ESRF
USER MEETING

3-5 February
2020

3 FEBRUARY

8 Tutorials for users

4 FEBRUARY

Plenary Session day

5 FEBRUARY

3 User-Dedicated
Microsymposia

CONTENTS

ESRF USER ORGANISATION

ESRF
The European Synchrotron

EPN Science Campus

71 avenue des Martyrs,
38000 Grenoble

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User Meeting 2020

Monday 3 February – Tutorials

T1. SB: Serial crystallography data processing & BAG Meeting
T2. XAS data analysis
T3. X-ray Photon Correlation Spectroscopy to study dynamical properties of matter
T4. Introduction to coherent X-ray imaging techniques and data analysis
T5. Volume image analysis of tomographic data
T6. SAXS analysis strategies & softwares
T7. Laue microdiffraction
T8. PyFAI - Data reduction tools for scattering experiments: Application to SAXS and WAXS

Tuesday 4 February – Plenary Session

Venue: ESRF Auditorium

Administrative Assistants: Catherine Blanc – Agnès Carlet – Sonya Girodon

Tel: +33 (0)4 76 88 23 58 / 25 52 / 28 80 – usermeet@esrf.fr

Wednesday 5 February – Microsymposia

UDM1 In situ and operando X-ray absorption spectroscopy for the study of catalysts and functional materials
Venue: **ILL Chadwick Amphitheatre**
contact: udm1-um20@esrf.fr
Administrative Assistant: Eva Jahn-Feppon
Tel: +33 (0)4 76 88 26 19

UDM2 Nanomaterials life cycle: from nanoengineering to public health
Venue: **IBS Seminar Room**
contact: udm2-um20@esrf.fr
Administrative Assistant: Sonya Girodon
Tel: +33 (0)4 76 88 28 80

UDM2 Multi-crystal and serial data collection in Structural Biology
Venue: **ESRF Auditorium**
contact: udm3-um20@esrf.fr
Administrative Assistant: Claudine Romero
Tel: +33 (0)4 76 88 20 27

Practical information

Badge information

You have been given a **BADGE** at the site entrance: **this badge is strictly personal and due to the strengthened security measures, you MUST wear your badge (and its purple lanyard) at all times on site over the whole period of the event**. It is your pass for the site entrance and, on site, for the Guesthouse and the canteen. **Please note that the access to the Experimental Hall is strictly forbidden.**

Lunches

Lunches **are served** from

11:30 - 13:30

ground floor - restaurant

11:30 - 13:00

1st floor - restaurant

Please present your **BADGE** to the cashier.

Monday 3 February

Soirée Bistrot

18:30 – 20:30

under the marquee

Tuesday 5 February

Poster Session & Cocktail

18:00 – 19:30

under the marquee

followed by the

User Meeting Dinner

19:30 – 22:00

at ESRF / ILL Restaurant

Bus transfer from ESRF > Centre of Grenoble

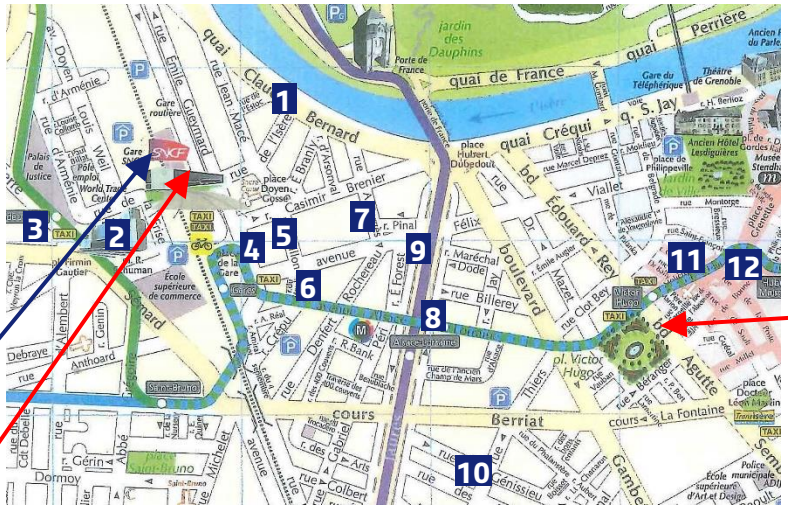
TUESDAY 4 February (after the dinner) > **a bus will leave the ESRF at 22:00**
With **TWO STOPS ONLY** (see map below)

HOTELS

1 Hotel Ibis Gare
2 Novotel Centre
3 Hotel Europole
4 Residhotel Central'Gare
5 Hotel Institut
6 Kyriad direct Grenoble centre - *Hotel des Alpes*

Train Station
(SNCF)

STOP 1
In front of the train station



HOTELS

7 Hotel Gloria
8 Hotel Touring
9 Hotel Ibis Style Gare
10 Splendid Hotel
11 Novotel Centre
12 Hotel de l'Europe

STOP 2
Place Victor Hugo

TAXI GRENOBLOIS
+33 (0)4 76 54 42 54

For further information, please contact: Catherine Blanc, Agnès Carlet, Sonya Girodon
ESRF Central Building – Room 004 (ground floor) – Tel: +33 (0)476 88 23 58 / 25 52 / 28 80

List of exhibitors

We thank the following commercial exhibitors for their presence at the 2020 ESRF User Meeting

Agilent

Trusted Answers

Exhibitors attending the 2020 ESRF User Meeting, 3 - 5 February 2020, Grenoble, France

Added Value Solutions AVS Xixilion 2, Bajo. Pabellón 10 Poligono de Sigma 20870 Elgoibar Spain	Pedro Noguera Tel: +34 (0) 661325019 pnoguera@a-v-s.es
Agilent Technologies France 3 Avenue du Canada Parc Technopolis – Z.A. Courtaboeuf C.S. 90263 91978 Les Ulis Cédex France	Yann Lacroix Lagrandeur Tel: +33 (0) 685066860 yann.lacroix@agilent.com
Altec Equipment 51 Square des Feuillants 78150 Le Chesnay France	Arnaud Aubert Tel: +33 (0) 632420769 a.aubert@altec-equipment.com
Attocube Systems AG Eglfinger Weg 2 85540 Haar Germany	Philipp Leubner info@attocube.com
Carl Zeiss SMT GmbH Carl-Zeiss-Str. 22 73447 Oberkochen Germany	Norman Niewrzella Tel : +49 7364205971 norman.niewrzella@zeiss.com
Cedrat Technologies 59 Chemin du Vieux Chêne 38246 Meylan Cedex France	Mathieu Thomachot Tel: +33 (0) 456580400 sandrine.hugi@cedrat-tec.com
JTEC Corporation 2-5-38 Saito-Yamabuki 567-0086 Ibaraki Osaka Japan	Masahiko Kanaoka Tel : +81 (0) 726552786 masahiko.kanaoka@j-tec.co.jp

Exhibitors attending the 2020 ESRF User Meeting, 3 - 5 February 2020, Grenoble, France

Leybold France Parc du Technopolis, Bâtiment Beta 3 Avenue du Canada 91940 Les Ulis France	François d'Armancourt Tel: +33 (0) 169824817 carine.larfouilloux@leybold.com
MKS Newport **Micro-Controle Spectra-Physics** 9 Rue du Bois Sauvage 91055 Evry France	Dalila Ait Amir Tel: +33 (0) 160916868 ariane.billard@newport.com
NEYCO 30 Avenue de la Paix 92170 Vanves France	Olivier Costa Tel: +33 (0) 141905050 olivier.costa@neyco.fr
Physik Instrumente France 380 Avenue Archimède 13100 Aix en Provence France	Stéphane Sage Tel: +33 (0) 442975230 s.sage@pi.ws
PINK GmbH Vakuumtechnik Gyula-Horn-Str. 20 97877 Wertheim Germany	Markus Debes Tel: +44 (0) 93428720 sales@pink-vak.de
Vacuum FAB Via Asilo 74 20010 Cornaredo (MI) Italy	Guido Giorgi Tel: +39 (0) 290363318 paola@beretta@vacuumfab.it
XGLab Via Conte Rosso, 23 20134 Milan Italy	Alessandro Tocchio Tel: +39 (0) 249660460 alessandro.tocchio@bruker.com

Tutorials

3rd February 2020

- Overall Programme
- Tutorials and Practicals Venue Map

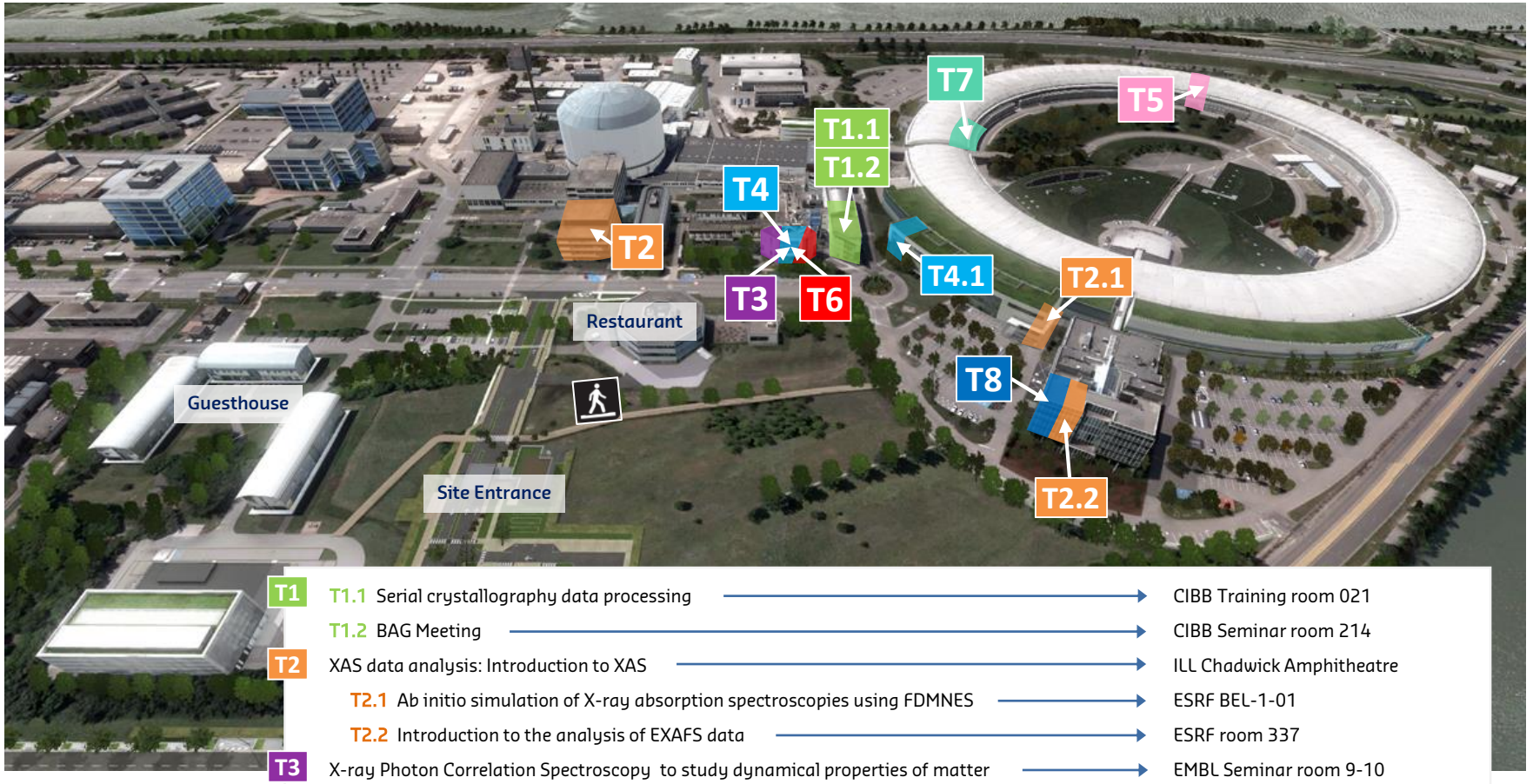
USER MEETING 2020 - TUTORIALS
Monday 3 February 2020

REGISTRATION in the ESRF Central Building from 8:15 to 9:00 and from 13:00 to 14:00

WELCOME COFFEE in the ESRF Central Building from 8:15 to 9:00

	TUTORIAL TITLE	ORGANISERS	TIME		MAX. PARTICIPANTS	MEETING ROOM	MEETING POINT	COFFEE BREAKS	EQUIPMENT REQUIRED
T1	SB: Serial crystallography data processing & BAG Meeting	Gianluca Santoni Max Harunobu Nanao	**9:00 - 12:00**	Merging of Serial crystallographic data by Hierarchical Cluster Analysis and Genetic Algorithms	**20**	CIBB Training room 021	8:30 under the marquee	CIBB Seminar room 214	
		Gordon Leonard David Flot Deborah Davison	**14:00 - 17:30**	BAG Meeting (by invitation) 2 sessions aroud coffee break	**45**	CIBB Seminar room 214	13:30 under the marquee	In the meeting room	
T2	XAS data analysis	Kirill Lomachenko	**9:00 - 12:00**	Introduction to XAS: The Whys, The Whats and The Hows	**no limit**	ILL Chadwick Amphitheatre	Directly in the amphitheatre	ILL Entrance Hall	
		Yves Joly	**14:00 - 17:00** *Parallel sessions*	1. Ab initio simulation of X-ray absorption spectroscopies using FDMNES	**15**	ESRF BEL-1-01	Directly in the meeting room	In the meeting room	Personal laptop (Mac, Windows or Linux) with a software to plot spectra (Origin, Keleidagraph, NOT Excel)
		Francesco d'Acapito		2. Introduction to the analysis of EXAFS data	**8**	ESRF room 337	Directly in the meeting room	In the meeting room	Personal laptop with the (free) software already installed before the session.
T3	X-ray Photon Correlation Spectroscopy to study dynamical properties of matter	Yuriy Chushkin Beatrice Ruta Federico Zontone	**9:00 - 10:00**	Coherence at 4th generation synchrotron sources *Common introduction with Tutorial T4.*	**50**	EMBL Seminar room 9-10	8:30 under the marquee	In the meeting room	
			10:15 - 12:15	Lecture, data analysis tools and practice New horizons at EBS	**25**				
T4	Introduction to coherent X-ray imaging techniques and data analysis	Vincent Favre Nicolin Steven Leake Manfred Burghammer Peter Cloetens	**9:00 - 10:00**	Coherence at 4th generation synchrotron sources *Common introduction with Tutorial T3.*	**50**	EMBL Seminar room 9-10	8:30 under the marquee	In the meeting room	
			10:15 - 12:30	Lecture: introduction to coherent imaging techniques	**18**	ESRF LOB-1-45	Directly in the meeting room	In the meeting room	Personal laptop for data analysis
			14:00 - 17:00	Hands-on data analysis using PyNX					
T5	Volume image analysis of tomographic data	Alexander Rack	**10:00 - 12:00 & 13:30 - 15:00**	Lecture session	**20**	ESRF MD-1-21	9:15 under the marquee	In the meeting room	Participants can bring their own data sets on USB key for preliminary tests and discussion
			15:30 - 18:30	Practicals, questions and demonstrations					
T6	SAXS analysis strategies & softwares	Michael Sztucki	**14:00 - 16:00**		**50**	EMBL Seminar room 9-10	13:30 under the marquee	In the meeting room	
T7	Laue microdiffraction	Jean-Sébastien Micha	**9:00 - 12:30**		**10**	MF-1-06	8:15 under the marquee	Corridor next to training room	
T8	PyFAI - Data reduction tools for scattering experiments: Application to SAXS and WAXS	J. Kieffer	**9:00 - 12:00**		**20**	ESRF room 500 - 501	Directly in the meeting room	In the meeting room	Personal laptop with working WIFI

