University of Wroclaw, Department of Physics and Astronomy, Institute of Experimental Physics, 50 - 204 Wroclaw, Poland

<sup>3</sup>Wroclaw University of Science and Technology, Department of Semiconductor Materials Engineering, 50-370 Wroclaw, Poland

 <sup>4</sup>Łukasiewicz Research Network – PORT Polish Center for Technology Development, 54-066 Wrocław, Poland <sup>5</sup>University of Manchester, Department of Chemistry, Oxford Road, Manchester, M13 9PL, UK
 <sup>6</sup>AGH University of Krakow, Academic Centre for Materials and Nanotechnology, 30-059 Krakow, Poland <sup>7</sup>University of Liverpool, Stephenson Institute for Renewable Energy and Department of Physics, <sup>1</sup>Liverpool, Liverpool, Stephenson Institute for Renewable Energy and Department of Physics,

Liverpool, L69 7ZF, UK

\*e-mail: agata.sabik@uj.edu.pl

The GeS<sub>x</sub>Se<sub>1-x</sub> materials are semiconducting alloys considered as promising absorbers in photovoltaic.<sup>1</sup> Although, the high applicable potential of GeS<sub>x</sub>Se<sub>1-x</sub>, many of their fundamental optoelectronic properties are poorly explored. We present comprehensive experimental and theoretical investigations on the electronic structure of the GeS<sub>0.5</sub>Se<sub>0.5</sub> crystal. As confirmed by transmission electron microscopy and synchrotron radiation based X-ray photoelectron spectroscopy (SR-XPS), the studied crystals, prepared by melt-growth technique, exhibit high and crystalline quality and phase purity. By combination of XPS with the optical absorption and photoreflectance spectroscopy, the band energy diagram of GeS<sub>0.5</sub>Se<sub>0.5</sub> is driven. Herein, the ionization potential of 5.7 eV is determined based on XPS investigations, whereas the optical spectra reveal the direct character of the fundamental optical bandgap with the energy of

1.37 eV at room temperature. The optical transition is highly sensitive to the incident light beam polarization, which indicates the anisotropy of the optical properties. The total density of states obtained by calculations based on density functional theory is in a reasonable agreement with the valence band spectra measured by SR-XPS. The theoretical results highlight the presence of Ge 4s states at the valence band edge desirable for photovoltaic absorbers.

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## References

1. M.J. Smiles et al., J. Mater. Chem. A 9 (2021) 22440-22452.



## NAP-XPS - new end station at SOLARIS synchrotron

A. Sabik<sup>1\*</sup>, T. Sobol<sup>1</sup>, E. Beyer<sup>1</sup>, B. Wolanin<sup>1</sup>, A. Lewera<sup>2</sup>, M. Zając<sup>1</sup> and A. Wach<sup>1</sup> P33

<sup>1</sup>National Synchrotron Radiation Centre SOLARIS, Jagiellonian University, 30-392 Krakow, Poland <sup>2</sup>Faculty of Chemistry, University of Warsaw, 02-089 Warsaw, Poland

### \*e-mail: agata.sabik@uj.edu.pl

The continuous advancement of X-ray photoelectron spectroscopy (XPS) systems has significantly increased their capabilities far beyond the standard of ultrahigh vacuum (UHV) conditions. As a result, near ambient pressure X-ray photoelectron spectroscopy (NAP-XPS), which allows sample analysis at relatively high pressures in the range of millibars, has become a powerful tool with a wide variety of applications.<sup>1</sup> On the one hand, the NAP-XPS allows to study the unconventional materials, including moderately volatile liquids, biological samples, porous or polymeric materials, which are not UHV compatible. On the other hand, it enables operando investigations of processes occurring at solid-gas, solid-liquid, or liquid-gas interfaces, opening new avenues for research.

A new NAP-XPS end station, designed for studies of solid samples in the presence of a gaseous atmosphere, will be assembled at PHELIX beamline<sup>2</sup> in the SOLARIS facility.<sup>3</sup> PHELIX is a soft X- ray beamline featuring two branches dedicated to complementary photoemission investigations. Currently, only one branch is in operation intended for experiments under UHV conditions. The ongoing development of the beamline covers the second branch construction and its integration with the NAP-XPS system. The NAP-XPS end station will be dedicated to in situ and operando XPS experiments in the presence of a controlled gaseous atmosphere within mbar pressure range as well as under UHV conditions, with the sample heating up to 1000°C. To probe the interfaces between solids and gases at the ambient pressures, the end station will be equipped with an ambient pressure (AP) cell. The solid samples will be placed at the AP cell docked to the electron energy analyser during the experiments, wherein the defined gas will be dosed. The NAP-XPS end station will offer the XPS investigations with high temporal resolution together with simultaneous infrared reflection absorption spectroscopy measurements and analysis of the outlet gas composition by mass spectrometry. Such combination of experimental techniques will allow to follow the chemical reaction and probe the geometrical, chemical, and electronic catalyst structure relate to catalyst function. The first planned user experiments are scheduled for second half of 2027.

