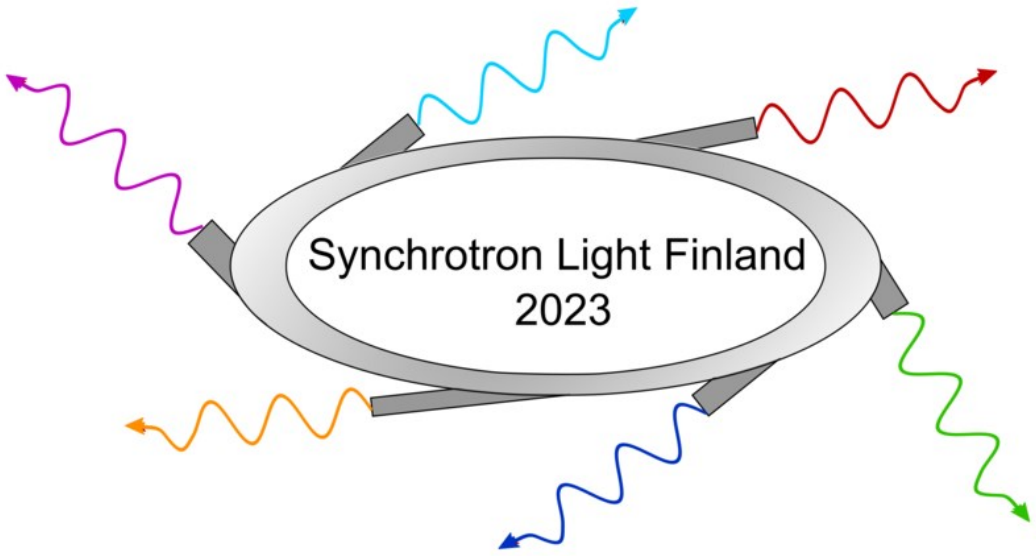
Abstracts of

Synchrotron Light Finland 2023

Aalto University

Aalto University, Finland

November 30–December 1, 2023



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Version November 27, 2023

Programme

Day 1, November 30 (Otakaari 1, Hall U147)

11:30-12:30 Registration

12:30-12:40 Opening

Session 1 (Chair: Paavo Penttilä, Aalto University)

12:40-13:20 K1 Marianne Liebi, PSI/EPFL Switzerland
Unraveling hierarchical structures with SAXS imaging in 2D and 3D

13:20-13:40 O1 Shuvashis Das Gupta, University of Oulu
Mineral crystal thickness in calcified cartilage and subchondral bone in healthy and osteoarthritic human knees

13:40-14:00 O2 Arturo Rubio Ruiz, Tampere University
In-situ characterisation of the failure mode and fragmentation pattern of granite and carbon fiber reinforced polymer by fast synchrotron X-ray phase-contrast imaging

14:00-14:30 Coffee break

Session 2 (Chair: Minna Patanen, University of Oulu)

14:30-14:50 O3 Ian Corfe, Geological Survey of Finland
Multiscale synchrotron tomography reveals the mid-Jurassic origins of mammalian growth patterns

14:50-15:10 O4 Ryan Trevorah, University of Helsinki
New insights on the molecular structure of tear film lipids revealed by surface X-ray scattering

15:10-15:30 O5 S. Assa Aravindh, University of Oulu
Combined experimental and theoretical investigations on the battery material $LiNiO_2$

15:30-15:35 Presentation by AMETEK Finland Oy

15:35-15:40 Closing

15:40-17:30 Poster session (16:00-17:00 FSRUO Annual Meeting)

18:00-22:00 Conference dinner (Factory Otaniemi, Otakaari 5)

Day 2, December 1 (Otakaari 1, Hall A123)

Session 3 (Chair: Paavo Penttilä, Aalto University)

9:20-10:00 K2 Tanja Kallio, Aalto University
Characterizing materials for electrochemical energy conversion applications

10:00-10:20 O6 Veera Langi, Tampere University
Use of synchrotron X-ray diffraction for investigations of mechanical response of multiphase steels at a wide range of strain rates

10:20-10:50 Coffee break

Session 4 (Chair: Ville Liljeström, Aalto University)

10:50-11:10 O7 Igor Prozheev, University of Helsinki
Study of local effect in Al-rich AlGaN with XAS

11:10-11:30 O8 Miguel A. Caro, Aalto University
Combining XPS and machine learning potentials for inferring the structure of complex materials

11:30-11:40 Closing

Posters and exhibition

P1 Mira Viljanen, Lund University:
Combined full-field tomography and SWAXS at ForMAX

P2 Enriqueta Noriega Benítez, Aalto University:
X-ray scattering analysis of wood aided by machine learning

P3 Aleksi Zitting, Aalto University:
Moisture imbibition in wood studied by time-resolved X-ray scattering

P4 Heikki Takala, University of Jyväskylä:
Phytochrome-based optogenetic modules

P5 Eemeli A. Eronen, University of Turku:
Statistical analysis of X-ray spectra of aqueous triglycine

P6 Szymon Stolarek, Xenocs SAS:
Extending measuring length (and time) scales of laboratory SAXS/WAXS beamlines