USER MEETING 2020 - Tutorials & Practicals Venues



T1	T1.1 Serial crystallography data processing	CIBB Training room 021
	T1.2 BAG Meeting	CIBB Seminar room 214
T2	XAS data analysis: Introduction to XAS	ILL Chadwick Amphitheatre
	T2.1 Ab initio simulation of X-ray absorption spectroscopies using FDMNES	ESRF BEL-1-01
	T2.2 Introduction to the analysis of EXAFS data	ESRF room 337
T3	X-ray Photon Correlation Spectroscopy to study dynamical properties of matter	EMBL Seminar room 9-10
T4	Introduction to coherent X-ray imaging techniques and data analysis	EMBL Seminar room 9-10
	T4.1 Lecture & hands-on data analysis using PyNX	ESRF LOB-1-45
T5	Volume image analysis of tomographic data	ESRF MD-1-21
T6	SAXS analysis strategies & softwares	EMBL Seminar room 9-10
T7	Laue microdiffraction	ESRF MF-1-06
T8	PyFAI - Data reduction tools for scattering experiments	ESRF room 500 - 501

Plenary Session

4th February 2020

- Programme
- Abstracts of lectures

Plenary Session
Tuesday 4 February 2020
Venue: ESRF Auditorium

08.15 - 09.00 - REGISTRATION - ESRF Central Building (hall) and Welcome coffee

MORNING SESSION	
09.00 - 09.05	Opening and welcome by the User Organisation
09.05 - 09.50	Keynote Lecture 1: "Snapshots of actively transcribing influenza polymerase" **Stephen Cusack**, EMBL, Grenoble, France
09.50 - 10.35	Keynote Lecture 2: "Nanostructure analysis in real space: PDF studies of nanoparticle chemistry" **Kirsten M. Ø. Jensen**, University of Copenhagen, Denmark
10.35 - 11.00	*Coffee break*
11.00 - 12.30	Facility report & EBS news: • Introduction - F. Sette (5') • EBS storage ring commissioning - P. Raimondi (15' + 5' questions) • Beamline status and restart plan - H. Reichert (15' + 5' questions) • Data management challenges at the ESRF - J. Susini or R. Dimper (15' + 5' questions) • Control system and data policy implementation - A. Goetz (15' + 5' questions)
12:30 – 13:30	*Lunch at the EPN campus restaurant*
AFTERNOON SESSION	
14.00 - 14.45	Keynote Lecture 3: "LEAPS Sets Sails" **Caterina Biscari**, ALBA Synchrotron, Barcelona, Spain, and LEAPS
14.45 - 15.30	Keynote Lecture 4: "Coherent X-rays: high-resolution imaging for all" **Vincent Favre Nicolin**, ESRF, Grenoble, France
15.30 - 16.00	Poster Clips (1)
16.00 - 16.20	*Coffee break*
16.20 - 17.10	Young Scientist Award
17.10 - 17.40	Tribute to Rosalind Franklin **Elspeth Garman**, University of Oxford, UK
17.40 - 18.00	Poster Clips (2)
18.00 - 19.30	Poster session and cocktail
19.30 - 22.00	Dinner at the EPN campus restaurant Young Scientist and Best Poster Awards

Snapshots of actively transcribing influenza polymerase

Stephen Cusack

European Molecular Biology Laboratory, Grenoble, France, cusack@embl.fr

The flu is caused by the highly infectious, rapidly evolving and potentially dangerous influenza virus. The genetic material of the influenza virus is single-stranded RNA. During an influenza infection, the viral RNA-dependent RNA polymerase uses the genomic RNA as a template firstly to synthesise viral messenger RNA, which is then translated into viral protein by the cellular protein synthesis machinery, and secondly, in a distinct process, to generate genome copies. The genome copies together with the newly synthesized viral proteins are then packaged into progeny virions that can go on to infect other cells and organisms. Our goal is to understand at atomic resolution the unique mechanisms whereby influenza polymerase performs transcription and replication of the viral genome. This is not only of fundamental interest but will also help understand avian to human interspecies transmission of the virus and promote development of new anti-influenza drugs targeting the polymerase.

To achieve this goal we have used a combination of the complementary methods of X-ray crystallography and single particle cryo-electron microscopy (cryoEM), most often performed at the ESRF, to determine structures of the polymerase in various functional states. In particular, recent advances in cryoEM have permitted a series of snapshots of transcribing polymerase to be obtained that, for this system, are superior in resolution to that previously obtained by X-ray crystallography. These structures constitute a molecular movie of the polymerase machine in action. In addition, structures will be presented showing the mode of action of the newly approved anti-influenza drug Xoflusa (baloxavir marboxil) that directly inhibits transcription by the polymerase and possible ways how the virus can become resistant to the drug.

References

[1] - Structure of influenza A polymerase bound to the viral RNA promoter. Pflug, A. et al., Nature 2014.

[2] - Structural insight into cap-snatching and RNA synthesis by influenza polymerase. Reich, S. et al. Nature 2014.

[3] - Influenza polymerase can adopt an alternative configuration involving a radical repacking of PB2 domains. Thierry, E. et al. Mol Cell. 2016.

[4] - Structural analysis of specific metal chelating inhibitor binding to the endonuclease domain of influenza pH1N1 (2009) polymerase. Kowalinski, E. et al. PLoS Pathog. 2012.

[5] - Structural basis for an essential interaction between influenza polymerase and Pol II C-terminal domain. Lukarska, M. et al. Nature 2017.

[6] - Capped RNA primer binding to influenza polymerase and implications for the mechanism of cap-binding inhibitors. Pflug A, et al. Nucleic Acids Res. 2017.

[7] - Characterization of influenza virus variants induced by treatment with the endonuclease inhibitor baloxavir marboxil. Omoto S, et al.. Sci Rep. 2018.

[8] - Structural snapshots of actively transcribing influenza polymerase. Kouba, T. et al. NSMB. 2019.

Nanostructure analysis in real space: PDF studies of nanoparticle chemistry

Kirsten M. Ø. Jensen

Department of Chemistry and Nanoscience Center, University of Copenhagen, kirsten@chem.ku.dk

Nanomaterials have come to play a huge role in modern materials chemistry: By nanosizing the functional materials used for a range of applications, e.g. batteries and catalysis, many properties can be improved. This development has challenged our understanding of structure/property relations, as the conventional techniques for material characterization break down for structures on the nanoscale. However, total scattering combined with Pair Distribution Function analysis allows us to look further into nanostructure and establish this relation for many advanced functional materials,[1] opening a whole new level of insight for material chemists.

Here, I will present recent work illustrating how we use total scattering to characterize nanomaterial structure and show how Pair Distribution Function analysis can be used to elucidate the atomic arrangements in even the tiniest of nanoparticles and nanoclusters, with special focus on metal and metal oxide nanoparticles.[2,3] Using PDF, we observe that new structural motifs, unstable in the bulk form, become dominant in nanoscale materials. In gold clusters, for example, we use PDF analysis to determine the atomic arrangement in 2 nm particles, where *fcc* structures are no longer stable.[2] In oxide materials, we see that defects know from bulk materials completely dominate the atomic structure on the nanoscale, and changes the atomic arrangement significantly, highly affecting their properties.[3] We also apply total scattering techniques to gain a new understanding of the reactions and processes taking place during crystallization of materials. In situ PDF methods allow following structural changes throughout a synthesis, all the way from an ionic cluster in solution, over amorphous intermediates and to the final crystalline material, making it possible to gain new insight into nucleation mechanisms.[5] I will furthermore show how total scattering combined with computed tomography can open for a range of new *in situ* studies, as nanostructure can now be positionally resolved.

References

[1] - S. J. L. Billinge, I. Levin, *Science* **316**, 561-565, (2007).
[2] - K. M. Ø. Jensen, P. Juhas, M. A. Tofanelli, C, K, Heinecke, G. Vaughan, C. Ackerson, S. J. L. Billinge, *Nature Communications* **7**, 11859, 2016.
[3] - T. L. Christiansen, E. D. Bøjesen, M. Juelsholt, J. Etheridge, K. M. Ø. Jensen, *ACS Nano*, 13, 8725-8735, 2019
[4] - K. M. Ø. Jensen, C. Tyrsted, M. Bremholm, B. B. Iversen, *ChemSusChem* **7**, 1594-1611, 2014.
[5] - M. Juelsholt, T.L. Christiansen, K. Jensen, *J. Phys. Chem. C*, 123, 8, 5110-5119, 2019

LEAPS Sets Sails

Caterina Biscari

ALBA Synchrotron, LEAPS, cbiscari@cells.es

The European Synchrotrons and Free Electron Lasers have a long history of fruitful collaborations in a healthy competitive environment. Now they are joining forces to master the challenges of the next decades by adding their capabilities in smart specializations, and developing together the European strategy for photon science.

LEAPS, the League of European Accelerator-based Photon Sources, includes sixteen institutions as founding members, hosting national and international facilities, and representing a total community of 35000 researchers. SESAME has joined as first Associated Member.

Scientific and technical developments for next generation sources, innovation programs, technological transfer for industrial capacitation, better services to users, opening to the world, training and education, are the main stream of the collaboration.

LEAPS is developed in cooperation with the European Commission, calling to a new paradigm of co-funding participation of national funding agencies to such kind of programs. A first Pilot Project, LEAPS-INNOV, will be presented in the forthcoming H2020 call, while organizing the future programs to be developed in Horizon Europe.

Coherent X-rays: high-resolution imaging for all

Vincent Favre-Nicolin

ESRF, Grenoble, France, vincent.favre-nicolin@esrf.fr

The use of coherent X-rays for imaging has been steadily increasing for the past 25 years, from phase contrast imaging to coherent diffraction and ptychography experiments, resulting in two and three-dimensional material inspection with a spatial resolution down to about 10 nanometers. This progress will soon be enhanced by the Extremely Brilliant Source upgrade, leading to an improved spatial and temporal resolution. However for a long time coherent diffraction techniques were mostly used by a limited community as much focused on the methodology than on the materials.

I will present the various techniques which can be used for standard small-angle imaging, yielding the sample's electronic density, with applications from brain imaging to fuel cells, as well as those in the Bragg geometry, giving access to strain information, e.g. for semi-conductor nano-structures or catalysts. Most importantly I will show how algorithms and data processing have improved during the last few years, providing a more robust data analysis which can be performed with limited supervision or hand-tuning. Additionally, efforts on a more efficient use of modern computing resources allows much faster two or three-dimensional reconstructions, both during and after the experimental time. These advances, along with the improved photon flux, should pave the way for the application of high-resolution coherent imaging techniques with a larger community.

User-Dedicated Microsymposium UDM 1

5th February 2020

In situ and operando X-ray absorption spectroscopy for the study of catalysts and functional materials

UOC Organiser	Michela Brunelli
ESRF Organisers	Kirill Lomachenko
	Dipanjan Banerjee
	Wouter van Beek
	Pieter Glatzel

- Programme
- Abstracts of Keynote & User Talks

UDM1. In situ and operando X-ray absorption spectroscopy for the study of catalysts and functional materials

Wednesday, 5th February 2020 - Microsymposium UDM1 Venue: ILL Chadwick Amphitheater	
8:30	Registration
9:00	Introduction by the organizers

Morning session

	Session I – Chair: Kirill Lomachenko	
9:05 – 9:50	**Keynote talk 1:** About active sites in heterogeneous catalysts	**Jeroen van Bokhoven** *ETH Zürich, Switzerland*
9:50 – 10:10	Monitoring structural changes in MoxSy phase encaged within the confinement of zeolites via HERFD-XAS and VtC-XES measured under operando sulfidation/hydrogenation reaction conditions	**Rachit Khare** *Technical University of Munich, Germany*
10:10 – 10:30	Operando XAS on atomically precise Pt-CO clusters for oxygen reduction reaction	**Martina Fracchia** *University of Pavia, Italia*
10:30 – 11:00	*Coffee break*	
	Session II – Chair : Dipanjan Banerjee	
11:0 – 11:45	**Keynote talk 2:** Understanding catalysis for realistic supported catalysts: Methane oxidation and CO_2 methanation	**Per-Anders Carlsson** *Chalmers University of Technology, Sweden*
11:45 – 12:05	Multivariate statistical analysis of in situ and operando X-ray Absorption Spectroscopy data	**Samuel Regli** *Norwegian University of Science and Technology, Norway*
12:05 – 12:25	Fe-based bimetallic catalysts: evidencing the interplay between the two metals using in situ/operando XAS and chemometrics	**Eric Marceau** *Université Lille, France*
12:25 – 14:00	*Lunch at the EPN campus restaurant*	

UDM1. In situ and operando X-ray absorption spectroscopy for the study of catalysts and functional materials

Afternoon session

	Session III – Chair: Pieter Glatzel	
14:00 – 14:45	**Keynote talk 3:** From nanoparticles synthesis in solution to functional devices – a perspective based on in situ synchrotron studie	**Dorota Koziej** *University of Hamburg, Germany*
14:45 – 15:05	Operando X-ray absorption spectroscopy studies of Pd-based catalysts	**Aram Bugaev** *Southern Federal University, Russia*
15:05 – 15:25	Identification of mobilized Cu-oxygen pairs and of their role in the low temperature NH3-Selective Catalytic Reduction	**Tommaso Selleri** *Politecnico di Milano, Italy*
15:25 – 15:45	CO oxidation over nanocomposite CuFeAl catalysts: In situ XAS study	**Andrey Saraev** *Boreskov Institute of Catalysis, Russia*
15:45 – 16:10	*Coffee break*	
	Session IV - Chair: Michela Brunelli	
16:10 – 16:30	In situ Surface Resonant X-Ray Diffraction to probe the electronic structure at electrochemical interfaces	**Yvonne Soldo** *Institut Néel, CNRS & Université Grenoble Alpes, France*
16:30 – 16:50	Reaction cells for XAS and HERFD-XAS operando characterization	**Antonio Aguilar** *Institut Néel, Université Grenoble Alpes, France*
16:50 – 17:10	Sample environment laboratory at the ESRF	**Yves Watier** *ESRF, France*
17:10 – 17:30	BL updates from ESRF and CRG staff	
17:30	**End of the meeting**	

About active sites in heterogeneous catalysts

Jeroen A van Bokhoven[1]

[1]ETH Zurich and Paul Scherrer Institute

Heterogeneous catalysts are essential and widely applied in the production of fuels and chemicals and in pollution abatement. Despite their widespread use, the molecular level description of the catalytic process remains poor. One of the reasons is the difficulty to identify the structure of the ensemble of atoms that constitute the active site, being the minority of species and / or being short-lived. In situ and especially operando characterization, determining structure and performance simultaneously, has become the standard. However, even under operando conditions it remains very difficult if not impossible to quantify the number of active sites and their structure.
In beneficial cases, transient and time-resolved structural measurements enable the identification of active sites. In other cases, quantitative relations between a certain species and catalytic activity has been established. Because of the essential role of characterization in the time-domain, development and application of time-resolved x-ray and x-ray photoemission spectroscopies will be described. From such measurements, the need for relevant pressures during measurement, thus in situ, becomes clear.

References

René Kopelent, Jeroen A. van Bokhoven, Jakub Szlachetko, Jacinta Edebeli, Cristina Paun, Maarten Nachtegaal and Olga V. Safonova, Angewandte Chemie. Int. Ed. 54 (2015) 8728-8731
Simon A. Kondrat and Jeroen A. van Bokhoven, Topics in Catalysis, DOI: 10.1007/s11244-018-1057-4
Luca Artiglia, Fabrizio Orlando, Kanak Roy, René Kopelent, Olga Safonova, Maarten Nachtegaal, Thomas Huthwelker and Jeroen A. van Bokhoven, The Journal of Physical Chemistry Letters 8 (2017) 102-108.

