HIGHLIGHTS 2022

Partners

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if not otherwise stated, Kenneth Ruona, Johan Persson, Emelie Hilner

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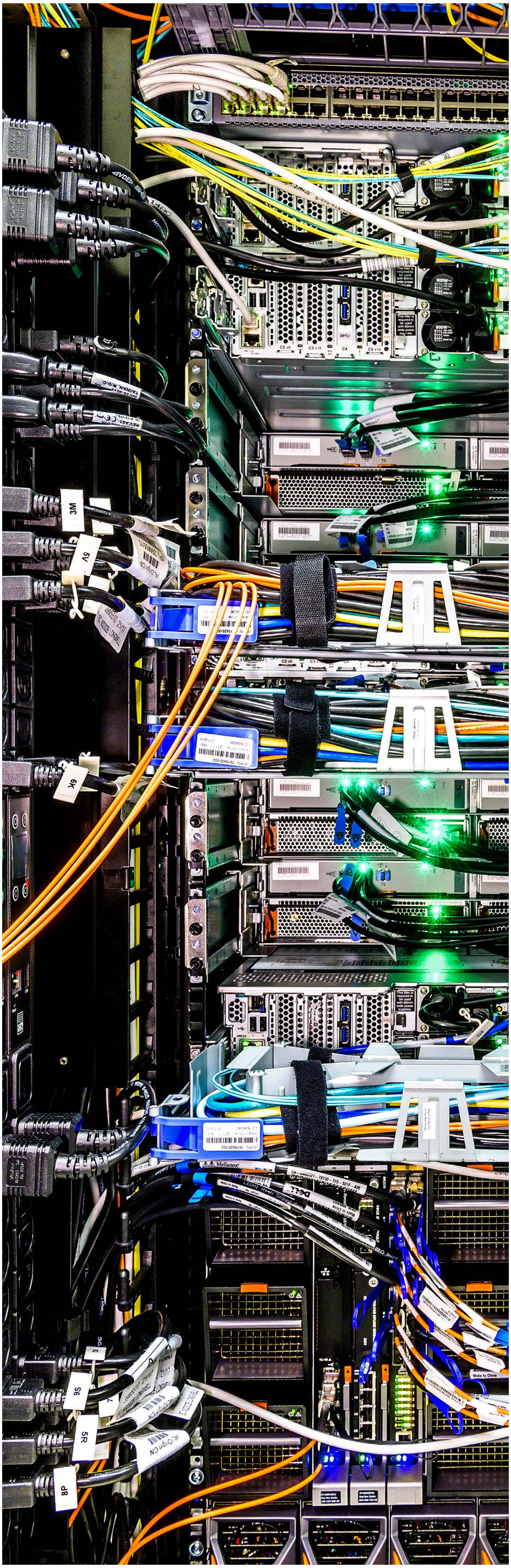


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Foreword

It is with great pleasure that we present our 2022 Science Highlights, illustrating our research achievements of the past year, while we especially celebrate an important milestone: all 16 beamlines at MAX IV have received X-rays!

In addition to our portfolio of 14 beamlines open to general users, we approached the completion of our last two funded beamlines:

ForMAX, funded by the Knut & Alice Wallenberg Foundation and Swedish industry via the Treesearch consortium, provides a research platform for new materials and chemicals from forest raw materials. It performed its first commissioning experiments in November 2022.

MicroMAX, the Novo Nordisk Foundation funded beamline, will provide novel research opportunities. MicroMAX will offer unprecedented possibilities to investigate the structure of molecules that are interesting but quite difficult to study because they only form microcrystals. The beamline will also enable users to perform time-resolved protein studies down to milli- and microsecond timescales. A first protein crystal data set was collected in December 2022.

The year 2022 also witnessed two additions to the laboratory's Senior Management team. We welcomed Professor Olof "Charlie" Karis as Interim Director and Anna Hultin-Stigenberg as the Technical Division Director. Their expertise and leadership are excellent assets to continue developing the laboratory as a world-leading user facility. Furthermore, the approval of our Strategic Plan for 2023-2032 by the MAX IV Board marked another milestone in our journey. This document, available on our website, stands as a testament to the collaborative efforts of our numerous stakeholders, whose invaluable input and ongoing dialogue shape our laboratory's future trajectory.

MAX IV continues to support the ever-growing needs of our user communities, as indicated by the 21% increase in our publications discussing results which involve measurements at our beamlines compared to 2021. A record 315 proposals were accepted from 655 submitted, thanks to the increasing number of our beamlines welcoming users. Overall, in 2022, MAX IV supported the research needs of approximately 1400 users from 140 institutions across 32 countries. Our user base is principally located in Sweden (47%) and retains a solid northern Europe component (i.e., Sweden, Denmark, Norway, Finland, Iceland, and the Baltic countries with 71%). We also saw a growing number of requests from industry, with a record 33 companies that purchased beamtime for proprietary use.