Exhibition table by AMETEK Finland Oy

Keynote presentation K1

Unraveling hierarchical structures with SAXS imaging in 2D and 3D

Marianne Liebi[1,2]

1. Paul Scherrer Institut, Photon Science Division, PSI Villigen, Switzerland
2. Ecole Polytechnique Fédérale de Lausanne (EPFL), Institute of Materials, Lausanne, Switzerland

Imaging with small-angle X-ray scattering (SAXS) contrast allows to probe nanoscale features in reciprocal space in macroscopic samples with the resolution given by the beam- and step size of scanning. This allows to bridge between length scales and is thus a powerful tool for life and material science samples with a hierarchical structure. As an example, the arrangement of mineralized collagen fibrils in the nanoscale can be studied over mm-cm of bone, providing a complementary tool to high resolution techniques, which often are limited in field of view. Combining scanning SAXS with computed tomography allows the study of three-dimensional samples.[1,2] In SAXS tensor tomography, additional tilt angles of the tomographic axis and extended algorithms are allowing to reconstruct also the orientation information contained in scattering. This results in a 3D reciprocal space map in each voxel, extending the method to 6 dimensions.

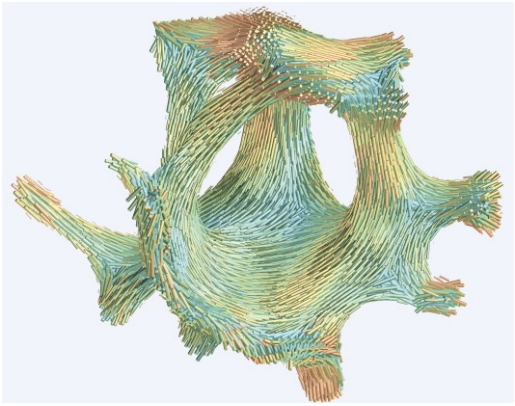

Fig. 1. SAXS tensor tomography on trabecular bone extracted from human vertebra showing the orientation of the mineralized collagen fibrils.

[1] Liebi, M., Georgiadis,M., Menzel,A., Schneider,P., Kohlbrecher,J., Bunk,O., Guizar-Sicairos, M (2015), *Nature*, 527, 349-352, DOI

[2] Liebi, M., Georgiadis, M., Kohlbrecher, J., Holler, M., Raabe, J., Usov, I., Menzel, A., Schneider, P., Bunk, O., Guizar-Sicairos, M. (2018), *Acta Cryst A*, 74, 12-24, DOI

[2] Nielsen, L.C., Erhart P., Guizar-Sicairos, M., Liebi, M. (2023), *Acta Cryst A*, accepted

Keynote presentation K2

Characterizing materials for electrochemical energy conversion applications

Lilian Moumaneix[1], Mattia Colalongo[1,2], Tanja Kallio[1]

1. Aalto University, Department of Chemistry and Materials Science, Espoo, Finland
2. European Synchrotron Radiation Facility, Grenoble, France

The ongoing green technology transition increases drastically consumption of mineral resources and hence for sustainability perspective requires development of material efficient energy technologies. Key technologies for this transition include electrochemical Power-to-X and lithium batteries, which enable evolution of fossil raw material free chemical industry, energy, and transport sectors.

To foster resource wise adoption of the aforementioned technologies, options based on alternative materials are investigated and developed. This requires understanding functioning and ageing of the alternative active materials and components made thereof using in-situ and operando characterization techniques. Those yield crucial information on evolution of the active material structure under relevant operation conditions – information which cannot be obtained in ex-situ studies of the pristine or aged materials.

Here, operando measurements for two case studies are discussed. In the first case study, palladium is utilized as an electrode material [1]. Palladium is one of the electrocatalyst used for emerging Power-to-X and X-to-Power technologies. It promotes CO_2 conversion to HCOOH and can serve as a hydrogen electrode material for alkaline fuel cells. One feature of Pd is its ability to form hydrates, PdH_x, when polarized towards negative electrochemical potentials where the CO_2 and H_2 reactions take place. In the operando XRD measurements, we have investigated the PdH_x phase evolution when Pd/C electrocatalyst based electrodes are polarized under different operation conditions.

In the other case study, an emerging lithium battery active material $LiNi_{0.8}Mn_{0.1}Co_{0.1}O_2$ is investigated [2]. When changing and discharging the battery, in the context of the electrochemical reactions, lithium is reversibly deintercalated and intercalated in the active material structure. This is accompanied with active material phase and volume changes. Their detailed understanding is crucial for developing approaches for enhancing the battery power performance and life span.

[1] Moumaneix, L., Viola, A., Chattot, R., Maillard, F., Kallio. T., Hydrogen and deuterium induced phase changes in Pd/C electrocatalysts: Operando study, submitted.

[2] Colalongo, M., et al., Investigation of Zr-doping in NMC811 cathode material for Li-ion batteries, submitted.