Monitoring structural changes in Mo_xS_y phase encaged within the confinement of zeolites via HERFD-XAS and VtC-XES measured under *operando* sulfidation/hydrogenation reaction conditions

R. Khare[1], R. Weindl[1], H. Shi[1], A. Jentys[1], J.A. Lercher[1]

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Transition metal sulphides encaged in the (sub)nanometric confines of ordered porous materials like zeolites are a class of promising catalysts for processing heavy petroleum feedstock. However, rather limited knowledge has been accumulated over the past decades regarding the dispersion, local structure, and electronic state, of the Mo_xS_y phase encaged within these zeolites, in its as-prepared state or under *operando* sulfidation/hydrogenation reaction conditions. We monitored structural changes in the Mo_xS_y phase encapsulated within zeolite-Y (Si/Al ~2.4) under sulfidation and hydrogenation (of toluene or ethylene) reaction conditions using *operando* HERFD-XANES (high energy resolution fluorescence detected – x-ray absorption near edge structure), VtC-XES (valence-to-core x-ray emission spectroscopy), and EXAFS (extended x-ray absorption fine structure) measurements.

HERFD-XANES, VtC-XES, and EXAFS measurements were performed at the ID26 beamline of the European Synchrotron Radiation Facility (ESRF) at Mo K-edge (20 keV) under *operando* conditions using a quartz capillary reaction cell. For HERFD-XANES measurements, the energy of the monochromator was scanned between 19.95 keV and 20.2 keV while for VtC-XES, monochromator energy was fixed at 20.1 keV. Data analysis was performed using PyMCA v5.4.1 and Athena/Artemis software packages. All spectra were normalized and flattened for further analysis. *Operando* measurements were performed under sulfidation reaction conditions or under hydrogenations conditions. Time dependent density function theory (TDDFT) calculations were performed using the Orca package v4.1. Relativistic affects were taken into account using ZORA. All electron Karlsruhe basis sets were used throughout. TDDFT calculation were performed using Tamm-Dancoff approximations and up to 100 roots were calculated allowing for transitions only from Mo 1s orbitals. We also performed TDDFT calculations on six different reference materials: Mo^0-foil, $Mo^{IV}S_2$, $Mo^{VI}O_3$, $K_2Mo^{VI}O_4$, $(NH_3)_6Mo^{VI}_7O_{24}$, and $Mo^0(CO)_6$, using eight different functionals: BP86, BLYP, PBE, TPSS, PBE0, B3LYP, BHLYP, and TPSSh. The performance of each functional was assessed by comparison with HERFD-XANES and VtC-XES experimental data obtained at ID26.

We observed that the predominant Mo-species under sulfidation reaction conditions is Mo_2S_6 with Mo-Mo coordination of ~1 and Mo-S coordination of ~4. Under hydrogenation conditions at 673 K, however, the dominant Mo-species observed was Mo_4S_8 with a Mo-Mo coordination of ~3 and Mo-S coordination of ~3. We also observed that this process is reversible with Mo_2S_6 species forming back on re-sulfidation of the reduced catalyst. HERFD-XANES and EXAFS measurements at ID26, together with VtC-XES, and complemented with TDDFT calculations, enhanced our understanding of the structure of Mo_xS_y phase in zeolite-Y under *operando* reaction conditions. An enhanced understanding of the structure as well as the electronic state of Mo_xS_y will further our knowledge of the mechanism of ethene or toluene hydrogenation on these catalysts.

Operando XAS on atomically precise Pt-CO clusters for oxygen reduction reaction

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The progressive lack of fossil fuels together with the growing awareness of the current environmental problems has led to an extensive research on renewable and sustainable energy sources. Hydrogen, in particular, has been proposed as the future environmentally sustainable energy vector, since it can be produced locally by renewable sources (*e.g.* by photocatalytic water splitting) and can be then employed in fuel cells. Metal-air batteries have also been considered as possible alternatives for energy storage/production, due to their low environmental impact and versatility. In the above-mentioned technologies, the Oxygen Reduction Reaction (ORR) covers an important role, being the bottleneck of the entire process. It is well known that Pt supported by carbon is among the best catalysts for the ORR reaction; however, Pt and other noble metals are extremely expensive and show low abundancy.

A possible alternative consists in designing atomically-precise Pt-based materials and depositing them on inert or synergistic matrixes [1], thus reducing the total loading of the noble material. In the work here presented, platinum carbonyl clusters $[Pt_{24}(CO)_{30}]^{2-}$, $[Pt_{12}(CO)_{24}]^{2-}$ and $[Pt_{15}(CO)_{30}]^{2-}$ were considered as valid alternatives to platinum nanoparticles and were investigated through *in situ* and *operando* X-ray Absorption Spectroscopy (XAS). In the recent years, we have deeply investigated several (photo)electrocatalytic materials [2,3], searching for new strategies to catch the structural and electronic modifications occurring in a catalyst during a given electrochemical process (*e.g.* Fixed Energy X-ray Absorption Voltammetry or $\Delta\mu$ methods [4]). In this work, the platinum carbonyl clusters were employed as cathode materials in a properly-designed spectroelectrochemical cell [5] and investigated at different potentials by combining spectroscopic and electrochemical measurements. A detailed analysis of the EXAFS region clearly showed that even after polarization to cathodic potentials, the clusters retain their initial local structure. These findings, coupled to transmission electron microscopy, proved that the particles do not show aggregation maintaining their high catalytic activity. Moreover, the activity of the clusters under hydrogen evolution was investigated, following the correspondent modifications of the white line and of the EXAFS signal; in particular, the $\Delta\mu$ method showed that hydrogen is preferentially adsorbed at n-fold sites.

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Understanding Catalysis for Realistic Supported Catalysts: Methane Oxidation and CO_2 Methanation

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The development of industrial heterogeneous catalytic processes often relies on trial-and-error approaches in which rich chemical experience, common sense and intuition form the starting point. Combined with statistical methods such as catalysis informatics, these will likely continue to be used in the foreseeable future. For an increasing number of important chemical reactions, however, the trial-and-error approach is insufficient to bring about the next generation catalysts. Even when combined with statistical methods, new catalyst formulations are challenging to present as the dynamic, surface-sensitive and multiscale nature of heterogeneous catalysis need to be incorporated. Excelled scientific understanding and data retrieval as to enter the paradigm of knowledge-based catalyst design, is thus not only of pure academic interest but is increasingly desired by catalyst manufacturers. The progress in experimental methods during the last two decades, not least at large-scale facilities, with increased focus on material dynamics and surface characterisation under relevant conditions promotes this paradigm shift to happen.

This contribution will present foremost experimental work on methane oxidation and CO_2 methanation over supported noble metal catalysts. Much but not all of the work has been joint academia-industry efforts always with the overall goal of building understanding. A bottom-up experimental approach has been employed to understand individual catalyst components and the interplay between them using transient *operando* XANES [1], XAFS [2], HE-XRD [3] at ESRF, PETRA III, MAX IV and *operando* IR spectroscopy [4] at Chalmers. For methane oxidation, the active state of platinum, palladium and an alloy between them as well as the mechanistic consequences of palladium dispersion and water will be discussed. For CO_2 methanation, the mechanistic pathway over rhodium-ceria will be discussed.

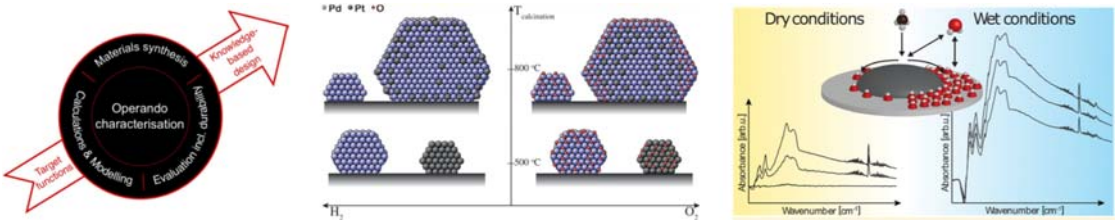

Figure 1: (left) Process for knowledge-based design of catalysts, (middle) surface composition of Pd-Pt alloyed particles in reducing and oxidizing environments, the latter resembles that during dry methane oxidation and (right) pathways for hydroxyl formation on Pd/Al_2O_3.

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Multivariate statistical analysis of *in situ* and *operando* X-ray Absorption Spectroscopy data

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X-ray Absorption Spectroscopy (XAS) is an element specific analysis which focuses on the local order and electronic structure of the absorbing atom. The high penetrating power of X-rays allows this method to be carried out *in situ* and *operando*. Provided that measurements are performed with adequate time-resolution, the underlying reaction mechanisms and the nature of the chemical intermediates involved can be identified.

The large amount of data collected during a typical *operando* X-ray absorption fine structure experiment and the interest to thoroughly investigate and comprehend the processes occurring, need an advanced data analysis approach. Established methods, such as the linear combination of known standard materials, do often not accurately represent the measured data at relevant temperatures, pressure and reactants present. This mismatch is explained by a different chemical nature and often stable pure states of a compound do not exist to be measured *ex situ*. In this context, multivariate statistical analysis has gained attention, which allows the identification of the number and their abundance of the chemical species involved, with limited *a priori* information on the studied system. [1]

After a brief historical introduction and the basic insights on the technique of multivariate statistical analysis, I will provide a selection of XAFS examples and case studies, to discuss and demonstrate approaches to determine the number of components and their abundance in the dataset by matrix factorization. Given this information, initial guesses by blind-source separation through Evolving Factor Analysis [2], independent component analysis [3] or purest variables [4] will be discussed. Rank deficiency and strategies to try to resolve beyond will be presented. Furthermore, a closer look at the regression and the applied constraints will be elaborated, for example for samples where the absorption step is not constant. A comparison to compare information obtained by commonly applied methods such as white-line intensity, edge-position, pre-edge features and linear combination will be showcased.

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Fe-based bimetallic catalysts: evidencing the interplay between the two metals using *in situ/operando* XAS and chemometrics

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A strong asset of X-ray absorption spectroscopy (XAS) is to bring insight into the interplay establishing between the two metals on bimetallic catalysts. Here, XAS was applied to Fe-containing bimetallic systems, which are often difficult to characterize because of the small number of spectroscopic techniques to which Fe (and sometimes the second metal) responds. Fe-Ni/SiO_2 catalysts are known for their selectivity in the hydrogenation of unsaturated organic molecules. Time-resolved XAS was implemented in the transmission mode on beamline ROCK, SOLEIL, in order to understand the formation process and structure of Fe-Ni nanoparticles [1]. Quick-EXAFS spectra were recorded *in situ* under H_2 flow, simultaneously at the Fe and Ni K edges. The proportions of the Fe and Ni species were determined by the multivariate MCR-ALS method. The reduction of Fe ions was found to take place over three stages: reduction of Fe^{3+} to Fe^{2+}, of Fe^{2+} to Fe^0, progressive migration of Fe^0 into Fe-Ni fcc particles. The formation of Fe^0 is exactly triggered by the reduction of Ni above 350°C (Fig. 1). The late reduction of Fe leaves an excess of Fe in the outer shells of the particles, from which the high selectivity of Fe-Ni in hydrogenation reactions derives.

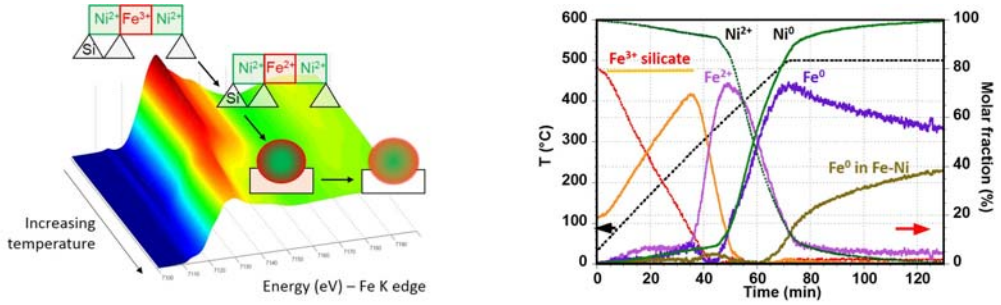

Figure 1: Formation process of Fe-Ni nanoparticles followed by XAS and analyzed by MCR-ALS procedure

In the case of Fe-Bi bimetallic catalysts supported on carbon nanotubes (CNT), that have been found to be very stable in the Fischer-Tropsch synthesis [2], XAS was performed in *operando* conditions (capillary under pressure of CO and/or H_2) on beamline DUBBLE, ESRF. While Fe transforms into carbide, XAS at the Bi L_3 edge shows that Bi reduces to the elemental state under CO or H_2. Interestingly, Bi re-oxidizes under CO when the temperature decreases, which does not occur for a mechanical mixture of Fe/CNT and Bi/CNT catalysts. The phenomenon is reversible upon increase of the temperature. This proves the interaction between Fe and Bi on Fe-Bi/CNT, and the role of Bi in scavenging oxygen from CO. XAS thus provides invaluable evidence on an element otherwise very difficult to characterize.

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From nanoparticles synthesis in solution to functional devices – a perspective based on in situ synchrotron studies

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Over the past years we have developed various approaches to fabricate materials with sophisticated chemical and structural complexity. We have focused on synthesis in non-aqueous solution since this approach is not limited to one particular class of materials. Thus, it gives us flexibility to tailor the composition and properties of materials in respect to the application.

In this talk, I will present how X-ray synchrotron methods, far from merely providing new tools, are extending the ways we study, understand and design such complex structures. Particularly, combination of spectroscopic and scattering methods and rapid data acquisition help to uncover the complex chemical world behind the synthesis of functional materials. It gives complementary information about chemical reaction in solution and nucleation, growth and crystal phase transition of nanoparticles. [1-2]

Moreover, I will discuss how the possibility to select with high-energy resolution the incident and emission X-ray energies offers unprecedented site selectivity and give access to determine structure – function relationship of electrochemical materials. [3,4,5]

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Operando X-ray absorption spectroscopy studies of Pd-based catalysts

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This contribution summarizes recent results (both published and unpublished) on *in situ*, *operando* and time-resolved investigation of Pd-based catalysts by means of X-ray absorption spectroscopy and complementary techniques, obtained during a series of experiments at BM23, ID24, BM26 and BM31 beamlines of ESRF.

A combination of simultaneous EXAFS and XRD allowed us to highlight the difference of the local atomic structure in the bulk and surface structure of monometallic Pd nanoparticles upon formation of hydride phase [1,2]. Further utilization of XANES region, allowed us to discriminate bulk palladium carbides and surface adsorbed hydrocarbons [2,3].

The above knowledge was used to follow the evolution of the working catalyst under *operando* conditions [4,5]. We showed that during hydrogenation of ethylene, palladium carbide phase is formed progressively and irreversibly even in the instant excess of hydrogen, co-existing with palladium hydride and metallic palladium (Figure 1).

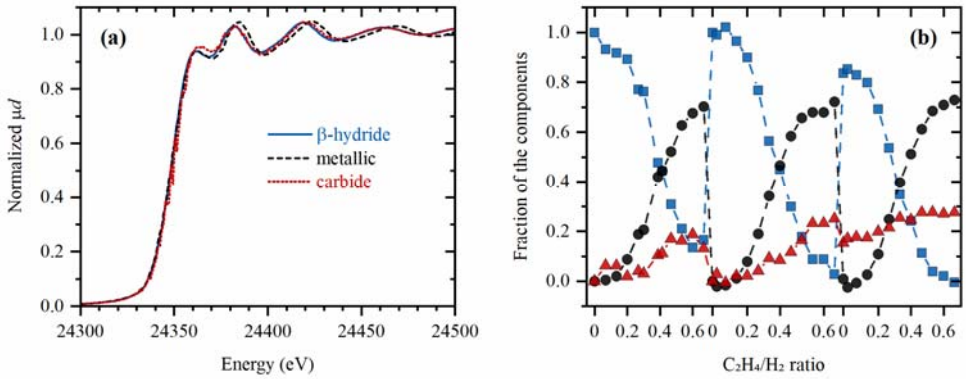

Figure 1: XANES spectra (a) of pure species extracted from the experimental dataset and their respective concentrations as a function of C_2H_4/H_2 ratio and time [4].