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- 1. J. Schnadt, J. Knudsen, N. Johansson, J. Condens. Matter Phys. 32 (2020) 413003.
- 2. M. Szczepanik-Ciba, T. Sobol, J. Szade, Nucl. Instrum. Methods Phys. Res. 492 (2021) 49-55.
- 3. J. Szlachetko et al., Eur. Phys. J. Plus 138 (2023) 10.



## PHELIX beamline as an advanced tool dedicated to surface science

B. Wolanin<sup>1\*</sup>, T. Sobol<sup>1</sup>, E. Beyer<sup>1</sup> and A. Sabik<sup>1,2</sup>

**P34** 

<sup>1</sup>National Synchrotron Radiation Centre SOLARIS, Jagiellonian University, 30-392 Krakow, Poland <sup>2</sup>University of Wroclaw, Department of Physics and Astronomy, Institute of Experimental Physics, 50-204 Wroclaw, Poland

\*e-mail: barbara.wolanin@uj.edu.pl

Phelix is one of seven research beamlines located at the National Synchrotron Radiation Center SOLARIS in Krakow. The PHELIX end station is dedicated to surface science providing various spectroscopic techniques, using an polarizing undulator as a radiation source for soft X-rays in the energy range of 50 eV to 1800 eV.

The PHELIX end station is a multi-chamber vacuum system equipped with, among others, a hemispherical Phoibos 225 energy analyzer, an advanced preparation chamber, and a station for cleaving samples. Available measurement techniques include: XPS (X-ray Photoelectron Spectroscopy), ARPES (Angle Resolved Photoelectron Spectroscopy), XAS (X-ray Absorption Spectroscopy), and XMCD (X-ray Magnetic Circular Dichroism). Regarding the needs of the user community, the beamline team intends to extend the research capabilities of the PHELIX beamline with SR-ARPES (Spin Resolved - Angle Resolved Photoelectron Spectroscopy), soon. PHELIX's end station enables the use of complementary techniques and tools both for preliminary sample characterization and in situ creation of completely new research systems (i.e Low Energy Electron Diffraction and Molecular Beam Epitaxy, by a system of various vapor sources).

The very advanced system operating in ultra-high vacuum with advantages such as tunable energy and polarization of synchrotron light allows for studying of chemical, electronic, and magnetic properties of various surface systems or interfaces. The station's spectrum of research capabilities is best reflected in user community publications, which touch upon such issues as: comparison of electronic properties in NZFO/f-MWCNTs nanocomposite systems obtained by ex-situ and in-situ synthesis [1], or explanation of the influence of the defected  $TiO_2$ /perovskite interface on optoelectronic properties [2].

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- 1. A. Bajorek et al., Journal of Magnetism and Magnetic Materials, 585 (2023) 171150.
- 2. C. Das et al., ACS Applied Materials and Interfaces, 15 (2023) 56500.



## Spectroscopic Synergy at the ASTRA Beamline: Combining X-ray Absorption and Raman Spectroscopy for Advanced Analysis of Complex Materials P35

L. Alluhaibi<sup>1\*</sup>, A. Maximenko<sup>1</sup>, G. Gazdowicz<sup>1</sup>, D. Zalka<sup>1</sup>, M.Brzyski<sup>1</sup>, M. Piszak<sup>1</sup>, H. Lichtenberg<sup>2</sup>, A. Prange<sup>2</sup> and J. Hormes<sup>3</sup>

<sup>1</sup>Solaris National Synchrotron Radiation Centre, Jagiellonian University, 30-392 Kraków, Poland <sup>2</sup>Hochschule Niederrhein University of Applied Sciences, Krefeld, Germany <sup>3</sup>Institute of Physics, Rheinische Friedrich-Wilhelms-Universität, Bonn, Germany

\*e-mail: lulu.alluhaibi@uj.edu.pl

Combining X-ray Absorption Spectroscopy (XAS) with vibrational techniques like Raman spectroscopy provides a powerful approach for studying structural and chemical changes in materials, particularly catalysts. Simultaneously recording XAS and Raman data enables the observation of structural transformations in solid materials while monitoring adsorbed species on their surfaces, offering a more comprehensive understanding of dynamic processes.