Oral presentation O1

Mineral crystal thickness in calcified cartilage and subchondral bone in healthy and osteoarthritic human knees

Shuvashis Das Gupta[1], Mikko Finnilä[1], Mikael J. Turunen[2], Iida Hellberg[1], Viviane Lutz-Bueno[3], Elin Folkesson[4], Mirko Holler[5], Neserin Ali[4], Velocity Hughes[4], Hanna Isaksson[6], Jon Tjörnstrand[7], Patrik Önnerfjord[8], Aleksandra Turkiewicz[4], Manuel Guizar-Sicairos[5], Martin Englund[4], Simo Saarakkala[1]

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2. Department of Applied Physics, University of Eastern Finland, Kuopio, Finland.
3. Laboratory of Food and Soft Materials Science, ETH Zürich; Zürich, Switzerland.
4. Clinical Epidemiology Unit, Lund University, Lund, Sweden.
5. Photon Science Division, Paul Scherrer Institut; Villigen PSI, Switzerland.
6. Department of Biomedical Engineering, Lund University, Lund, Sweden.
7. Department of Orthopaedics, Skåne University Hospital; Lund, Sweden.
8. Rheumatology and Molecular Skeletal Biology, Lund University; Lund, Sweden.

Osteoarthritis (OA) is the most common joint disease, often characterized by the degradation of articular cartilage alongside sclerosis of the subchondral bone. However, the association between OA and tissue mineralization at the nanostructural level is currently not understood. Notably, investigating calcified cartilage, where poorly understood pathological processes like tidemark multiplication and progression occur, presents substantial technical challenges. In this study, we used state-of-the-art micro-focus small-angle X-ray scattering with a 5μm spatial resolution to determine the size and organization of the mineral crystals at the nanostructural level in human subchondral bone and calcified cartilage. Moreover, we applied Raman microspectroscopy on adjacent sections to gain insights into the connection between mineral composition and mineral nanostructure. Specimens with a wide spectrum of OA severities were acquired from both medial and lateral compartments of medial compartment knee OA patients (n=15) and cadaver knees (n=10).

Opposing the common notion, we found that calcified cartilage has thicker and more mutually aligned mineral crystals than adjoining bone. In addition, we, for the first time, identified a well-defined layer of calcified cartilage associated with pathological tidemark multiplication, containing 0.32nm thicker crystals compared to the rest of calcified cartilage. Raman spectral measurement of mineral crystallinity index was directly associated with mineral crystal thickness. Finally, we found 0.2nm thicker mineral crystals in both tissues of the lateral compartment in OA compared with healthy knees, indicating a loading-related disease process since the lateral compartment is typically less loaded in medial compartment knee OA. In summary, we report novel changes in mineral crystal thickness during OA. Our data suggest that unloading in the knee might be involved with the growth of mineral crystals, which is especially evident in the calcified cartilage.

Oral presentation O2

In-situ characterisation of the failure mode and fragmentation pattern of granite and carbon fiber reinforced polymer by fast synchrotron X-ray phase-contrast imaging

Arturo Rubio Ruiz[1], Nazanin Pournoori[1], Mikko Kanerva[1], Mikko Hokka[1]

1. Tampere University, Engineering Materials Science, Tampere, Finland

The energy-intensive nature of rock drilling and comminution operations, and excessive tool wear, have driven the pursuit of non-conventional techniques to enhance rock breakage. An example of these methods involves the application of alternating current (AC) excitations to induce damage in quartz-bearing rock due to piezoelectric effects. However, the damage mechanisms triggered by this technique are not yet understood. The use of XPCI enables the visualisation of alterations in rock failure modes between intact and AC-exited rocks. Therefore, it is possible to correlate changes in fragmentation patterns, material stresses and deformations with potential damage mechanisms triggered by the AC excitations prior to loading. Carbon Fiber Reinforced Polymer (CFRP) composites have been used extensively in lightweight applications that require impact-resistant structural design. The inherent anisotropy of FRP composites, arising from the orientation of reinforcing fibres in a laminate, significantly affects the levels of strength, stiffness, and resistance to specific failure modes. Therefore, predicting failure modes of CFRP under impact loads is complex. It is crucial to comprehend the effects of lay-up configurations and strain rate on damage and failure modes to develop impact damage-tolerant structures. This presentation describes successful cases of the application of synchrotron X-ray phase-contrast imaging (XPCI) as a tool for characterising the failure and fragmentation processes of granite and CFRP. To investigate the rapid progression of damage in the studied materials, mechanical testing was integrated with ultra-fast synchrotron XPCI at the ID-19 beamline of the European Synchrotron Radiation Facility (ESRF). This technique was synchronised with a Split Hopkinson Pressure bar device, providing the required mechanical impact loading. Correlating compressive loads and displacements with in-situ images of the failure of the material allowed to discern the onset of damage and the resulting crack propagation patterns in both materials. Regarding the observations in granite, it was possible to observe differences in the failure and fragmentation process of AC exited and intact granite specimens. The correlation of such images with the mechanical history of the samples suggests that the AC excitations induced cracks in the rock, leading to rock weakening and reduced energy absorbed during failure. Regarding the observations in CFRP specimens, the compressed angle-ply laminates with special cut out in 45° directions failed due to combined normal and shear stresses and disintegrated into pieces by interlaminar crack growth. The XPCI images also indicated delamination and fracture at the interface of the 0° and ±45° angled layers inside the laminate. XPCI offers a helpful tool for observing the formation of internal damage in different materials under dynamic loading conditions. These observations serve as a qualitative reference for calibration and validation of numerical models to simulate damage evolution in granite and CFRP. Some examples of how the XPCI were used to validate finite element models used to simulate the fragmentation process of granite and CFRP are shown in this presentation.