Since XANES was shown to play an ultimate role in detecting Pd-C and Pd-H bonds, several important approaches to its analysis were established [6-8]. The first one implements the PCA approach to determine the number of independent components in the datasets and convert them into physically meaningful spectra by means of target matrix transformation [6]. The second involves machine learning algorithms to extract structural information from XANES exploiting the library of theoretical spectra [7-8]. Both approaches were successfully applied to analyse big experimental datasets of *operando* and time-resolved data.

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Identification of mobilized Cu-oxygen pairs and of their role in the low temperature NH_3-Selective Catalytic Reduction

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Recent literature studies [1] indicate that the formation and reactivity of $[Cu_2(NH_3)_4O_2]^{2+}$ complexes, whose structure is currently unknown, are crucial steps in the redox mechanism of the NH_3-SCR (Selective Catalytic Reduction) on Cu chabazite (Cu-CHA) zeolites, i.e the state-of-the-art technology for NOx abatement from lean burn engines [2]. To fill this gap, we herein apply a combination of techniques such as *operando* X ray absorption (XAS), performed at the BM23 beamline at ESRF, and Diffuse Reflectance (DR) UV-Vis spectroscopies, DFT optimized structures for EXAFS fitting and Wavelet Transform Analysis (WTA) to a Cu-CHA sample with Si/Al=15 and Cu/Al=0.5 (Cu density ~ 0.45 Cu/1000 $Å^3$).

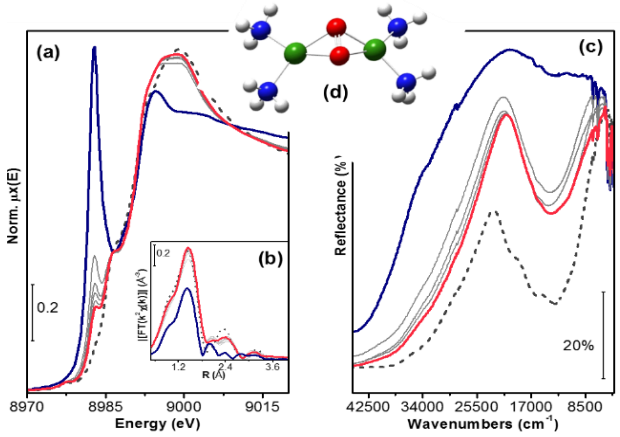

Figure 1: *Operando* (a) XANES, (b) EXAFS and (c) UV-Vis DR spectra of Cu-CHA exposed to O_2/He at 200 °C (light grey to red) after reduction in NO/NH_3/He at 50 °C (blue). Dark grey: O_2-activated. (d) $[Cu_2(NH_3)_4O_2]^{2+}$ peroxo side-on complex, Cu, green; H, white; O, red; N, blue

Figure 1 reports the XAS and DR UV-Vis spectra observed after reaction of reduced $Cu^I(NH_3)_2$ sites (blue) with O_2 at 200 °C (light grey to red). The Cu^I/Cu^{II} transformation indicates the formation of $[Cu_2(NH_3)_4O_2]^{2+}$ complexes. This transformation is fast and ~ 85% of Cu is oxidized, as predicted [2]. The final Cu^{II} state is clearly different from that obtained by activation in O_2 starting from a hydrated sample (red vs dashed dark grey). The EXAFS data match a DFT-computed peroxo-side on structure (Figure 1d). The presence of Cu-Cu dimers was shown for the first time by WTA, allowing the discrimination of second shell scattering contributions around the absorber. The reactivity of $[Cu_2(NH_3)_4O_2]^{2+}$ with NO and NH_3 has also been investigated, giving new insights in the reaction mechanism for NH_3-SCR.

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CO oxidation over nanocomposite CuFeAl catalysts: In situ XAS study

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Gasification of solid fuels and following catalytic combustion of the resultant gas is allows utilizing low-grade fuels such as lignite, peat, and firewood as well as various industrial wastes. Besides, catalytic combustion produces a significantly lower amount of harmful emissions then "traditional combustion" of fuels. The application of this technology is limited by the absence of the effective and low cost catalysts. One of the promising catalysts for this process is CuFeAl nanocomposite which demonstrates high activity and stability in the oxidation of CO containing mainly in the resultant gas of solid fuel gasification [1,2]. To develop the catalysts with highest possible activity and stability, the origin the active species and the mechanism of the oxidation of CO over CuFeAl nanocomposite should be investigated. Here we present the results of in situ studies of the catalyst state in the oxidation of CO. Operando XAS experiments were performed at P65 stations at PETRA III (Hamburg, Germany).

We found that the fresh CuFeAl-composite catalysts consist of $Cu_xAl_yFe_{3-x-y}O_4$ spinel, CuO, and α-Fe_2O_3. According to the linear combination fitting of Cu K-edge XANES spectrum the copper presents in two phases: CuO (25%) and $Cu_xAl_yFe_{3-x-y}O_4$ spinel (75%). To obtain information about the catalytic process Cu5Fe78Al17 was studied by XANES and EXAFS directly during heating in the CO flow and $CO:O_2$ = 2:1 and 1:1 mixtures. In a CO flow, the reduction of copper from Cu^{2+} to Cu^{1+} and Cu^0 started at temperature about 200°C; at 600°C copper is mainly in the metallic state. In $CO:O_2$ = 2:1 mixture, the reduction of copper from Cu^{2+} to Cu^{1+} started at temperature about 300°C. And it should be stressed that copper in the metallic state was also observed, moreover, at 600°C approximately 65% of copper is in the Cu^0 state (Cu^{1+} is 25% and Cu^{2+} is 10%). The increase of oxygen pressure at 600°C leads to full re-oxidation of copper to Cu^{2+} state and the catalyst returns to the initial state ($Cu_xAl_yFe_{3-x-y}O_4$ spinel and CuO). We believe that the lattice oxygen in the $Cu_xAl_yFe_{3-x-y}O_4$ spinel has more lability then one in the copper oxide and in the iron oxides that leads to increase the activity of catalysts with promotion of Fe-Al oxide by copper.

Acknowledgement. This work was supported by the Russian Science Foundation, grant 17-73-20157.

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In situ Surface Resonant X-Ray Diffraction to probe the electronic structure at electrochemical interfaces

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Electro-catalysts allow speeding up electrochemical reactions typically occurring at the electrochemical interface, singular domain of some Ångstrom of thickness where the charge exchange between the conducting electrode and the electrolyte occurs. Such materials have important applications in several domains, like energy storage, chemical synthesis, bio-sensors…

In this context, the description of the electrochemical interface and the comprehension of the related electro-catalytic mechanisms are of primary importance. It is first and foremost a question of material of electrode and more specifically a problem of structural and electronic properties of its surface. However, there is currently no experimental method to specifically probe the electronic structure of the surface directly under electrochemical operation and thus experimental evidence for the theoretical predictions regarding charge distribution is still lacking.

In this frame, we aim at developing the surface resonant x-ray diffraction (SRXRD) into a standard technique to probe charge distribution and electronic densities for electrochemical systems in situ conditions. Indeed, SRXRD couples surface X-Ray diffraction, widely used to solve the atomic structure at the surface of single crystals, to X-Ray absorption near edge spectroscopy (XANES), highly sensitive to the oxidation states. The experimental approach is supported by the theoretical ab initio calculations of the FDMNES home-made software, recently implemented with the simulation of surface diffraction experiments [1] and further developed for electrochemical interfaces description.

We present here an exploratory in situ SRXRD study (F-CRG-D2AM) coupled to FDMNES calculations of Pt(111) in 0.1M H2S04, a particularly interesting electrochemical model interface for the investigation of competitive anions adsorption processes hindering the kinetics of the electrocatalytic processes. Although this system has been extensively studied, the description of adsorbed sulfates in acidic media as a function of the applied potential and of the related charge exchange is still controversial.

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Reaction cells for XAS and HERFD-XAS *operando* characterization

A. Aguilar Tapia[1*], S. Ould-Chikh[2], E. Lahera[3], A. Prat[1], W. Delnet[3], O. Proux[3], I. Kieffer[3] and J-L Hazemann[1]

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Heterogeneous catalysts commonly exhibit a variety of different surface sites that are difficult to identify. Identification of the active sites is critical for the design and development of improved catalytic materials. Ideally, characterization of a catalyst involves the measurement of its corresponding properties during the catalytic reaction, *i.e.*, *operando* conditions. However, performing analytic measurements of the reaction process under realistic conditions is highly challenging. Currently, X-ray absorption spectroscopy (XAS) is one of the most widely used techniques for analysis of catalysts under reaction conditions due to the penetration depth of the high-energy X-rays, enabling adequate analysis of the electronic and structural properties of heterogeneous catalysts. Our setup is derived from the previous high-pressure/high-temperature cell available on the FAME beamline.1 Therefore, it is possible to operate at high temperatures (up to 1000 °C). The design offers the capability of using fluorescence and transmission detection modes. The reaction cell includes a plug-flow reactor made from glassy carbon which allows almost all of the X-rays to be transmitted to the sample2 (Figure 1 left). The cell is available in BM30 beamline. Further modifications of the reaction cell resulted in a new cell with a 70° aperture, which allows irradiation of the 14-crystal analyzer spectrometer (CAS) available in BM16. This new HT cell allow the collection of HERFD-XANES and XES spectra in *operando* conditions (Figure 1 right). Both reaction cells count with a completely automated gas distribution system, which is used to deliver a mixture of gases through the reaction cell, and the venting. The gas composition from the reactor is monitored on-line by an EcoCat-P portable mass spectrometer system operated remotely. The system is equipped with two identical capillary inlets for the on-line analysis of the reactor outlet and a bypass line. The system also allows quantitative analysis and has the capability to monitor up to 64 species in real time. It offers detection levels down to ppb levels.

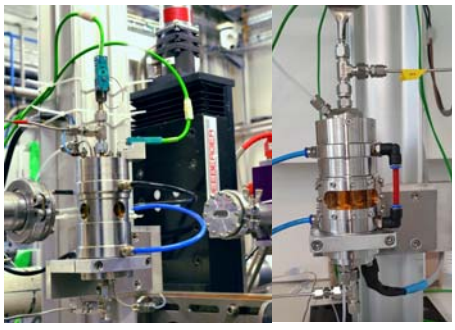

Figure 1: HT operando reaction cells available in BM30 (left) and BM16 (right).

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User-Dedicated Microsymposium UDM 2

5th February 2020

Nanomaterials life cycle: from nanoengineering to public health

UOC Organiser	Beatrice Ruta
ESRF Organisers	Hiram Castillo-Michel
	Sylvain Bohic
	Ana Pradas del Real
	Giulia Veronesi

- Programme
- Abstracts of Keynote & User Talks

UDM2. Nanomaterials life cycle: from nanoengineering to public health

Wednesday, 5th February 2020 - Microsymposium UDM2 Venue: IBS Seminar Room		
8:15	Registration	
8:45	Introduction to the microsymposium UDM2	
	Session I - Design and Applications - Chair: Giulia Veronesi	
8:50 – 9:20	**Keynote talk**: Dissecting the Intracellular Fate of Indium Phosphide Quantum Dots in vivo Using Synchrotron XRF and XANES	**Claudia Tortiglione** *CNR - Istituto di Scienze Applicate e Sistemi Intelligenti, Italy*
9:20 – 9:40	Advanced magnetic spectroscopies for the fine characterization of bimagnetic nanoparticles and ferrofluids	**Amélie Juhin** *IMPMC Paris, CNRS, France*
9:40 – 10:00	Structural evolution of supported lipid bilayers intercalated with quantum dots	**Magdalena Wlodek** *Polish Academy of Sciences, Poland*
10:00 – 10:20	Multi-modal scanning microscopy for nanomaterials	**Michael Stuckelberger** *DESY, Germany*
10:20 – 10:50	*Coffee break*	
	Session II - Nanomaterials and Human Health - Chair: Sylvain Bohic	
10:50 – 11:20	**Keynote talk**: Nanowire technology and toxicity	**Laurent Charlet** *ISTerre, UGA, France*
11:20 – 11:40	Asbestos bodies in human lung tissue: toward a definitive characterization	**Fabrizio Bardelli** *CNR - Nanotec, Italy*
11:40 – 12:00	Understanding nanoparticle cellular entry: A physicochemical perspective	**Wuge Briscoe** *University of Bristol, UK*
12:00 – 12:20	Synchrotron-based imaging reveals silver ions trafficking within hepatocytes exposed to silver nanoparticles	**Aurélien Deniaud** *Université Grenoble Alpes, France*
12:20 – 12:50	**Keynote talk**: Synchrotron imaging of nano-particles released from implants in human and an outlook on new tools to investigate the associated tissue response in 4D	**Bernard Hesse** *Xploraytion GmbH, Berlin Germany*
12:50 – 14:00	*Lunch at the EPN campus restaurant*	
	Session III - Fate in the environment and disposal - Chair: Hiram Castillo-Michel	
14:00 – 14:30	**Keynote talk**: X-ray imaging and X-Ray Absorption Spectroscopy applied to Environmental Nanotechnologies	**Mélanie Auffan** *CEREGE, CNRS, France*
14:30 – 14:50	Investigating the fate of TiO_2 nanoparticles in soils	**Geraldine Sarret** *ISTerre, UGA, France*
14:50 – 15:10	Determining the influence of time, soil properties and the plant–soil interface on silver nanomaterial speciation using x-ray absorption spectroscopy	**Sam Harrison** *UK Centre for Ecology & Hydrology, UK*
15:10 – 15:30	Advanced techniques to investigate the internalization mechanism of TiO_2 NPs in the roots grown in a biosolid-amended agricultural soil	**Eliana Tassi** *Research Institute on Terrestrial Ecosystems, CNR Pisa, Italy*
15:30 – 15:50	Nanoscience Foundries and Fine Analysis (NFFA): the widest range of tools for research at the nanoscale	**Cecile Girardot** *ESRF, France*
15:50	**Discussion - End of meeting**	

Dissecting the Intracellular Fate of Indium Phosphide Quantum Dots in vivo Using Synchrotron XRF and XANES

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The syntheses of environmental friendly semiconductor nanocrystals (QDs) such as Indium Phosphide (InP) QDs are nowadays well-established and they possess high-quality optical properties. Despite these synthetic advances, gaps in knowledge of their intracellular fate, persistence, and excretion from the targeted cell/organism still exist, preventing clinical applications. In this study by using a simple model organism having a tissue grade organization, we determined the toxicological impact of InP QDs [1]. Moreover, we analysed their biodistribution by X-ray fluorescence and complemented these information by mapping the single elements with X-ray absorption near edge structure spectroscopy, achieving unique information on *in situ* chemical speciation. We observed an unexpectedly fast dynamics of QD degradation, occurring within the first hour post incubation [2]. Our study brought new insights into the intracellular fate of photoluminescent nanocrystals after the loss of their optical properties and pave the way for the design of more biological stable InP QDs for biomedical purpose.

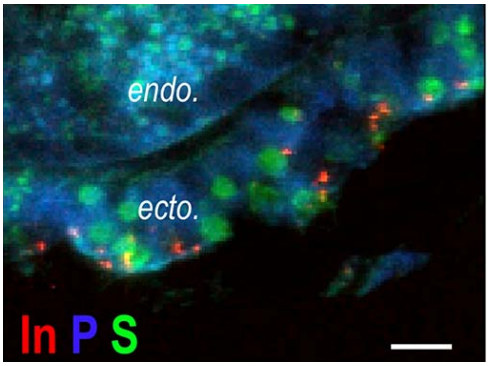

Figure 1. RGB representation ofindium (red), sulfur (green), and phosphorus (blue) distribution in transversal sections of Hydra exposed to core shell InP-QDs

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Advanced magnetic spectroscopies for the fine characterization of bimagnetic nanoparticles and ferrofluids

A. Juhin[1], N. Daffé[1,2,3], M. Sikora[4], J. Zecevic[5], K. Trohidou[6], J. Nogués[7], N. Bouldi[1,2], V. Gavrilov[3], S. Neveu[3], F. Choueikani[2], V. Dupuis[3], P. Ohresser[2], M.-A. Arrio[1], M. Rovezzi[8,9], P. Glatzel[8] and Ph. Sainctavit[1,2]

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We will show how the combination of advanced magnetic spectroscopies (XMCD and hard x-ray RIXS-MCD [1,2]) can allow a profound understanding of the electronic and magnetic structures in complex magnetic nanomaterials such as bimagnetic nanoparticles and ferrofluids, and reveal emergent properties.