Figure 1. Preliminary results recorded at the ASTRA beamline: (A) S K-edge XANES spectra acquired in transmission mode at room temperature under 30 Torr  $N_2$ ; (B) Raman spectra collected using a 785 nm laser at room temperature.

Raman spectroscopy is a versatile tool for in situ studies of chemical structures and interactions in gases, liquids, solids, and interfaces. When combined with XAS, it provides detailed correlations between the local electronic structure (XAS) and molecular vibrations and bonding environment (Raman). This integration is particularly valuable for investigating complex materials, where subtle changes in local structure can significantly impact overall properties. While XAS probes oxidation states, coordination geometry, and molecular frameworks, Raman spectroscopy reveals vibrational characteristics, allowing for cross-validation and deeper insights into chemical states and bonding environments.

In July 2024, a Raman spectrometer with two laser sources (782 nm and 532 nm) was successfully installed at the XAS beamline ASTRA (SOLARIS synchrotron). This advancement enables making XAS and Raman measurements at the beamline during one beamtime, providing a unique and powerful tool for comprehensive material analysis. Once fully integrated, this capability will significantly enhance research possibilities, particularly for studying complex materials and processes under real-world conditions in the tender energy range. This presentation will discuss the technical aspects of the beamline, along with selected results from ex situ and in situ experiments.

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# Tailoring the ceria properties by lanthanide and transition metal co- doping

**P36** 

K. Kujawska<sup>1\*</sup>, S. Amatori<sup>2</sup>, A. Ducka<sup>1</sup>, M. Dominów<sup>1</sup>, P. Błaszczak<sup>1</sup>, A. Witkowska<sup>1</sup>, K. Sowa<sup>3</sup>, P. Korecki<sup>3</sup>, F. d'Acapito<sup>4</sup> and B. Bochentyn<sup>1</sup>

<sup>1</sup>Advanced Materials Center, Faculty of Applied Physics and Mathematics, Gdańsk University of Technology, 80-233 Gdańsk, Poland <sup>2</sup>CERIC-ERIC c/o ESRF LISA CRG, 38000, Grenoble, France <sup>3</sup>National Synchrotron Radiation Centre SOLARIS, Jagiellonian University, 30-392, Kraków, Poland <sup>4</sup>CNR-IOM Grenoble c/o ESRF LISA CRG, 38000, Grenoble, France

\*e-mail: kinga.kujawska@pg.edu.pl

Ceria compounds can be used as anode materials in Solid Oxide Fuel Cells to improve their catalytic performance and stability in alternative fuels. In this work the properties of five different ceria compounds co-doped with elements from lanthanide and transition metal groups were investigated. The following compositions:  $Ce_{0.9}La_{0.05}Ni_{0.05}O_{2-\delta}$ ,  $Ce_{0.9}La_{0.05}Cu_{0.05}O_{2-\delta}$ ,  $Ce_{0.9}La_{0.05}Co_{2-\delta}$ ,  $Ce_{0.9}La_{0.05}Co_{2-\delta}$ ,  $Ce_{0.9}La_{0.05}Fe_{0.05}O_{2-\delta}$  were synthesized using the Pechini method and reduced in dry H2 to verify the possibility of forming metal nanoparticles on the surface of the material. The microstructure of the compounds was characterized with a Scanning Electron Microscopy and the phase composition of synthesized materials was analysed with the X-ray Diffraction method. BET analysis was also performed to determine the specific surface area for each compound, as well as the reducibility of particular compositions was investigated using the TPR/TPO method. Additionally, the compounds were examined at the National Center of Synchrotron Radiation on the PolyX line, where the X-ray microimaging and X-ray microspectroscopy were performed.

The SEM analysis of as prepared samples confirms their porous structure and fine crystallinity. After reduction, the formation of round-shape nanoparticles is visible on the surface of grains and in the grain boundaries. They may act as catalytic centers in SOFC anodes. Furthermore, the XRD analysis before reduction showed the presence of NiO phase and after reduction in  $H_2$ , the signal from the metallic Ni phase was present. Depending on the type of dopant, there is a clear difference in the amount, shape and distribution of forming particles. This work aims to compare the properties of these compounds and also to determine if the metal is built into the ceria lattice or it rather deposits in the form of oxides on the lattice and/or in the grain boundaries.