Oral presentation O3

Multiscale synchrotron tomography reveals the mid-Jurassic origins of mammalian growth patterns

Elis Newham[1], Ian Corfe[2,3], Pam Gill[4], Thomas Martin[5]

1. Queen Mary University of London, School of Engineering, London, UK
2. Geological Survey of Finland, Research Laboratory, Espoo, Finland
3. University of Helsinki, Institute of Biotechnology, Helsinki, Finland
4. University of Bristol, Department of Geology, Bristol, UK
5. University of Bonn, Institute of Geology and Palaeontology, Bonn, Germany

The mid-Jurassic evolutionary expansion (adaptive radiation) of mammals has been linked to changes in physiology related to the evolution of mammalian endothermy (warm-bloodedness). These include increases in basal/maximum metabolic rate [1,2], and the evolution of rapid juvenile growth rates that are reduced at sexual maturity – the mammalian determinate growth pattern. To test these hypotheses, we used multiscale synchrotron X-ray tomographic phase contrast imaging to map the origin and evolution of metabolic rates and mammalian determinate growth. Annual growth increments in the mineralised jawbone and tooth root cementum tissues [3] were imaged at 5µm, 0.67µm, 0.33µm and 25nm voxel sizes on four beamlines at two synchrotron light sources. The sample consisted of almost 600 Early-to-Late Jurassic (~200 to 145 million years old) fossil mammals and their closest ancestors. Our previous estimation of basal metabolic rates using maximum lifespan suggested that Early Jurassic close mammal relatives had low/slow metabolisms comparable to living reptiles [1]; our new data shows some mid-Jurassic early mammals had metabolic rates approaching the lowest values seen in living mammals. Annual growth increment widths indicate that close mammal relatives had slow growth rates with relatively little change through life. The earliest mammals had significantly faster growth rates as juveniles, that were reduced at the attainment of sexual maturity. One group of mid-Jurassic fossil mammals are the earliest to show distinct contrasts in structure between juvenile and adult cementum, as do modern mammals [4]. All fossils analysed had lower maximum growth rates, longer maximum lifespans, lower basal metabolic rates, and delayed sexual maturity relative to comparably sized living mammals. Our results show that the mammalian determinate growth pattern first appeared amongst early mammals during their mid-Jurassic adaptive radiation, but suggest full modern mammalian endothermy evolved later.

[1] Newham, E… & Corfe, I.J. (2020). Reptile–like physiology in Early Jurassic stem–mammals. *Nature Communications*, 11, 5121, DOI: 10.1038/s41467-020-18898-4
[2] Newham, E., Gill, P. G. & Corfe, I. J. (2022). New tools suggest a middle Jurassic origin for mammalian endothermy. *BioEssays* 44, 2100060, DOI: 10.1002/bies.202100060
[3] Newham, E.,… Corfe, I. J., & Schneider, P. (2021). A robust, semi-automated approach for counting cementum increments imaged with synchrotron X-ray computed tomography. *PLoS One*, 16(11), e0249743, DOI: 10.1371/journal.pone.0249743
[4] Newham, E., Corfe, I. J. et al. (2020). Synchrotron radiation-based X-ray tomography reveals life history in primate cementum incrementation. *Journal of the Royal Society Interface*, 17(172), 20200538, DOI: 10.1098/rsif.2020.0538

Oral presentation O4

New Insights on the Molecular Structure of Tear Film Lipids Revealed by Surface X-ray Scattering

Ryan Trevorah[1], Mira Viljanen,[1] Tuomo Viitaja,[2,3] Henrik Stubb,[2] Julia Sevón,[2] Oleg Konovalov,[4] Maciej Jankowski,[4] Philippe Fontaine,[5] Arnaud Hemmerle,[5] Jan-Erik Raitanen,[2] Filip S. Ekholm,[2] and Kirsi J. Svedström*[,1]

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4. The European Synchrotron Radiation Facility – ESRF, 71 Avenue des Martyrs, CS 40220, Grenoble Cedex 9 38043, France
5. Synchrotron SOLEIL, L'Orme des Merisiers, Départementale 128, 91190, Saint-Aubin, France

The tear film lipid layer (TFLL) is a unique biological membrane which serves a pivotal role in the maintenance of ocular surface health. Reaching an overarching understanding of the functional principle of the TFLL has been hampered by lack of insights on the structural and functional roles played by individual lipid classes. In order to bridge this knowledge gap, we herein focus on studying films formed by principal lipid classes by surface scattering methods. Through Grazing Incidence X-ray Diffraction (GIXD) and X-Ray Reflectivity (XRR) studies we reveal quantitative data on the lattice distances, molecular tilt angles and mono/multilayer thickness and density profiles for central TFLL lipid classes close to simulated physiological conditions. In addition, we discuss the correlation of the results to those earlier obtained with the natural lipid composition of meibum.