First, we have investigated **bimagnetic core-shell nanoparticles** that currently focus high interest owing to their applications in biomedicine and technology. The fine tailoring of particles requires a deep knowledge of their internal structure and morphology, from which the properties are directly inherited. In nominally γ-Fe_2O_3/Mn_3O_4 nanoparticles, RIXS-MCD gives the smoking gun evidence for the existence of a magnetic interdiffused inner shell growing from a γ-Fe_2O_3core and a Mn_3O_4 shell. Combined with TEM-EELS experiments, a quantitative multilayered structure is proposed, which allows understanding the influence of the interface quality on the measured magnetic properties [3].

Second, we have studied the magnetic anisotropies in a **ferrofluid of monodispersed $MnFe_2O_4$@$CoFe_2O_4$ nanoparticles** dispersed in heptane. Ferrofluids are well-known for their applications in optical waveguides, medicine or in fine arts. Their magnetic properties arise from both magnetic anisotropies of individual particles and interparticle interactions that are mediated by the liquid carrier. Using a dedicated liquid cell, developped in collaboration with ID26 beamline of the ESRF, we have measured element-selective magnetic properties in the liquid phase and in the frozen phase. This allowed investigating separately the cationic distribution and magnetic anisotropies in the core and those in the shell, as well as their mutual influence [4].

Third, a **binary ferrofluid of $MnFe_2O_4$ and $CoFe_2O_4$ nanoparticles** dispersed in heptane was investigated. The measurement of element-selective hysteresis curves, supported by cryogenic TEM experiments and Monte-Carlo simulations of the magnetic properties, has allowed quantifying the effect of interparticle interactions on the magnetic properties for each of both magnetic phases [5].

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Structural evolution of supported lipid bilayers intercalated with quantum dots

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Understanding interactions between functional nanoparticles and lipid bilayers is important to many emerging biomedical and bioanalytical applications. In particular, quantum dots (QDs; semiconductor nanoparticles) are promising components of functional systems for medical imaging, theranostics, targeted therapy, drug delivery and biosensing. Increasing use of QDs in biological applications also motivates the investigation of their physical interactions with biological systems, particularly the cell membrane. However, little is known about the influence of embedded hydrophobic QDs on the formation process of supported lipid bilayers (SLBs) *via* vesicle fusion, and how the presence of hydrophobic QDs influences the morphology and structure of the SLBs. Such an understanding is important to their bioanalytical applications and potential cytotoxicity.

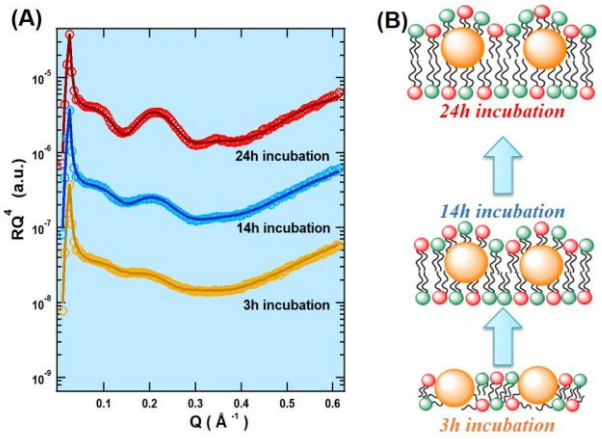

Figure 1. XRR curves of POPC/POPE lipid bilayers with 4.9nm CdS QDs at 3h, 14h and 24h incubation time (A) with schematic of the structural evolution and QD rearrangement (B).

The main goal of this study was to investigate the structural changes of negatively charged POPC/POPE supported lipid bilayers intercalated with QDs on a PEI monolayer as a function of incubation time, using *in situ* synchrotron XRR, with the resulting morphology imaged by atomic force microscopy (AFM) [1]. The structural properties such as thickness, roughness and surface coverage were observed over a period of 3-24 h. Our results show time-dependent perturbations in the SLB structure due to the interaction upon QD incorporation. Compared to the SLB without QDs, at 3 h incubation time, there was a measurable decrease in the bilayer thickness and a concurrent increase in the scattering length density (SLD) of the QD-SLB. The QD-SLB then became progressively thicker with increasing incubation time, which – along with the fitted SLD profile – was attributed to the structural rearrangement due to the QDs being expelled from the inner leaflet to the outer leaflet of the bilayer (Figure 1B) [2]. These findings will provide valuable information on the structure of QD-containing fluorescent SLBs, giving unprecedented mechanistic insights, which could have potential implications for drug delivery, cell toxicity, and related aspects of NP-cell interactions.

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Multi-modal scanning microscopy for nanomaterials

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Nanomaterials pose unique challenges to X-ray microscopy for their characterization with respect to alignment, beam damage, and signal-to-noise ratio. At the same time, relevant information about nanomaterials is typically extracted from the point-by-point correlation of different properties, which requires the same spot being in the same condition for all measurements. Particularly for *in-situ* and *operando* measurements, this is not possible without the simultaneous evaluation of the critical measurement modes.

At various synchrotrons worldwide (APS, PETRA III, NSLS II, CLS, ESRF), we have set up experiments for multi-modal measurements involving up to 5 different modalities of nanomaterials and devices such as contacted nanowires or solar cells as depicted in Fig. 1. They allow the simultaneous evaluation of composition, structure, and performance.

In this contribution, we will demonstrate the application of multi-modal X-ray microscopy to nanoscale semiconductors and electronic devices, and discuss detector arrangement and compatibility with different scan modes and samples. Beyond state-of-the-art measurements such as shown in Fig. 2, we will give an outlook to new opportunities and challenges at nanoprobe endstations of 4th generation synchrotrons such as ID-16-B at ESRF-EBS.

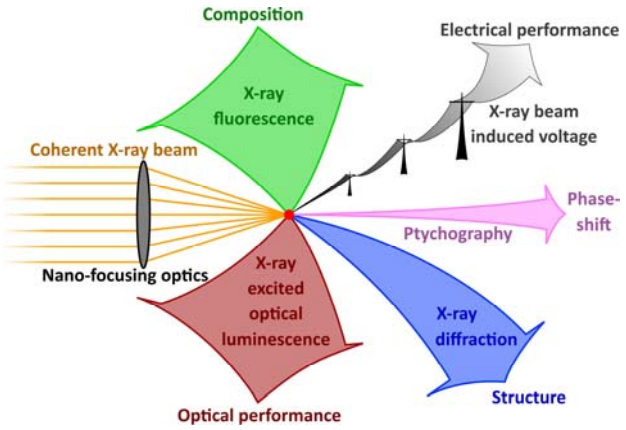

Figure 1: A "dream experiment" involving five-fold multi-modality in scanning X-ray microscopy [1]

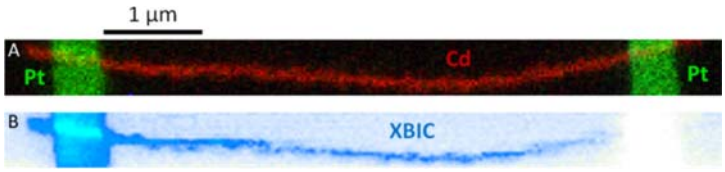

Figure 2: Multimodal measurement of the composition (top) and the performance (bottom) of a nanowire [2].

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Nanowire technology and toxicity

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Fibrous particles interact with cells and organisms in complex ways that can lead to cellular dysfunction, cell death, inflammation, and disease. The development of conductive transparent networks (CTNs) composed of metallic silver nanowires (AgNWsfor flexible touchscreen displays raises new possibilities for the intimate contact between novel fibers and human skin. Here, we report that a material property, nanowire-bending stiffness that is a function of diameter, controls the cytotoxicity of AgNWs to nonimmune cells from humans, mice, and fish without deterioration of critical CTN performance parameters: electrical conductivity and optical transparency. As shown by ID 16A holographic X-ray phase contrast maps and 2D elemental maps, completed by ID21 Ag LIII-edge X-ray absorption spectra, both 30- and 90-nm-diameter AgNWs are readily internalized by cells, but thinner NWs are mechanically crumpled by the forces imposed during or after endocytosis, while thicker nanowires puncture the enclosing membrane and release silver ions and lysosomal contents to the cytoplasm, thereby initiating oxidative stress. This finding extends the fiber pathology paradigm and will enable the manufacture of safer products incorporating AgNWs.

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Asbestos bodies in human lung tissue: toward a definitive characterization

F. Bardelli, F. Brun[1], G. Veronesi[2], A. Cedola, P. Cloetens[3]

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Thanks to their dimensions, 10-100µm in length and 200-300nm in diameter, and elevated bio-persistency, asbestos fibers can penetrate the lungs by inhalation and remain there for long time (even decades). In the lungs, alveolar macrophages are in charge of removing bad particles. They can do an excellent job while dealing with the majority of particulate matter, but when it comes to high aspect ratio materials, as asbestos (>20), macrophages are no more able to completely engulf them (frustrated phagocytosis). Organic and inorganic material start to deposit on the foreign fibers giving rise to an *in vivo* biomineralization process that leads to the formation of peculiar structures, consisting of the original asbestos fibers plus an Fe-rich layer. These structures are commonly referred as Asbestos Bodies (AB, Fig. 1a).

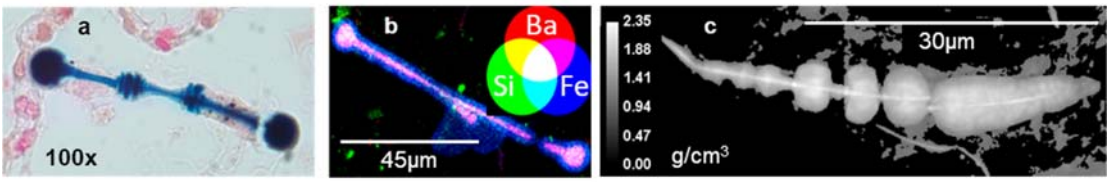

Figure 1: a) Optical microscopy image of an AB in lung tissue; b) µXRF map of an AB (ID21@ESRF, pixel size 0.5x0.5µm^2); c) Density map of an AB in lung tissue (ID16A@ESRF, pixel size 0.07x0.07µm^2).

Asbestos can lead to mesothelioma, an aggressive cancer of the lung lining. Despite the link between asbestos exposure and mesothelioma has long been demonstrated, little advancement has been made in anti-cancer therapies and, still, only one person over twenty is alive five years after the diagnosis. In fact, since the latency period from the first exposure to the development of mesothelioma can be very long (20-40 years, on average), while AB start to form soon after asbestos reaches the lungs, it is reasonable to believe that they may play a major role in the pathogenesis. Indeed, AB have been shown to induce the formation of reactive oxygen species and DNA damage. One of the open questions that prevent developing a sound model to explain the carcinogenic mechanism is that a full knowledge of the growth mechanism and composition of the AB is still lacking, preventing determining their role in the pathogenesis. The common methods exploited to study AB require invasive sample preparation, such as recovering them after removing the biological tissue by chemical digestion. Nevertheless, this could alter their composition and the spatial information is usually lost. Conversely, synchrotron-based imaging and micro-probe techniques available at the ESRF, allowed studying single AB without altering the original lung tissue.

Scanning micro X-ray fluorescence (µXRF) and Fe K-edge micro X-ray absorption spectroscopy (µXAS) have been performed at the ID21 beamline, allowing for the determination of the elemental distribution and of the Fe speciation [1] (Fig. 1b).

Phase-contrast and fluorescence x-ray nano-tomography have been performed at the ID16A beamline to reveal the 3D morphology, and obtain a reliable elemental quantification. The latter was achieved by combining elemental distribution, thickness, and mass density high resolution maps (down to 25nm, Fig. 1c). The above techniques can be successfully exploited also to study other types of health-threatening micro- and nano-materials, such as, for example, those that are believed to be able to cross the brain barrier or the placenta.

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Understanding nanoparticle cellular entry: A physicochemical perspective

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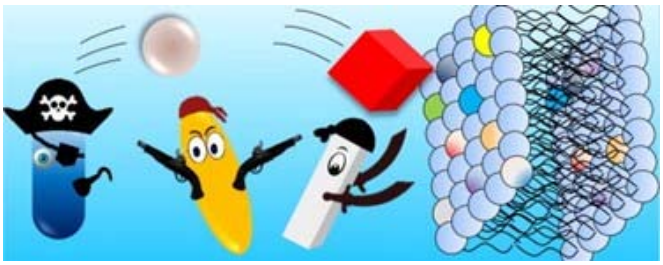
A key mechanism for nanoparticles (NPs) to impart toxicity is to gain cellular entry directly. Many parameters affect the interactions of nanomaterials in a cellular environment with cell membranes, including their size, shape and surface chemistry as well as factors such as the cell type, location and external environment (e.g. other surrounding materials, temperature, pH and pressure) [1, 2].



In addition to *in vitro* and *in vivo* experiments, model cell membrane systems have been used in both computer simulations and physicochemical experiments. We have used model membrane systems and physicochemical methodologies to study nanoparticle-membrane interactions. Our results from high pressure small angle X-ray scattering (HP-SAXS) show that *hydrophobic* nanoparticles could encourage the lamellar to inverted hexagonal phase transition [3], whereas the effect of *hydrophilic* nanoparticles depends on their concentration [4], with more recent work showing that dendritic polymer nanoparticles could cause membrane thinning and structural disorder in lipid mesophases [5]. In addition, using X-ray reflectivity (XRR), we have observed structural re-organisation in supported lipid bilayers intercalated with quantum dots [6, 7] and dendritic nanoparticles [8].

These results shed light on how the fundamental energetic process of NP cellular entry can be evaluated by studying the effects of nanoparticles on lipid mesophase transitions and structural disorder. This highlights both the challenge and the opportunity in this interdisciplinary area, where collaborative efforts from the insights and expertise of biological and physical scientists are urgently needed for future progress.

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Synchrotron-based imaging reveals silver ions trafficking within hepatocytes exposed to silver nanoparticles

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The widespread use of silver nanoparticles (AgNP) in consumer goods raises concerns about their toxicity to humans and their impact on environment [1]. AgNP toxicity in cells and animals has been extensively studied and it has been shown that the toxicity depends upon the release of Ag(I) ions from the NP [2,3]. Besides, Ag accumulates in liver following AgNP exposure [4]. In this context, we studied AgNP internalization and fate into hepatocytes. We made use of a synchrotron nanoprobe to visualize the subcellular distribution of silver. The combined use of X-ray fluorescence (XRF) microscopy on whole cells and electron microscopy allowed the discrimination between the nanoparticle form located inside endosomes and lysosomes and the ionic species that distribute throughout the cell [5]. Besides, synchrotron X-ray absorption spectroscopy showed that Ag(I) recombines with sulphur in hepatocytes in the form of AgS_2 and AgS_3 complexes [5,6].