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# The Scanning Transmission X-Ray Microscope at SOLARIS optimized for fast scanning and environmental experiments P37

K. Matlak<sup>1,\*</sup> T. Strączek<sup>1</sup>, M. Brzyski<sup>1</sup>, B. Wolanin,<sup>1</sup> A. Mandziak<sup>1</sup>, P. Nita<sup>1,2</sup>, M. Zając<sup>1</sup> and T. Tyliszczak<sup>1,3</sup>

<sup>1</sup>SOLARIS National Synchrotron Radiation Centre, Jagiellonian University, Kraków, Poland <sup>2</sup>Faculty of Physics Astronomy and Applied Computer Science, Jagiellonian University, Kraków, Poland <sup>3</sup>Lawrence Berkeley National Labotatory, Berkeley CA94720, USA

\*e-mail: krzysztof.matlak@uj.edu.pl

The SOLARIS National Synchrotron Radiation Centre is a 1.5 GeV 3<sup>rd</sup> generation synchrotron located in Krakow, Poland. As a low energy storage ring, this facility is going to specialize in building its beamlines mostly in the soft X-ray region. One of them is the DEMETER beamline, on which the Scanning Transmission X-ray Microscopy (STXM) is located. This beamline is a classic Plane Grating Monochromator (PGM) design with an Elliptically Polarizing Undulator (EPU) as a source and works in 100-2000 eV photon energy range. The STXM microscope has been designed and built in-house. The main goal of the design is to provide a flexible microscope that can be easily reconfigured to a specific experiment (for materials engineering, Earth and environmental science, biology, electrochemistry, etc., when ultra-high vacuum conditions are not required). Microscope's relatively compact chamber enables faster sample load/unload and more economical purging with pure He, in case a sample cannot be subjected to vacuum.



This STXM design is based on the Advanced Light Source (ALS) beamline 5.3.2 STXM [1] but there is a significant difference. The microscope does not have sample stages in the *z* direction (along the X-ray beam). It has the Order Sorting Aperture (OSA) motion in this direction, instead. This modification reduces sample vibration and helps in the focusing procedure. Additionally, a custom high efficiency Field Programmable Gate Array (FPGA) is used there for timing, acquisition and fast shutter control.

Figure 1. The STXM end station in SOLARIS

This STXM is operating on a modern undulator beamline, therefore acquisition time of a singlepixel can be even shorter than 1ms, enabling fast scanning and low radiation damage. To maintain the sample position at a level of several nm, especially with changing X-ray energy, we use a laser interferometer in a closed feedback loop. This STXM design is suitable for a commercial electrochemical cell system manufactured by Hummingbird Scientific, in which "operando-study" experiments can be performed using a potentiostat and a microfluidic pump. Thanks to the use of an undulator with variable polarization, it is also possible to perform measurements using the XMCD or XMLD effect for magnetic samples.

Apart of our STXM design and capability details, some example results of interesting experiments will be presented.

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### **References:**

1. A.L. D. Kilcoyne, T. Tyliszczak, W.F. Steele, S. Fakra, P. Hitchcock, K. Franck, E. Anderson, B. Harteneck, E.G. Rightor, G.E. Mitchell, A.P. Hitchcock, L. Yang, T. Warwick, and H. Ade, J. Synchrotron Rad. 10 (2003) 125–136.



## Understanding of mechanism of Cu reduction induced by photon beam in Cu thin films

A. Figura-Jagoda<sup>1\*</sup>, C. Hain<sup>2</sup>, S. Klejna<sup>1</sup>, K. Maćkosz<sup>2</sup>, C.Minzoni<sup>2</sup>, J.Stępień<sup>1,3</sup>, A. Szkudlarek<sup>1</sup>, I. Utke<sup>2</sup>, M. Zając<sup>3</sup> and M. Sikora<sup>1,3</sup>

<sup>1</sup>Academic Centre for Materials and Nanotechnology AGH, 30-055 Kraków, Poland <sup>2</sup>Empa - Swiss Federal Laboratories for Materials Science and Technology, 3603 Thun, Switzerland <sup>3</sup>SOLARIS National Synchrotron Radiation Centre , 30-392 Kraków, Poland

\*e-mail: afigura@agh.edu.pl

X-ray absorption spectroscopy is a technique that finds numerous applications in materials research providing key information for understanding the chemistry of investigated structures. Its high sensitivity is advantageous in study of thin films and nanostructures. Unfortunately, during investigation of nanoscale objects with XAS, artifacts can appear due to irradiation with intensive X-ray beam, such as evaporation of the film due to local heating [1] or reduction of effective charge of some elements [2,3]. Although often reported and effectively circumvented during regular measurements understanding these effects is still incomplete.