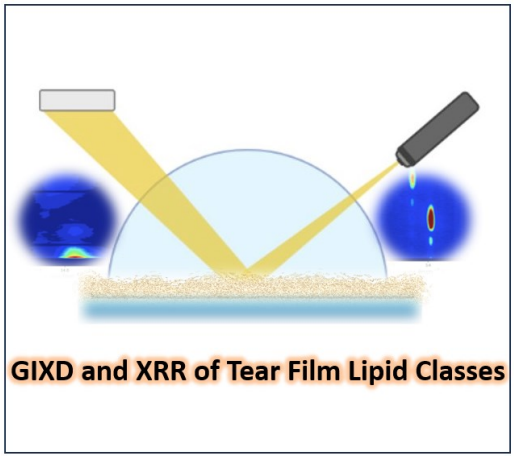

Fig. 1. Artistic impression of x-ray scattering experimental setup, with representative spectra shown in blue.

Oral presentation O5

Combined experimental and theoretical investigations on the battery material $LiNiO_2$

Presenting author: S. Assa Aravindh, Nano and molecular systems research unit, University of Oulu, Finland.

Email: Assa.Sasikaladevi@oulu.fi

A combined experimental and density functional theory based investigation is carried out on the well known battery material, $LiNiO_2$, to reveal the structure-property relationships. We show that using density functional theory simulations, optical energy loss function can yield insight into the results of Resonant Inelastic X-ray Scattering (RIXS) experiments on $LiNiO_2$ cathode material [1]. We demonstrate that strong electronic correlations in this material demand higher level approximations beyond standard generalized gradient approximation to accurately predict the correct electronic structure. Further, the redox orbitals of $LiNiO_2$ can be obtained from spherical Compton profiles using parallel X-ray Compton scattering experiments and first principles simulations. The nature of the hole states in Li-doped NiO is revealed from these analyses, that indicate the hybridization of O 2*p* and Ni 3*d* orbitals [2]. Our studies also reveal that first principles simulations can provide useful insight into the ferromagnetic ground state and create a pathway toward rational design of next-generation battery materials.

These studies are carried out as a collaboration of University of Oulu, LUT - Lappeenranta, Helsinki University, Northeastern University - Boston, University of Jyvaskyla, Gunma University – Japan and Japan Synchrotron Radiation Research Institute.

Reference:

1. First Principles Calculations of the Optical Response of LiNiO2. Veenavee et al. Condens. Matter **2022**, 7(4), 54; **https://doi.org/10.3390/condmat7040054**

2. Compton scattering study of strong orbital delocalization in LiNiO2 cathode, Veenavee et al, (under review)

Oral presentation O6

Use of synchrotron X-ray diffraction for investigations of mechanical response of multiphase steels at a wide range of strain rates

Veera Langi[1], Lalit Pun[1], Matti Isakov[1], Mikko Hokka[1]

1. Tampere University, Engineering Materials Science, Tampere, Finland

The automotive sector is increasingly adopting the use of multiphase steels in vehicle body structures as a way to reduce carbon emissions. These steels contain a softer metastable austenite phase that transforms gradually to harder α'-martensite during plastic deformation. This strain-induced martensitic phase transformation improves the mechanical properties such as strength, ductility, and formability of steels. However, during fast deformation, the temperature of the material will increase due to adiabatic heating. The increased temperature in the material will cause the phase transformation to suppress considerably at high deformation rates, for example, during car crashes. Moreover, preferred orientation of austenite grains and microstructural level stress/strain triaxialities is affected by the strain rate, which significantly alter the stability of the austenite phase.

Unfortunately, the means to study such complex microstructural level phenomena in situ during mechanical loading over a wide strain rate range are very limited. The effects of strain rate on the strain-induced martensitic phase transformation are commonly investigated using ex-situ characterization techniques such as EBSD, TEM, optical microscopy, etc. In such studies, the loadings are paused and for that reason, they do not represent the same thermal history as that of continuously deforming specimen. Therefore, novel characterization technique to investigate the mechanical behavior at different loading rates was designed together with the scientists of the DanMax beamline of the MAX IV laboratory in Lund, Sweden.

This work presents and discusses the use of high-energy synchrotron X-ray diffraction for the characterization of bulk microstructural changes at a wide range of strain rates from 10-3 s-1 to 1 s-1. Three different steels were studied in this work, including fully austenitic stainless steel and two industrially produced multiphase steels with different austenite morphologies.