More recently, we developed a nano-XRF method performed on cell sections (Figure 1) that can be correlated with electron microscopy to reveal Ag(I) species distribution at the organelle level under long-term exposure to non-toxic concentration of AgNPs. We thus observed Ag(I) species in different organelles including in the nucleus [7]. This approach was also used on sections from 3D hepatic cell cultures that mimic liver architecture including bile canaliculi. XRF allowed to visualize Ag(I) excretion into these intercellular structures. To get more insights into the fate and effects of AgNPs, these data were completed with 3D electron microscopy, STEM-EDX and physiology assays. The later revealed, for the first time, that Ag(I) species translocating into the nucleus can trigger an endocrine disruptor-like effect. Overall, synchrotron-based imaging was central in our studies that aim at understanding the fate of nanomaterials in cells and organisms.

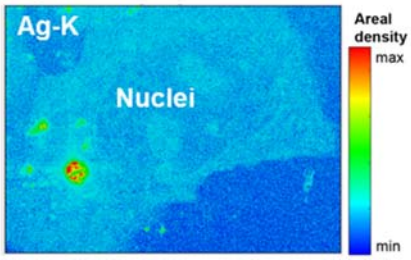

Figure 1: Ag XRF map of a hepatocyte section exposed to AgNPs.

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Synchrotron imaging of nano-particles released from implants in human and an outlook on new tools to investigate the associated tissue response in 4D

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The exposure to toxic elements in humans is an increasing concern. Sources of exposure are multifold: food, cosmetics, dust in air, tattoo colorants, pharmaceutical substances or medical implants, to only name a few. For medical implants the balance between patient benefit and associated risk can be delicate. The doctors decision crucially depends on the awareness and understanding of the exposure scenario and its impact on the cellular level and thus also on the patient's health. Regulations for the approval and market surveillance of applied biomaterials have recently been challenged on a cross-national level.

Our current findings revealed that human peri-implant bone and bone marrow are distinctively exposed to micron- and nanoparticles consisting of cobalt, chromium and titanium. The detected metal quantities and their element- and tissue-specific distribution provide evidence that the peri-implant membrane does not chemically isolate implant components. Our work reveals toxico-kinetic mechanisms and a novel view on the long-term effects of metallic degradation products. Our findings prompt for a paradigm shift towards a consideration of bone and bone marrow being the most relevant organs for pre-clinical testing and post-clinical risk-benefit evaluation of orthopedic biomaterials.

By proving that metal accumulation occurs in peri-implant bone and bone marrow, our findings add significance to in vitro studies using cells of bone and bone marrow origin as models for evaluating functional and cytotoxic effects of acute metal exposure.

To extend the meaningfulness of existing *in vitro* studies based on a bone-on-chip model we currently develop a sample holder that allows for a controlled liquid environment, usable in Synchrotron CT setups, exploiting the high sensitivity and spatial resolution at very high scanning speeds. The aim is to have a time-resolved 3D bone tissue response after controlled metal particle exposure as previously assessed in real human conditions. By that we will not only have assessed the exposure scenario but also its impact on living tissue helping for saver materials and thus better patient benefit. Existing methods rely on animal experiments. Bringing organ-on-a-chip to the Synchrotron will thus help to reduce use of animals which has not only an ethical but also a commercial advantage for R&D of pharmaceutical or medical device industry. Eventually, combining data obtained through high resolution chemical imaging at ID16B and ID21 with time-resolved 3D in-vitro imaging such as at ID19 will draw a picture that could not be generated without sophisticated facilities like the ESRF.

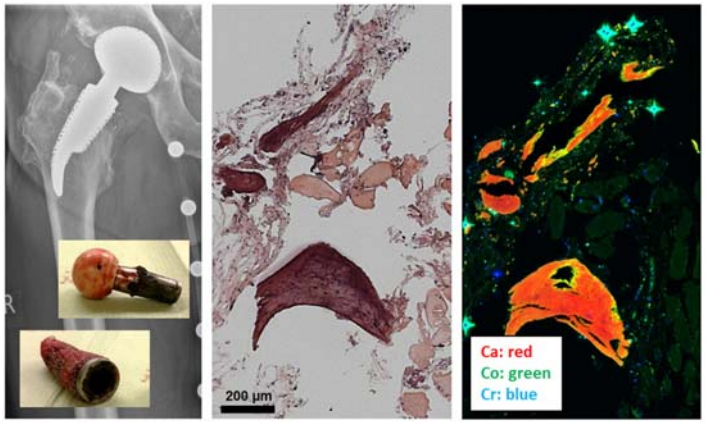

Figure 1: Left: Radiography of a hip joint and corroded implant pieces after explantation. Middle: Histology image showing bone and bone marrow. Right: XRF map collected at ID21 showing Co and Cr deposition into the bone marrow tissue regions.

X-ray imaging and X-Ray Absorption Spectroscopy applied to Environmental Nanotechnologies

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Engineered nanomaterials (ENMs) have become a fast growing economic sector. As a consequence of the many debates concerning their safety, efforts are developed at international and national levels to develop a code of ethics for a safe and responsible development of ENMs. A sustained growth of the nanotechnology industry will rely heavily on the characterization of risks to the environment (water and soil resources, trophic transfers, biodiversity) and human health that may be posed by ENMs in relevant exposure conditions (low doses, mid-/long-term, trophic and transgenerational transfers, etc.)

In this regard, physical-chemists, (micro)biologists, and ecologists need to conduct meaningful experiments to study the environmental risk of ENMs with access to relevant mechanistic data across several spatial and temporal scales (Auffan et al. 2019). Experimental devices as mesocosms that can be tailored to virtually mimic any ecosystem appear as particularly well-suited (Auffan et al. 2014) for the determination of the (bio)degradability, (bio)distribution, (bio)transformation, and impacts of ENMs. However, adhering to environmentally relevant exposure scenarios implicitly represents a technical challenge since it requires to explore the localization and the speciation of a target chemical element at relevant and consequently low doses in complex matrices, which is critical in the fields of environmental and biogeochemistry sciences.

These past few years, the significant improvement of X-ray imaging (2D and 3D) and X-Ray Absorption Spectroscopy techniques in term of detection limit and resolution (spectroscopic and spatial) helped us to determine unambiguously and with greater precision the speciation and distribution of the probed metal composing ENMs in sediment, biota, nanomaterials... The positive impact of these techniques will be discussed based on examples dealing with the behavior and fate of TiO2-, CeO2- and Ag-based ENMs in ecologically relevant conditions (Tella et al. 2014, 2015) and obtained both on synchrotron beamlines and laboratory apparatus.

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Investigating the fate of TiO_2 nanoparticles in soils

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Nanosized TiO_2 is one of the most produced nanomaterials, and there is an intense debate at the EU level on the ban of TiO_2 additive (E171) in food products. TiO_2 nanoparticles (TiO_2-NPs) released from consumer products end up into the sewer system, and are accumulated in sewage sludge. Agricultural soils are a major compartment of accumulation due to the use of sewage sludge as a soil amendment. TiO_2 is naturally present in soils, and distinguishing between anthropogenic and natural TiO_2 in soils is thus important to measure anthropogenic inputs and to assess the risks associated with the use of nano-TiO_2.

Methods to identify these materials in complex matrices such as soils are currently lacking. In this study, the potential of physical techniques (micro and nano X-ray fluorescence (XRF) performed on ID21 and ID16b, X-ray absorption spectroscopy (XANES), X-ray diffraction (XRD) performed on BM25 and transmission electron microscopy coupled with X-ray microanalysis (TEM-EDX)) to distinguish natural versus anthropogenic particles has been investigated [1]. Three matrices were compared: sewage sludge, agricultural soil that had never received sewage sludge, and sludge-amended soil. Particle size and crystal structure were not specific of the source. The morphology of the TiO_2 particles proved to be different in the two matrices studied, with smooth faceted particles in the sludge and rough irregular ones in the soil. In addition, natural TiO_2 particles were included in micro and macroaggregates of the soil and formed intricate assemblages with minerals and organic compounds. In the sludge, TiO_2 formed homo and heteroaggregates of simpler structure, richer in organic matter. The observed differences may attenuate over time due to the weathering of TiO_2 minerals and to the progressive incorporation of anthropogenic TiO_2 within soil aggregates. So it is likely that with time, engineered TiO_2 becomes indistinguishable from the natural background in soils.

Then, the effects of sewage sludge containing TiO_2-NPs used as an amendment in agricultural soil were assessed on plants (tomato) during a full plant life cycle (until fruit ripening)[2]. The sewage sludge amendment increased plant growth without causing major changes in biochemical responses, except for a decrease in leaf tannin concentration. Changes in elemental concentrations (mainly Fe, B, P, Na, and Mn) of plant stem, leaves and, to a lesser extent fruits were observed. No significant Ti enrichment was detected in tomato fruits. Fourier-transformed infrared (FTIR) analysis performed on ID21 showed effects on plant leaves (decrease in tannins and lignins and increase in carbohydrates) but no effects on fruits. In conclusion, the sewage sludge amendment containing TiO_2-NPs improved plant yield probably due to its high organic matter and nutrient content, and did not lead to significant changes in the edible part of tomato. Effects on the long term, with increasing TiO_2 inputs should be evaluated, as well as effects on other crops and on soil bacterial communities.

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Determining the influence of time, soil properties and the plant–soil interface on silver nanomaterial speciation using x-ray absorption spectroscopy

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Silver nanomaterials (nano-Ag) are one of the most abundant engineered nanomaterials in the consumer market, with a plethora of uses largely based on exploiting their antimicrobial properties, such as textiles, medical applications and cleaning products [1]. A major pathway for nano-Ag release into the environment is the application of sewage sludge to agricultural soils. Their chemical form in soils affects the overall impact they have on the soil ecosystem, for example in the rate at which nano-Ag is taken up by organisms [2] and the toxicity of nano-Ag which is taken up [3]. While nano-Ag behaviour in soils has been studied in some detail, few have extended this to examining uptake across the plant–soil interface. Here, we detail studies into nano-Ag speciation in a range of cropped soils with different properties, including the role of the plant–soil interface in determining speciation, as well as speciation in the plant roots themselves. The work was undertaken using Ag K-edge XANES and EXAFS spectroscopy performed at Diamond Light Source (Oxford, UK) and The Australian Synchrotron (Melbourne, Australia).

Three different soils with varying characteristics were dosed with three different forms of Ag: pristine 20-nm Ag nanoparticles, aged 27-nm Ag_2S nanoparticles and the dissolved, ionic form (as $AgNO_3$), to concentrations of 10 mg Ag/kg soil dry weight. Wheat (*Triticum aestivum*) was germinated in the dosed soil and the bulk soil sampled at determined time points. In addition, to investigate the effect of the plant–soil interface, for one of the soils at 14 days post emergence of shoots, soil fractions were operationally separated by their proximity to the root: as bulk soil (soil unaffected by the roots), loosely-attached soil (which could be removed from the roots by shaking) and rhizosphere soil (closely adhered to roots).

In this presentation, we will highlight the key results, demonstrating the importance of soil properties, time and proximity to roots on Ag speciation. For example, we show sulphidation is the dominant ageing transformation, but the rate at which pristine nano-Ag are sulphidised depends on soil properties. We will discuss these results in the context of environmental exposure and risk, and detail how these results have been incorporated into multimedia nanomaterial exposure models for next-generation exposure assessment.

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Advanced techniques to investigate the internalization mechanism of TiO_2 NPs in the roots grown in a biosolid-amended agricultural soil

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Plants play an important role in introducing the engineered nanoparticles (ENPs) into the food chain. The pathway of ENPs uptake from soil, their distribution in the edible plant parts, and their impact in the food production are important issues to be investigated. In the present study, *Pisum sativum* plants were grown at microcosm scale under medium-term TiO_2 NPs exposure, to possibly mime environmental conditions in an agricultural soil amended with biosolids from a wastewater treatment plant in Pisa, Italy. TiO_2 NPs were applied as pure rutile, pure anatase and a mixture of both crystalline phases in the biosolid amended-soil.

Micro-XRF and μ-XANES from ID21 beamline were used for Ti elemental mapping and crystalline phase identification to indicate a relative distribution/localization of TiO_2 crystalline phases within a given cross-section of roots, as well as the possible speciation and preferential crystalline phase uptake in the roots. Titanium in roots showed a main localization in the rizoderma, independently of the crystalline phase. Fewer Ti spots were found localized in the cortex or in vessel, however the roots grown in presence of a mixture of both phases showed a main presence of anatase, suggesting a preferential adsorption and translocation of this crystalline form through the roots. Our data indicated also a reduced translocation of Ti to the aerial part of the plant, confirming the chemical analysis of shoots and roots separately, which showed that Ti concentration was about 40 times lower in the upper part than in the below ground tissues.

The TiO_2 NPs were characterized on the basis of their size and shape by TEM analysis. Moreover, observations on cell ultrastructure of control and of anatase, rutile and mixture of both crystalline phases treated roots were performed. The root cells of plant grown in the presence of all NPs treatments shared the same alterations of ultrastructure: mitochondria with swollen cristae, nuclei with condensed chromatin, and part of the cytoplasm degraded, probably in consequence of an autophagic process. As detected by μ-XRF and μ-XANES, electron dense prismatic or round profiled particles of about 30-40 nm were observed mainly in form of aggregates in the intercellular spaces or crossing the wall of the cells next to rizoderma and in the cortex cells. Furthermore, the anatase treated cells were mostly damaged in respect to control and rutile treated roots, and more frequently internalized NPs were observed in these samples.

Nanoscience Foundries and Fine Analysis (NFFA): the widest range of tools for research at the nanoscale

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NFFA•EUROPE is a distributed research infrastructure which offer access to state-of-the-art facilities and instruments for multidisciplinary research projects at the nanoscale. It deals with a large panel of research field from synthesis to nanocharacterization to theory and numerical simulation. In particular fine analysis with Synchrotron, FEL and Neutron radiation sources. Each institutes is integrated in a local node to offer transnational access and enable European and international researchers from diverse disciplines to carry out advanced proposals impacting science and innovation. NFFA enables coordinated access to infrastructures on different aspects of nanoscience research that is not currently available at single specialized ones and without duplicating their specific scopes.

Approved user projects have access to the best-suited instruments and support competences for performing the research. Their access includes several “installations” and is coordinated through a single entry point portal to build up a personalized access programme with an increasing return on science and innovation production. Until now, the majority of submitted proposal are linked with materials science, chemistry or physics, but there is a will to enlarging the community of NFFA’s user and especially increase proposal dealing with nanosafety research topics, nanosafe by desing and life cycle assessment of nanoparticles.

We will present NFFA project and collect every feedback for potential future enlargement of the catalogue, in particular to intercept the needs from the nanosafety community.

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User-Dedicated Microsymposium UDM 3

5th February 2020

Multi-crystal and serial data collection in Structural Biology

UOC Organiser	Marina Mapelli
ESRF Organisers	Max Nanao
	Daniele de Sanctis

- Programme
- Abstracts of Keynote & User Talks

UDM3. Multi-crystal and serial data collection in Structural Biology

Wednesday, 5th February 2020 - Microsymposium UDM3 Venue: ESRF Auditorium		
8:15 – 8:50	Registration	
8:50 – 9:00	Introduction to the microsymposium UDM3 by Marina Mapelli	
	Session I - Challenges in Serial Crystallography - Chair: Max Nanao	
9:00 – 9:30	**Keynote talk**: Non-isomorphism: ancient enemy? Or blessing in disguise?	**James Holton** *Advanced Light Source Berkeley, USA*
9:30 – 9:50	Measuring energy-dependent photoelectron escape in microcrystals	**Selina Storm** *Diamond Light Source Didcot, UK*
9:50 – 10:10	X-ray data collection and structure solving of Dps protein by multiple-crystal macromolecular crystallography methods	**Yurii Krupyanskii** *Semenov Institute of Chemical Physics Moscow, Russia*
10:10 – 10:40	*Coffee break*	
10:40 – 11:10	**Keynote talk**: Theory and methods in microcrystallography of biological macromolecules	**Michele Cianci** *Ancona University, Italy*
11:10 – 11:40	**Keynote talk:** Synergy between synchrotrons and free electrons laser to study ion transport with time-resolved serial crystallography	**Przemyslaw Nogly** *ETH Zurich, Switzerland*
11:40 – 12:00	Free-jet sample injection for synchrotron serial data collection	**Bruce Doak** *MPI Heidelberg, Germany*
12:00 – 14:00	*Lunch at the EPN campus restaurant*	
	Session II - Serial Experimentation and Data Analysis - Chair: Daniele de Sanctis	
14:00 – 14:30	**Keynote talk:** Data analysis methods for two-dimensional serial femtosecond crystallography: paving the way to the time-resolved study of large-scale movements in membrane proteins	**Cecilia Casadei** *PSI Villigen, Switzerland*
14:30 – 14:50	Towards a protein-based crystal host system	**Janina Sprenger** *University of Copenhagen, Denmark*
14:50 – 15:10	An automated and universal approach for high-throughput serial crystallography of membrane proteins using CrystalDirect technology	**Shibom Basu** *EMBL Grenoble-outstation, France*
15:10 – 15:30	Serial crystallography at the SPB-SFX instrument at the European XFEL	**Adam Round** *European XFEL Schenefeld, Germany*
15:30 – 16:00	**General discussion and closing remarks**	

Non-isomorphism: ancient enemy or blessing in disguise?