In XAS study we have observed that shape of L-edge of copper in thin films exposed to focused synchrotron beam shows a strong reduction in the intensity of Cu-II feature and increase in the intensity of spectral features corresponding to Cu-I and metallic copper (Figure 1). Our measurements showed that this effect is strongly associated with the intensity of photon beam, but can also depend on the film thickness, substrate, and morphology dependent on deposition method. On the other hand it seems to be independent from the energy of photon beam. As long as in the typical measurement procedure the signal is collected from the same spot for different elements/edges, this reduction effect will play significant role and can lead to misinterpretation of the results. Better understanding of the mechanism of Cu photoreduction shall help providing procedures for preventing or minimizing X-ray induced effects in XAS study of nanostructures.



Figure 1. Consecutive Cu L3-edge spectra of thin film collected with FY (left) and TEY (right). Early scans (light blue) shows the spectral shape corresponding to high amount of Cu-II, opposite to the late ones (dark blue).

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### References

- 1. E.L. Bright, et al., J. Synchrotron Rad. 28 (2021) 1377–1385.
- 2. L. Monico, et al., Anal. Chem. 92 (2020) 14164–14173.
- 3. F. Stellato, et al., Metallomics 11 (2019) 1401.

P38



## Time-Resolved X-ray Diffraction Investigation of Ultrafast Structural Changes in Fe

P39

O. Liubchenko<sup>1,\*</sup>, J. Antonowicz<sup>2</sup>, K. Sokolowski-Tinten<sup>3,4</sup>, P. Zalden<sup>5</sup>, R. Minikayev<sup>1</sup>,
I. Milov<sup>6,7,8</sup>, T. J. Albert<sup>3</sup>, C. Bressler<sup>5,9,10</sup>, M. Chojnacki<sup>1</sup>, P. Dłużewski<sup>1</sup>, P. Dzięgielewski<sup>2</sup>,
A. Rodriguez-Fernandez<sup>5</sup>, K. Fronci<sup>11</sup>, W. Gawełda<sup>12,13,14</sup>, K. Georgarakis<sup>15</sup>, A.L. Greer<sup>16</sup>,
I. Jacyna<sup>1</sup>, R.W.E. van de Kruijs<sup>8</sup>, R. Kamiński<sup>17</sup>, D. Khakhulin<sup>5</sup>, D. Klinger<sup>1</sup>, K. Kosyl<sup>1</sup>,
K. Kubicek<sup>5,9,10</sup>, A. Olczak<sup>2</sup>, N.T. Panagiotopoulos<sup>16</sup>, M. Sikora<sup>18</sup>, P. Sun<sup>19</sup>, H. Yousef<sup>5</sup>,
W. Zajkowska-Pietrzak<sup>1</sup>, S. Kryvyi<sup>1,20</sup> and R. Sobierajski<sup>1</sup>