Oral presentation O7

Study of local effect in Al-rich AlGaN with XAS

Igor Prozheev[1], Frank Mehnke[2], Marcel Schilling[2], Tim Wernicke[2], Michael Kneissl[2], René Bès[1], Ilja Makkonen[1], and Filip Tuomisto[1]

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[2]Technische Universität Berlin, Institute of Solid State Physics, Hardenbergstr. 36, D-10623 BERLIN, GERMANY

We study defects in Si-doped AlGaN layers with 90% Al content and their effect on n-type conductivity.[1, 2] In one part of the research, we study the effect of local environments of Si atoms with help of x-ray absorption spectroscopy (XAS) data and corresponding simulations. We further observe that Si donors reside in different immediate local environments, which depends on the doping levels. We can identify that the surroundings of Si atoms change from GaN-like matrices to AlN-like at 10^{19} cm^{-3}.

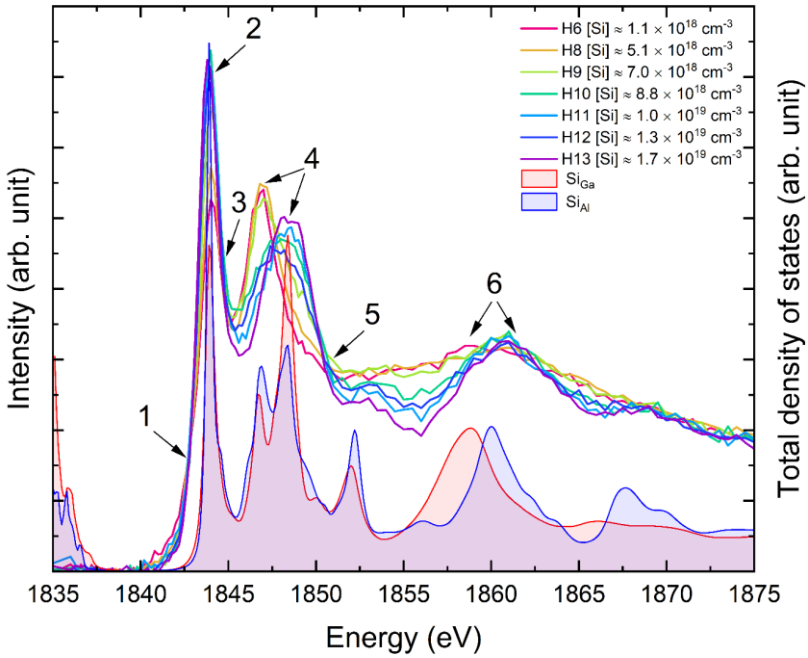

Fig. 1. Intensity of XANES versus the x-ray energy measured at Si k-edge in MOVPE AlGaN:Si samples shown by curves. Numbers 1-6 indicate characteristic isosbestic points and fingerprints. And theoretical unconvoluted XANES spectra at Si K-edge in GaN:Si and AlN:Si. Each spectrum represents total electron density of states of the system with 0.1 eV energy step.

[1] F. Tuomisto, and I. Makkonen, (2013) *Rev Mod Phys*, **85**(4), 1583.

[2] I. Prozheev, *et al., (2020) App Phys Lett*, **117**, 142103.

Oral presentation O8

Combining XPS and machine learning potentials for inferring the structure of complex materials

Tigany Zarrouk,[1] Rina Ibragimova,[1] Albert P. Bartók,[2,3] and Miguel A. Caro[1]

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2. Department of Physics, University of Warwick, Coventry, United Kingdom
3. Warwick Centre for Predictive Modelling, School of Engineering, University of Warwick, Coventry, United Kingdom

The interpretation of X-ray photoelectron spectroscopy data for complex (e.g., amorphous) materials is severely hindered by at least two facts. On the one hand, more than one chemical/structural motif may contribute to the same region of the spectrum because the corresponding core-electron binding energies (CEBEs) are the same. This means that the peak or spectral feature cannot be unambiguously deconvoluted. On the other hand, the set of reference CEBEs used to fit the experimental spectra may shift in the presence of new chemical motifs. For instance, pure carbon reference peaks (sp^2, sp^3, etc.) may shift towards higher energies in the presence of oxygen because of the modified electrostatic potential, even when the oxygen atom is not chemically bonded to the carbon atom in question.

Because of these difficulties, interpretation of XPS spectra and atomistic structure inference from them have long been the object of atomistic modeling, with mixed successes. The most common approach is to generate computational structural models of a material, derive a computational XPS spectrum from them, and then compare to experiment. This approach relies on 1) the availability and cost-effectiveness of a quantitatively accurate computational method to predict the XPS spectra (or, more usually, the CEBEs) and 2) sensible (i.e., "realistic") model structures, which often require a prior assumption of what the material might look like. Thus, this is not a fully predictive and "hands-off" approach.

We propose a new route to resolving the structure of materials from experimental XPS spectra, which can be generalized to include other spectroscopies and experimental techniques. Our method takes advantage of atomistic machine learning (ML) tools, which are driving a paradigm shift in computational materials modeling [1]. We combine ML interatomic potentials to provide a sensible description of the interactions between atoms, producing low-energy ("sound") structures, with a quantitatively accurate ML model for core-electron binding energy prediction [2] which is computationally efficient enough to be evaluated on the fly during a molecular dynamics or Monte Carlo simulation. With these tools, we can build hybrid simulation protocols which generalize structure optimization/prediction to take experimental input directly into account [3].