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The problem of non-isomorphism has plagued macromolecular crystallography since the beginning [1, 2, 3], but if used properly it may prove instrumental in solving the phase problem by over-sampling the molecular transform [4] and studying structural flexibility. Changes in unit cell can be a tell-tale sign of non-isomorphism, but in general these are neither necessary nor sufficient to indicate incompatibility of the underlying structure factors. Why are they incompatible anyway? If the underlying protein structure is the same, then why aren't the structure factors? Simple rigid-body motions cannot be the whole story because these lead to steric clashes. The true underlying distortion of the molecule is most likely smoothly varying from one end of the unit cell to the other, and, of course, must also obey crystallographic symmetry. Here I present how periodic rubber-like distortions may be modelled using a collection of sine waves in space. This spatial distortion field (SDF) is similar in mathematical form to the Fourier synthesis of electron density from structure factors. The main differences are that the SDF is not a scalar field like electron density but a vector field describing changes in atomic position at every point in the unit cell. In adition, the number of terms in the SDF required to describe typical non-isomoprhism is relatively small: 3-5 orders are usually sufficient. After applying this SDF to a protein model the RMS deviation between coordinates is comparable to rigid-body alignment with the exception that there are no steric clashes. In addition, the SDF may be applied to electron density, allowing multi-crystal averaging across non-isomorphous crystal forms. This is essentially equivalent to evaluating the molecular transform between the Bragg spots. Structural flexibility inherent to function may also be excited by these rubber-like distortions, making SDFs a potentially useful tool for elucidating subtle changes by eliminating the "noise" of rubber-like non-isomorphous distortion.

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Abstract submitted for contribution to the User-dedicated Microsymposium 3 (UDM3):

Measuring energy-dependent photoelectron escape in microcrystals

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With the increasing trend of using microcrystals and intense microbeams at synchrotron X-ray beamlines, radiation damage becomes a more pressing problem. Theoretical calculations by Nave and Hill [1] show that the photoelectrons primarily causing damage can escape microcrystals. This effect would become more pronounced with decreasing crystal size as well as at higher energies [2, 3]. To prove this effect, data from cryo-cooled lysozyme crystals of dimensions 5 x 3 x 3 μm^3 and 20 x 8 x 8 μm^3 mounted on cryo-transmission electron microscopy (TEM) grids were collected at 13.5 keV and 20.1 keV using a 2M CdTe Pilatus detector, which has similar quantum efficiency at both energies. Accurate absorbed doses were calculated with RADDOSE3D [4] through direct measurement of individual crystal sizes using scanning electron microscopy after the experiment and characterization of the X-ray microbeam. The data were processed with DIALS [5] and crystal lifetime was then quantified based on the $D_{1/2}$ metric. In this first systematic study, a longer crystal lifetime for smaller crystals was observed and crystal lifetime increased at higher X-ray energies supporting the theoretical predictions of photoelectron escape. The use of detector technologies specifically optimised for data collection at energies above 20 keV allows the theoretically predicted photoelectron escape to be quantified and exploited, guiding future microfocus beamline design choices.

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Abstract submitted for contribution to the User-dedicated Microsymposium 3 (UDM3):

X-ray data collection and structure solving of Dps protein by multiple-crystal macromolecular crystallography methods

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In recent years, the increasing brightness of X-ray beamline for macromolecular crystallography has made it possible to obtained appropriative diffraction data for further procession by standard crystallographic software tools from crystals of very small size. To obtain bright enough diffraction spots we should deliver more radiation dose to the crystal, which leads to the critical radiation damage before we able to collect enough data to solve the crystal structure. However, if a few degrees of oscillation data per crystal are available, diffraction images can be processed by standard crystallographic software, and when the resulting partial datasets were checked for high level of isomorphism, them could be merged to produce the final complete data set.

In present work the process of diffraction data collection from Escherichia coli bacteria Dps protein crystals 3-7 micron sized was described. The study of influence on final data set of various data collection parameters such as exposure time and diffraction wedge wideness per one crystal were carried out. Here, to achieve the best result in selecting the isomorphs partial datasets for merging hierarchical cluster analysis was applied. This method use distance between data sets a and b calculated from correlation coefficient (cc(a,b)) between common intensities of these sets ($d(a,b) = \sqrt{(1 - cc(a,b)^2)}$) as a metric for non-isomorphism. The calculations of these distance performed by ccCluster program [1].

Final diffraction data set consist of 256 monocrystal diffraction data, overall 450 monocrystals diffraction data was processed by XDS software [2]. The highest resolution of obtained structure is 2.2 Å, R-factor free value for this resolution equal 0.2598. Structure was deposited in PDB with unique four letter code as 6QVX.

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Abstract submitted for contribution to the User-dedicated Microsymposium 3 (UDM3):

Theory and methods in microcrystallography of biological macromolecules

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During the last decade, crystallography of microcrystals has become the method of choice for a large number of projects in structural biology. Still today, attempts to collect data from microcrystals of 5–20 μm at their longest dimension, require a dedicated strategy and multi-crystal data collection. While most of the crystal structures can be solved by molecular replacement, in many cases still experimental phasing from microcrystals is needed.

De novo determination of macromolecular structures requires accurate measurement of structure factors and thereby estimation of the phases from the crystals of the given specimen. The anomalous signal from naturally occurring (S, P, Ca, etc) or incorporated (Se, Hg, etc) anomalous scatterers, can be harnessed with a Single-wavelength Anomalous Dispersion (SAD) experiment. Today, the properties of new generation synchrotrons, or new long-wavelength tunable beamlines for microcrystals [1,2], optimization of the X-ray scanning routines, data collection and processing flows [3], new algorithms for data merging [4,5], allow to collect, in just few hours, a full data set with anomalous signal by merging data from more than hundred micro crystals collected thus enabling X-ray diffraction data collection and phasing in microcrystallography (Fig. 1) [5,6]. Moreover, by conducting an extensive survey of 115 PDB sulphur SAD depositions and testing the statistical distribution that these represented, a useful predictor for aiding experimental success using sulphur SAD was developed [7]. So, we will present the current state-of-art of theory and methods in our hands for data collection and phasing in microcrystallography of biological macromolecules and wishes for the future.

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Synergy between synchrotrons and free electron lasers in studying ion transport with serial crystallography

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Ion pumping microbial rhodopsins are integral membrane proteins employing a common 7-transmembrane helices architecture to transport different ion types. The specific residue composition impacts the protein dynamics and transport mechanism.

Rhodopsins utilize retinal chromophore to harvest light energy for protein activation, which makes them an ideal target for pump-probe experiments. We employ serial crystallography to capture structural intermediates in “real-time” and at non-cryogenic temperatures. I will present a combination of the X-ray Free Electron Laser and more accessible synchrotron data, which provide complementary insights into protein dynamics and ion transport.

Abstract submitted for contribution to the User-dedicated Microsymposium 3 (UDM3):

Free-jet sample injection for synchrotron serial data collection

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Seminal Serial Femtosecond Crystallography (SFX) measurements were carried out at the LCLS X-ray Free-Electron Laser (XFEL) in Dec-2009, one decade ago. It was obvious even then that XFEL experimentation would suffer from a severe limit on the possible number of XFEL endstations. In contrast, there is no sparsity of synchrotron endstations. Less than four years later, our group therefore initiated a collaboration with the PXII beamline of the Swiss Light Source (SLS) to adapt serial crystallography for synchrotron use. Instrumentation and techniques were developed during six SLS beamtimes between Oct-2013 and May-2014, with our existing rod-mount MPI XFEL High Viscosity Extrusion (HVE) injector simply bolted directly onto the PXII goniometer. A paper describing this very first Synchrotron Serial Crystallography (SSX) was submitted in Jun-2014 and published in due course [1]. An HVE injector head is much more massive than a sample loop, however, and so basically incompatible with the delicate fine motion drives of a synchrotron goniometer. As an interesting side note, this is *not* the case for a miniature low viscosity sample injector (a "GDVN" injector) we designed and fabricated at the same time, building it into a conventional SPINE button for universal and standard goniometer mounting. In Oct-2013 synchrotron GDVN was well ahead of its time, but will come into its own if high intensity synchrotrons eventually permit X-ray exposures of one microsecond duration or less. We later transitioned our SLS HVE experiments to our block-mount MPI injectors, attached via a miniature manual XYZ stage to the weight-tolerant spindle platform of the goniometer. This was a suitably robust arrangement, but not optimal. An alternative scheme is to mount the HVE injector on a separate, stout, remotely-controllable XYZ stage. This was the approach taken as our SSX technology was ported to the ESRF ID30A beamline in Jun-2016 and it remains a favoured approach in current SSX experiments, mostly since an existing injector can be employed with little or no redesign or modification. Nevertheless an HVE injector can certainly be mounted directly onto a synchrotron goniometer head, provided the weight and torque limitations of the head are carefully taken into account during the design. Constraints are also imposed by the X-ray collimator and beam stop, by the optical monitoring microscope and illumination, by the orientation of the goniometer axis (specifically, whether horizontal or vertical), and by the need to collect extruded sample. Our most recent MPI SSX injector, designed in this manner, has now been installed and is available for use at the BioMAX endstation of the MAX IX synchrotron. Apart from allowing the injector nozzle to be positioned by use of the goniometer drives, such a "bolt-on" injector permits any synchrotron endstation having the same goniometer fixture to immediately be converted to SSX use. This SSX injector development will be presented and the future of HVE injection at high brilliance synchrotrons discussed. The author is the leading international expert on design and use of free-jet sample injectors, both low and high viscosity and for both XFELs and synchrotrons.

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Data analysis methods for two-dimensional serial femtosecond crystallography: paving the way to the time-resolved study of large-scale movements in membrane proteins

C.M. Casadei[1], K. Nass[1], A. Barty[2], M. Hunter[3], X. Li[1], C. Padeste[1], M. Coleman[3], M. Frank[3], B.F. Pedrini[1]

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Serial diffraction images can be recorded from radiation-sensitive membrane protein two-dimensional (2D) crystals using ultra-short and ultra-bright free electron laser X-ray pulses focused to the sub-μm and a low background environment. The interest in this exotic and demanding data collection mode resides in that membrane proteins arranged periodically in a monolayer maintain their physiological dynamics.

A dedicated processing pipeline was developed for the analysis of serial femtosecond crystallography (SFX) data from 2D crystals. 2D-SFX data present common features with well established methods, in particular serial crystallography from three-dimensional crystals and 2D electron diffraction. Yet there are intrinsic differences with each of these techniques, requiring the development of customized code. On one hand, unlike diffraction intensities from 3D crystals, 2D-SFX intensities are continuous in the out-of-plane direction of reciprocal space. On the other hand, the need of merging techniques that account for indexing ambiguity in serial images complicates the analysis with respect to single-crystal methods [1]. Our processing method deals with such peculiarities and includes an algorithm that allows to extend the resolution limit of the usable data by improving the signal to noise ratio of the measured intensities, which is inherently poor due to the weak scattering power of monolayers [2].

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Abstract submitted for contribution to the User-dedicated Microsymposium 3 (UDM3):

Towards a protein-based crystal host system

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X-ray crystallography is a powerful tool in structural biology as most protein structures today have been determined using this method. With the aim to allow structural determination from X-ray diffraction also of proteins excluded from conventional crystallography - as they do not crystallize - we recently started to develop a crystal host (HOSTAL) method. This approach is similar to the crystal sponge methods for structure determination of small molecules [1] but purely protein based using protein crystal with large large solvent channels into which small to mid-size guest proteins can be incorporated via soaking (Figure 1).

With help of confocal microscopy we could show that small guest proteins such as fluorophore-labeled calmodulin can enter the solvent channels of a host crystal made of domain swapped TrpR protein [2] and occupy ~40 % of the host's solvent channels (manuscript in review). The analysis of the X-ray diffraction data indicates differences in the solvent channel electron density for the host with guest compared to the host alone. However, the guest structures could not yet be solved by conventional methods likely due insufficient crystallographic order of the guest. Present work makes use of different ways to order the guest proteins e.g. by promoting specific host-guest interactions. Aside single crystal also serial crystallography approaches are followed to enable the use of microcrystals to lower the guest diffusion times into the host. For the better interpretation of weak signals from diffraction of this semi-ordered crystal system we are furthermore trying to make use of the diffuse scattering signal as described in [3] to solve the guest structure.

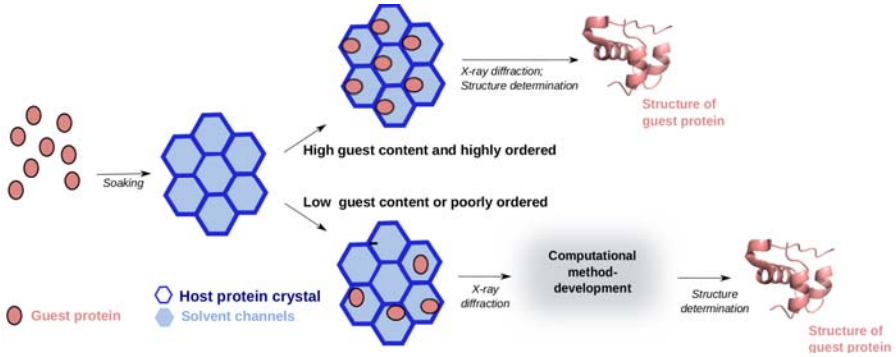

Figure 1: Schematic representation of the crystal host (HOSTAL) system

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Abstract submitted for contribution to the User-dedicated Microsymposium 3 (UDM3):

An automated and universal approach for high-throughput serial crystallography of membrane proteins using *CrystalDirect* technology

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Serial synchrotron crystallography (SSX) is considered as an attractive approach to determine structures of challenging membrane proteins, *especially*, when crystals are grown in Lipid Cubic Phase (LCP) [1]. However, transferring crystals from standard crystallization media to sample-delivery systems, compatible with SSX experiments can be difficult and often require reformulation of crystallization protocols. In addition to this, SSX typically consumes huge amounts of sample. In order to streamline sample delivery, reduce sample consumption and enable full automation of SSX experiments, we have developed a universally applicable approach from crystallization to structure determination – *CrystalDirect* for rapid SSX experiments on membrane proteins. We demonstrate that this technology [2] can be used for automated crystallization of two human transmembrane enzymes [4, 5] in Lipid Cubic Phase (LCP), which is otherwise an extremely tedious process [3]. Moreover, it can be used to streamline the preparation of samples for SSX experiments, either through automated crystal harvesting and cryo-cooling or by direct analysis through *in situ* diffraction experiments in CrystalDirect plates. This approach requires under 1 μg of sample and can be applied both to obtain cryogenic as well as room temperature structures in less than 20 mins. In this talk, various examples will be presented and the importance of full automation in SSX experiments will be discussed. The versatility and automation provided by the *CrystalDirect* technology, which can be easily adopted at any modern synchrotron, is expected to broaden SSX applicability by eliminating the need for complex experimental steps an increasing the throughput of SSX beamlines.