<sup>1</sup>Institute of Physics, Polish Academy of Sciences, 02668 Warsaw, Poland <sup>2</sup>Faculty of Physics, Warsaw University of Technology, 00-662 Warsaw, Poland <sup>3</sup>Faculty of Physics, University of Duisburg-Essen, 47048 Duisburg, Germany <sup>4</sup>Center for Nanointegration Duisburg-Essen (CENIDE), University of Duisburg-Essen, 47048 Duisburg, Germany <sup>5</sup>European XFEL, 22869 Schenefeld, Germany <sup>6</sup>Advanced Research Center for Nanolithography (ARCNL), 1098 XG Amsterdam, the Netherlands <sup>7</sup>Center for Free-Electron Laser Science CFEL, Deutsches Elektronen-Synchrotron DESY, 22607 Hamburg, Germany <sup>8</sup>Industrial Focus Group XUV Optics, MESA+Institute for Nanotechnology, University of Twente, 7522 NB Enschede, the Netherlands <sup>9</sup>Department of Physics, Universitat Hamburg, 22761 Hamburg, Germany <sup>10</sup>The Hamburg Centre for Ultrafast Imaging, 22761 Hamburg, Germany <sup>11</sup>Research Centre MagTop, Institute of Physics, Polish Academy of Sciences, 02668 Warsaw, Poland <sup>12</sup>Department of Chemistry, Universidad Autonoma de Madrid, Ciudad Universitaria de Cantoblanco, 28049 Madrid, Spain <sup>13</sup>IMDEA Nanociencia, 28049 Madrid, Spain <sup>14</sup>Faculty of Physics, Adam Mickiewicz University, 61-614 Poznan, Poland <sup>15</sup>School of Aerospace, Transport and Manufacturing, Cranfield University, Cranfield, MK43 0AL, UK <sup>16</sup>Department of Materials Science & Metallurgy, University of Cambridge, Cambridge, CB3 0FS, UK <sup>17</sup>Department of Chemistry, University of Warsaw, 02-089 Warsaw, Poland <sup>18</sup>Academic Centre for Materials and Nanotechnology, AGH University of Krakow, 30-059 Krakow, Poland <sup>19</sup>Dipartimento di Fisica e Astronomia "Galileo Galilei", Universita degli Studi di Padova, Padova 35131, Italy

<sup>20</sup>Institute for Nanoscience and Engineering, University of Arkansas, Fayetteville, AR 72701, USA

\*e-mail: liubchenko@ifpan.edu.pl

We have studied the rapid structural transformations in thin Fe films after pulsed laser annealing. Time resolved x-ray diffraction with femtosecond X-ray pulses at the EuXFEL facility was used to characterize the kinetics of the crystallization from the melted phase. Pulsed laser induced melting on the picosecond time was studied for various metals (e.g. Au, Pd)<sup>1, 2</sup>. However, the subsequent ultrafast cooling processes remain poorly studied.

Analysis of the changes in the integrated intensities of the Bragg peaks and liquid "halo" scattering peak allow to describe the kinetics of crystallization in Fe thin films during ultrafast cooling. The isothermal crystallization of the Fe layers in the partial melting regime for various pump laser energies is investigated. The high temperature bcc  $\delta$ -Fe phase is crystallized from liquid within 1 nanosecond time scale. Independent of the deposited energy density, the constant nucleation rate is observed following JMAK (Avrami) analysis.

These first experimental findings offer insights into the crystallization process of monoatomic Fe metal during ultrafast cooling.


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#### References

- 1. J. Antonowicz et al., Acta Materialia 276 (2024) 120043.
- 2. M. Z. Mo et al., Science 360 (2018) 1451-1455.



### The ESRF After the EBS Upgrade: A New Research Opportunities

M. Jankowski1\*

**P40** 

<sup>1</sup>The European Synchrotron Radiation Facility – ESRF, Grenoble , 38043 France

### \*e-mail: maciej.jankowski@esrf.fr

With its recent Extremely Brilliant Source (EBS) upgrade<sup>1</sup>, the ESRF has significantly increased the brilliance of the synchrotron radiation produced, enhancing the data quality and reducing measurement times. This advancement allows researchers to conduct previously unattainable experiments, solidifying the ESRF's position as a leader in cutting-edge research.

The facility operates advanced beamlines equipped to handle a variety of experiments using techniques such as X-ray diffraction, scattering, spectroscopy, imaging, and tomography. These capabilities enable researchers to explore matter at atomic and molecular scales while offering the ability to investigate larger scales through tomography. This range of techniques allows for a comprehensive understanding of materials, from their fundamental building blocks to their larger structural and dynamic behaviors.

The ESRF operates on a competitive beamtime allocation model, prioritizing innovative and impactful research projects from various fields. Potential scientific researchers are encouraged to apply for beamtime at the ESRF and utilize this exceptional resource to advance their scientific inquiries. Whether exploring new materials, studying complex biological systems, or probing fundamental processes, the ESRF—bolstered by the enhanced capabilities of the EBS— provides the tools and expertise needed to achieve breakthrough results. Additionally, the ESRF welcomes industrial research collaborations with industrial partners, further expanding opportunities for innovative research.

### References

1. Raimondi, P., Benabderrahmane, C., Berkvens, P. et al. The Extremely Brilliant Source storage ring of the European Synchrotron Radiation Facility. Commun Phys 6 (2023) 82.

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