[1] V.L. Deringer, M.A. Caro and G. Csányi. Adv. Mater. 31, 1902765 (2019).

[2] D. Golze, M. Hirvensalo, P. Hernández-León, A. Aarva, J. Etula, T. Susi, P. Rinke, T. Laurila, and M.A. Caro. Chem. Mater. 34, 6240 (2022).

[3] T. Zarrouk, R. Ibragimova, A.P. Bartók, and M.A. Caro. *Manuscript in preparation*.

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Luca Pini, Amptek - Ametek Finland

PLEIADES XRA-700 Detector Array

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Poster presentation P1

Combined full-field tomography and SWAXS at ForMAX

Mira Viljanen[1], Samuel Mcdonald[1], Vahid Haghighat[1], Kim Nygård[1]

1. MAX IV laboratory, Lund University, Lund, Sweden.

With the increasing demand of sustainable products, forest-sourced materials are essential for answering the needs of society and economics worldwide. To develop and manufacture biodegradable, long-lasting products with desired properties, understanding the structure on micro-, nano- and atomistic scales is crucial. Wood is a natural composite with extraordinary mechanical characteristics arising from the hierarchical architecture of the cellular structures and from the ultrastructural features of cellulose microfibrils. The crystalline properties and orientation of the cellulose microfibrils are important when considering the suitability of wood and other plant fibres for various applications [1,2].

Synchrotron-generated X-ray tomography and scattering techniques are an exceptional tool to generate multiscale structural information of wood and other hierarchical materials. At ForMAX beamline, at MAX IV Laboratory, the combination of full-field microtomography imaging (μCT) and small- and wide-angle X-ray scattering (SWAXS) can be used to characterize materials and their processing in time-resolved manner.

In this project we studied young aspen saplings using a combination of μCT-imaging and SWAXS to characterize the nanoscale properties of the cellulose microfibrils in selected microscale structures to highlight the unique capabilities of the ForMAX beamline.

[1] Jakob, M, et al (2022), *Progress in Materials Science*, *125*, 100916, https://doi.org/10.1016/j.pmatsci.2021.100916

[2] Eichhorn, SJ, et al (2022), *Journal of Materials Science*, *57*(10), 5697-5767.

Poster presentation P2

X-ray Scattering Analysis of Wood Aided by Machine Learning

Enriqueta Noriega Benítez[1], Mikko Mäkelä[2], Paavo Penttilä[1]

1. Aalto University, Department of Bioproducts and Biosystems, Espoo, Finland
2. VTT Technical Research Centre of Finland Ltd., Biomass Processing and Products, Espoo, Finland

Small and wide-angle x-ray and neutron scattering on wood and other (ligno)cellulosic materials can be applied to improve our understanding of their structure [1]. The development of methods for characterization is important for the understanding of the anatomical composition and nanoscale structure of biobased materials— this is fundamental to their utilization for sustainable applications.

Wood scanning with wide-angle and small-angle X-ray scattering (WAXS, SAXS) was done at the ID02 beamline of the ESRF synchrotron. The beam size was of about 30x30 μm² to observe wet and dry Norway spruce samples. The measurements were done at an energy of 12.23 keV, an exposure time of 0.2 s each, and a sample to detector distance of 1.4 m for the SAXS and 14 cm for the WAXS. Detector images were processed and fitted to obtain structural parameters such as the distance between microfibrils [2].

The results of the fitting from dry and wet samples were analyzed with principal component analysis (PCA) algorithms to depict the statistical information related to water content and wood structure. A clustering algorithm was able to distinct types of wood tissue (earlywood/latewood) and samples (wet/dry) based on the PCA output. Experiments of a different nature, such as near infrared (NIR) spectroscopic imaging, can also benefit from PCA and validate the methodology's findings [3]. Results highlight the sample and tissue type classifications on different experiments featuring different characteristics of the same wood sample but holding comparable classes based on the PCs. The outlook moves towards new automated procedures that will lead to more sophisticated supervised learning algorithms.

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Poster presentation P3

Moisture imbibition in wood studied by time-resolved X-ray scattering

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Wood is a commonly used material that has most of its properties change based on its moisture content. This makes studying wood-water interactions important. X-ray measurements provide a practical way to measure changes in the wood structure while it interacts with water. While modern lab sources can be sufficient for several different X-ray measurements of wood-moisture interactions, synchrotrons are still required when the timescale is short. One example of this is how dry wood and the fibres within absorb liquid water and how the nanoscale structure changes while the moisture travels across the wood sample.

We have measured the moisture imbibition of several different wood samples using the ForMAX beamline at MAX IV Laboratory. The samples consisted of spruce heartwood, spruce sapwood and birch sapwood dried using different methods (oven-, room-, and freeze-drying). Additionally, the effect of the fibre direction on the water transport was investigated by exposing the sample to water in a way where the water can either travel along or across the main fibre direction. Both small-angle and wide-angle X-ray scattering was measured simultaneously.

In order to measure the samples, we created a sample environment where the wood could be exposed to water through a small, exposed volume while simultaneously measuring X-ray scattering as soon as the wood begins to imbibe moisture. This allowed us to measure with good temporal (~1 s) and spatial resolutions (<1 mm), the changes in the wood structure as water is transported across the sample.