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Abstract submitted for contribution to the User-dedicated Microsymposium 3 (UDM3):

Serial crystallography at the SPB-SFX instrument at the European XFEL

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The Single Particles, Cluster and Biomolecules and Serial Femtosecond Crystallography (SPB/SFX) instrument [1] is optimised for biological structure determination benefitting from the unique properties and capabilities of the European X-Ray Free-Electron Laser. Serial crystallography is used predominantly with high speed jets greater than 30 m/s to enable data collection at the matching MHz rate [2] provided by the XFEL. A wide variety of GDVN nozzles are required to cover the different needs of each experiment we operate with glass capillary, ceramic and 3D printed nozzles which allow for double flow focusing as well as mix and inject enabling time resolved studies of chemical reactions. In addition to this we have just commissioned an additional instrument operating at atmospheric pressure which can be of benefit for the jets. It also enables a new simplified viscous injector setup as well as fixed target data collection [3] enabling serial data collection with low sample consumption. This range of delivery methods will be further extended with the ongoing developments to improve capabilities and reduce sample consumption such as drop-on-demand using acoustic droplet injection and drop in oil delivery methods.

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40	M.A. Andreeva, R.A. Baulin, M.M. Borisov, E.A. Gan`shina1, G.V. Kurlyandskaya, E.Kh Mukhamedzhanov, Yu.L. Repchenko and A.V. Svalov	X-ray magnetic dichroism of GdCo film studied by reflectivity with polarization selection
41	Marius Retegan	Crispy: a modern user interface for core-level spectroscopy calculations
42	K.S. Schulze, B. Grabiger, R. Lötzsch, B. Marx-Glowna, A.T. Schmitt, I. Uschmann, T. Stöhlker, R. Röhlsberger, G.G. Paulus	High precision X-ray polarimetry at extreme brilliant sources

43	Mustafacan Kutsal, Grethe Winthers, Carsten Detlefs, Henning Friis Poulsen	3DXRD microscopy with enhanced spatial resolution
44	C. Mansour , M. Benwadih, C. Revenant, G. A. Chahine.	Sol-Gel Barium Strontium Titanate thin films studied by GISAXS
45	A. Martini, E. Borfecchia, A.V. Soldatov, S. Bordiga	Wavelet Analysis of in situ and operando EXAFS data: a qualitative and quantitative approach.
46	A. Rack	The refurbished ID19 beamline: a versatile station for synchrotron-based full-field hard X-ray microimaging
47	Samuel K. Regli, Endre Fenes, Hongfei Ma, Kumar R. Rout, Terje Fuglerud, De Chen, Magnus Rønning	Elucidating the copper species in alumina supported CuCl2-catalysts during oxychlorination of ethylene to 1,2-dichloroethane by operando XAFS, PXRD and UV-Vis using MCR-AR analysis
48	A.A. Skorynina, A.L Bugaev, A. Lazzarini, L.G. Kamyshova, U. Olsbye, E. Borfecchia, K.A. Lomachenko, A. Guda, S. Bordiga, C. Ahoba-Sam, A.V. Soldatov	Structural characterization of Pd- and Pt-functionalized MOFs during in situ and operando experiments
49	O.A. Usoltsev, A.L. Bugaev, A.A. Guda, S.A. Guda, A.V. Soldatov	Extraction of structural parameters for XANES analysis of Palladium nanoparticles
50	M.V. Zakharkin, O.A. Drozhzhin, I.V. Tereschenko, D. Chernyshov, A.M. Abakumov, E.V. Antipov, K.J. Stevenson	High Voltage Activation of the NASICON-Type Na4MnV(PO4)3 Cathode studied by operando X-ray diffraction
52	C. Casadei, K. Nass, A. Barty, M. Hunter, C. Padeste, D. Ozerov, M. Colemann, X. Li, M. Frank, B. Pedrini	From two dimensional crystal serial diffraction to a three dimensional intensity set: paving the way to the time-resolved study of large scale movements in membrane proteins.
53	Hadrien Depernet, Guillaume Gotthard, Nathan C. Shaner, Xiaokun Shu, Gerard G. Lambert, Sylvain Aumonier, Gordon Leonard, Antoine Royant	Structural characterisation of near infrared and GFP-like fluorescent proteins
54	David Fernandez-Martinez, Eaazhisai Kandiah, Magali Mathieu, Gordon Leonard	Structural studies of multispecific Antibody/Antigen complexes by cryo-EM

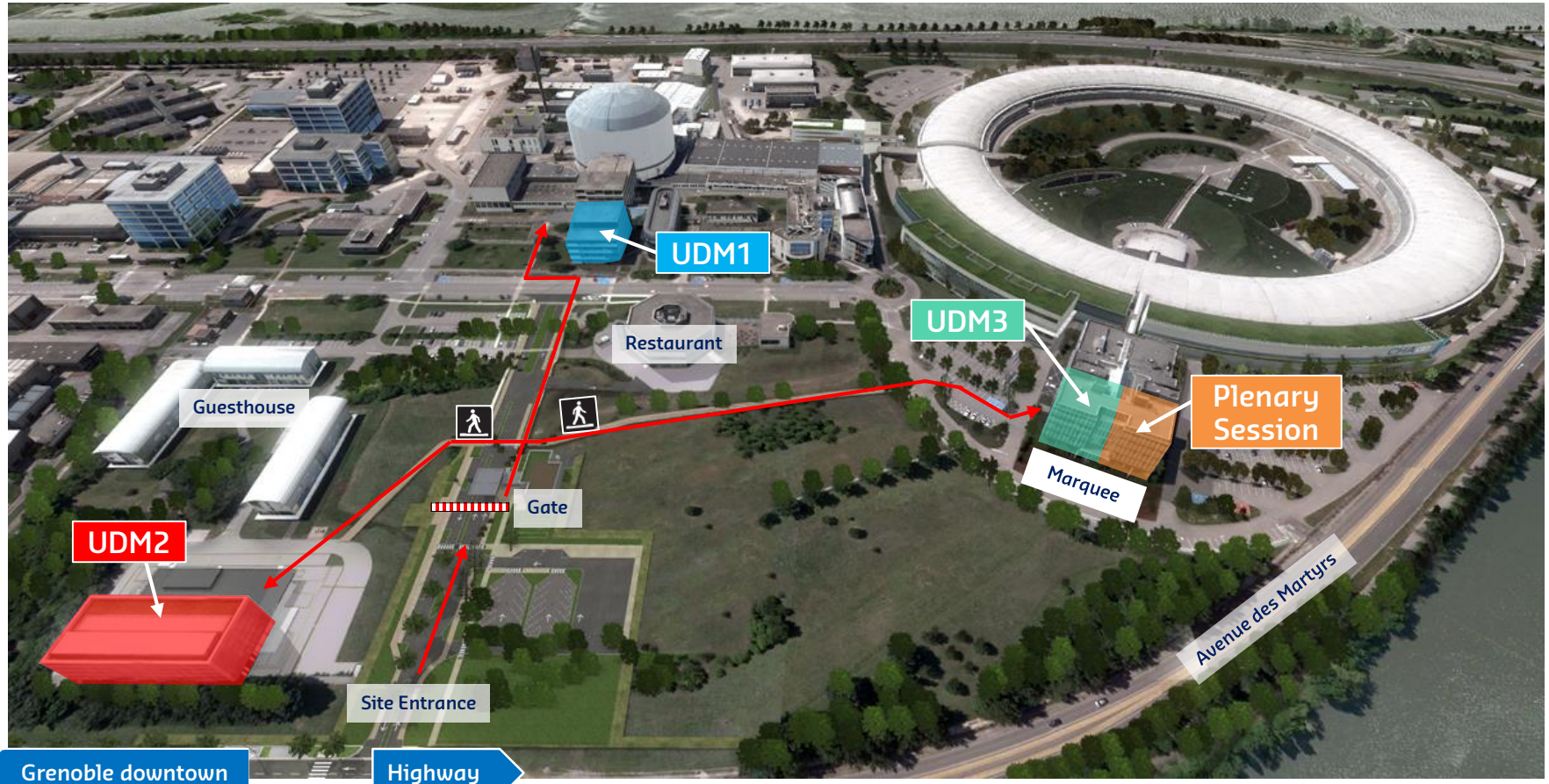
The name of the author submitting the poster is underlined

Poster Number	Authors	Poster Title
55	Giulia Salzano, Martha Brennich, Giordano Mancini, Thanh Hoa Tran, Giuseppe Legname, Paola D'Angelo, Gabriele Giachin	Deciphering copper coordination in the mammalian prion protein amyloidogenic domain
56	Alessandro Grinzato, E. Kandiah, C. Lico, C. Betti, S. Baschieri, G. Zanotti	Cryo-EM atomic structure of Potato Virus X
57	Shibom Basu, Robert Heale, Anne-Sophie Humm, Florine Dupeux, Cédric Leyrat, Andrea Pica, Andrew McCarthy, Sébastien Granier, José A. Márquez	An automated and universal approach for high-throughput serial crystallography of membrane proteins based on the CrystalDirect technology
58	V. Kovalenko, N. Loiko, E. Tereshkin, K. Tereshkina , A. Popov, Y. Krupyanskii	X-ray data collection and structure solving of Dps protein by multiple-crystal macromolecular crystallography methods
59	Carles Bosch, Tobias Ackels, Alexandra Pacureanu, Christopher Peddie, Manuel Berning, Norman Rzepka, Marie-Christine Zdora, Malte Storm, Isabell Whiteley, Lucy Collinson, Troy Margrie and Andreas T Schaefer	Ultrastructure and function of a genetically-identified mouse glomerular column studied by correlative in vivo physiology, synchrotron X-ray tomography and volume electron microscopy
60	Ilaria Clemente, Claudia Bonechi, Maria Bacia-Verloop, Claudio Rossi,Sandra Ristori	Green nonlamellar lipid phases as nanovectors for biomolecule delivery
61	Cristian Fernandez-Palomo, Jennifer Fazzari, Marine Potez, David Haberthur, Verdiana Trapetti, Elke Brauer-Krisch, Michael Krisch, Herwig Requardt, Jean Laissue, James Hainfeld, Valentin Djonov	Synchrotron Microbeam Radiation Therapy and Gold nanoparticles: a combined preclinical treatment in a mouse melanoma model
62	Cristian Fernandez-Palomo, Verdiana Trappetti, Marine Potez, Elke Brauer-Krisch, Herwig Requardt, Michael Krisch, Valentin Djonov	Complete Melanoma Remission After Fractionated Microbeam Radiotherapy
71	Marie Capron, Nicolas Daval, Joseph Hespel, Peter van der Linden, Pierre Lloria, Zakari Mechta, Alain Panzarella, Yuri Gerelli, and Diego Pontoni	PSCM Support Labs for Users, Partners and Staff
72	M. Sztucki, T. Zinn, P. Boesecke, T. Narayanan	Time-Resolved Ultra-Small-Angle X-ray Scattering (TRUSAXS) beamline at ESRF-EBS

73	Verdiana Trappetti, Cristian Fernández-Palomo, Paolo Pellicioli, Michael Krisch, Elke Bräuer-Krisch, Jean A. Laissue, Herwig Requardt, Alberto Bravin, Thomas Marti, Valentin Djonov	Early molecular mechanisms after Synchrotron Microbeam Radiation Therapy in normal lung tissue
74	Thomas Zinn, Theyencheri Narayanan et al.	Time-Resolved Small-Angle X-ray Scattering: Monitoring the Structural Development
75	Y. Krupyanskii, N. Loiko, V. Kovalenko , A. Moiseenko , A.Popov, K. Tereshkina, O. Sokolova	DNA condensation in bacteria
76	Pedro M Matias, Sónia Zacarias, Adriana Temporão, Melisa del Barrio, Vincent Fourmond, Christophe Léger & Inês A C Pereira	Improving the O2 resistance of a [NiFeSe] hydrogenase
77	Roman V. Moryachkov, Galina S. Zamay, Polina V. Artyushenko, Irina A. Shchugoreva, Vladimir N. Zabluda, Alexey E. Sokolov, Ekaterina A. Moryachkova, and Anna S. Kichkailo	Structure analysis of the aptamer - cancer cells magnetic separation agent
78	E. Pechkova, C. Riekel, C. Nicolini	Langmuir-Blodgett nanofilms as a potential tool for protein structural studies.
79	Phan Gilles, Housseini-b-Issa Karim, Michèle Salem, Marie-Bernard Lascombe, and Broutin Isabelle.	Structure of the two-component system response regulator ParR from Pseudomonas aeruginosa.
80	Anton Popov, Peter van der Linden, Diego Pontoni, Gordon Leonard	Recent development in 3D printed microfluidic devices for structural biology applications

The name of the author submitting the poster is underlined

USER MEETING 2020 – Plenary Session & Microsymposia Venues



Grenoble downtown

Train station
Highway to Lyon
Shuttle from/to Lyon Airport
Shuttle from/to Geneva Airport
Tramway

Highway

To Chambéry / Geneva

Plenary Session	→	ESRF Auditorium
UDM1	In situ and operando X-ray absorption spectroscopy for the study of catalysts and functional materials →	ILL Chadwick Amphitheatre
UDM2	Nanomaterials life cycle: from nanoengineering to public health →	IBS Seminar Room
UDM3	Multi-crystal and serial data collection in Structural Biology →	ESRF Auditorium

ESRF USER MEETING 2020 OVERALL PROGRAMME

	Monday 3 February		Tuesday 4 February		Wednesday 5 February	
	TUTORIALS		PLENARY SESSION		USER-DEDICATED MICROSYMPOSIA	
Morning	**08.15 – 09.00**	**Registration ESRF Central Building Welcome coffee**	**08.15 – 09.00**	**Registration ESRF Central Building Welcome coffee**	**08.15 – 09.00**	**Registration At EVENT VENUE**
	09.00 - 10.30	TUTORIALS	09.00 – 09.05	Opening and Welcome (UOC)	09.00 - 10.30	Welcome Keynote Speaker & Invited User Talks
			09.05 – 09.50	**Lecture 1 - Stephen Cusack**		
			09.50 – 10.35	**Lecture 2 - Kirsten M. Ø. Jensen**		
	10.30 – 11.00	Coffee Break	10.35 – 11.00	Coffee Break	10.30 – 11.00	Coffee Break
	11.00 – 12.00	TUTORIALS	11.00 – 12.30	Facility report & EBS news	11.00 – 12.30	Keynote speaker & Invited User Talks
LUNCH	12.00 – 13.30	LUNCH	12.30 – 13.30	LUNCH	12.30 – 13.30	LUNCH
Afternoon	**13.00 – 14.00**	**Registration ESRF Central Building**	14.00 – 14.45	**Lecture 3 - Caterina Biscari**	14.00 – 16.00	Keynote Speaker & Invited User Talks
	14.00 – 16.00	TUTORIALS	14.45 – 15.30	**Lecture 4 - Vincent Favre Nicolin**		
			15.30 – 16.00	Poster Clips (1)		
	16.00 – 16.30	Coffee Break	16.00 – 16.20	Coffee Break	**16.00**	**End of Meeting**
	16.30 – 18.30	TUTORIALS	16.20 – 17.10	Young Scientist Award		
			17.10 – 17.40	**Tribute to Rosalind Franklin Elspeth Garman**		
			17.40 – 18.00	Poster Clips (2)		
			18.00 – 19.30	Poster session and cocktail		
	18.30 – 20.30	Buffet dinner in marquee	19.30 – 22.00	User Meeting Dinner onsite		

Common sessions, in ESRF Auditorium
User-Dedicated Microsymposia (UDM)
Selected tutorials (all day or half day or few hours)