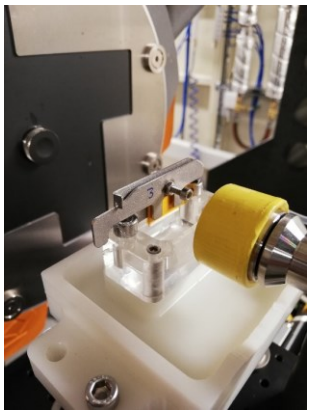

Fig. 1. Sample stage installed at the beamline. During the measurement, water will fill the container and be absorbed through the exposed bottom of the wood sample.

Poster presentation P4

Phytochrome-based optogenetic modules

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The research of our group focuses on photoreceptor studies and their application as optogenetic tools. Especially, we are interested in the function and applicability of red light-sensing bacterial phytochromes. Our research lies at the interface of structural biology, biochemistry, biotechnology, and cell biology, with a special focus on understanding and engineering optogenetic proteins that enable precise control of cellular processes with red light.

We have previously uncovered the full-length structure and biochemical function of a model bacterial phytochrome [1,2]. We have then utilized this knowledge by engineering optogenetic pREDusk tools [3], which enable the control of bacterial gene expression with red light. With these tools one can, for example, make cells produce a protein of interest and tune the production level by illuminating them with red light. Currently we are planning to extend these optogenetic tools to generate (cyano)bacterial cell factories, as well as engineer new phytochrome-based optogenetic tools for mammalian cells.

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Poster presentation P5

Statistical Analysis of X-ray Spectra of Aqueous Triglycine

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The folding of proteins is a complex and complicated question [1], affected by both intramolecular and intermolecular interactions. The process is ultimately dependent on the primary order of the amino acids and the surrounding solvent. While diffraction allows for atomistic structure determination of proteins, it requires a crystalline sample. Instead, owing to its localized mechanism, X-ray spectroscopy maintains its sensitivity to atomistic structure also in the liquid phase.

A liquid system allows for the movement of relatively strongly interacting molecules, which leads to a broad distribution of possible configurations. These individual configurations have been computationally observed to have significantly different X-ray spectra, only their ensemble mean predicting the experimentally observed spectrum. Although X-ray spectroscopy does not permit full structural reconstruction, its principles allow for discovery of structural information, especially when combined with statistical analysis [2-4] of computational results.

In this contribution, we present our ongoing statistical analysis of computational X-ray spectra of aqueous triglycine. Classical molecular dynamics was used to obtain the configuration distribution of the system. Then, X-ray absorption spectra were calculated with density functional theory. The main goal of our study [4] is to determine if X-ray spectroscopy could be used to gain new structural information of aqueous tripeptides. As the systems are complex, simple tools like linear correlations between the internal coordinates of the system and its X-ray absorption spectrum do not deliver satisfying results. We approach this problem by applying neural networks and a specialized dimensionality reduction method, as already done with a different system [3]. The results are compared to X-ray Raman scattering spectra of liquid triglycine at the nitrogen K edge.

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Poster presentation P6 (sponsor)

Extending measuring length (and time) scales of laboratory SAXS/WAXS beamlines

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State of the art laboratory SAXS/WAXS instruments used for soft matter or nanomaterials structural characterization are typically made of high brightness microfocus sources coupled to in-vacuum hybrid pixel detectors for low noise and high dynamic range detection. The achievable characteristic length scales are typically from few hundred nanometers down to atomic scale ($2\theta \geq 60°$) using standard Copper radiation source ideal for characterization of polymers, nanoparticles, colloids surfactants, proteins or lipid-based systems. Motorized change of sample to detector distance and multi-radiation sources have been introduced respectively for maximum automation, flexibility, or improved measuring performances for inorganic samples. More recently, advanced measuring options have been introduced to extend the measuring length scales up to more than 5 orders of magnitude using Bonse-Hart USAXS attachment for characterization of micron-sized particles.

We will present application cases illustrating the complementarity of laboratory SAXS beamlines with synchrotron sources. This includes in-situ characterization of block-copolymer nano-object formation during polymerization-induced self-assembly (PISA) reaction with time resolution of few minutes[1]. Another presented example is related to in-situ characterization of wood wetting using laboratory SAXS/WAXS, which was used as preliminary characterization step prior to further synchrotron experiments. Finally, we will describe how multimodal X-ray imaging can be combined with a laboratory SAXS beamline to achieve absorption, phase contrast and dark field imaging together with X-ray scattering measurements[2,3].

1 Continuous-Flow Laboratory SAXS for In Situ Determination of the Impact of Hydrophilic Block Length on Spherical Nano-Object Formation during Polymerization-Induced Self-Assembly, Macromolecules 2023, 56, 16, 6426–6435

2 Xeuss 3.0 laboratory beamline with InXight module https://www.xenocs.com/inxight-x-ray-imaging-module/

3 X-ray multi-modal intrinsic-speckle-tracking, Journal of optics 2020, Vol 22, Number 12