In October 2022, our 34th User Meeting took place, bringing together over 200 synchrotron users, researchers, industry guests, and students. We decided to adjust the timing of our next User Meeting to maximise the possibility of many of you attending. It will take place January 15-17, 2024. Mark your calendars! We look forward to meeting all of you at this important event.

We launched PRISMAS – an initiative in partnership with 9 Swedish universities and 43 organizations funded by the European Union’s COFUND programme. This new graduate student programme encompasses 40 doctoral students with scientific profiles closely aligned with research at MAX IV. Students will have the unique opportunity to undertake a secondment at the laboratory, benefitting from MAX IV's co-supervision and expertise in utilizing X-ray synchrotron radiation.

Our User Science Programme, as with programmes at all other European light sources, was threatened in 2022 by the volatility of electricity costs. However, thanks to the unwavering support of our stakeholders, the continuity of our User Science Programme has remained our top priority.

The diverse range of topics covered in our 2022 Science Highlights reflects the breadth and depth of the research accomplished at MAX IV, from super-alloys, nanowires, and cancer-related polymerase to ferroelectricity, protein self-assembly into fibrils, and hydrogels.

MAX IV continues to deliver a compelling User Science Programme, thanks to the dedication and engagement of our user communities and staff. We are pleased to invite you to read a selection among the many scientific articles published in 2022, resulting from research conducted by the users and staff at MAX IV.

Marjolein Thunnissen & Aymeric Robert
Scientific Directors


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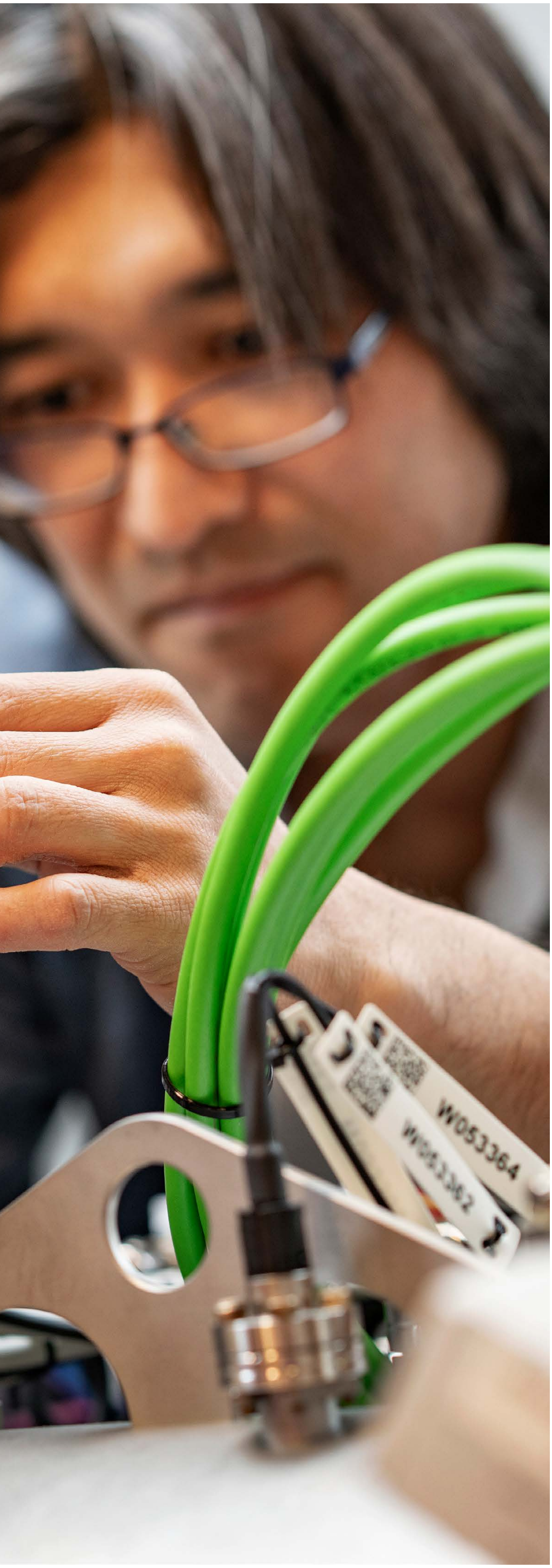
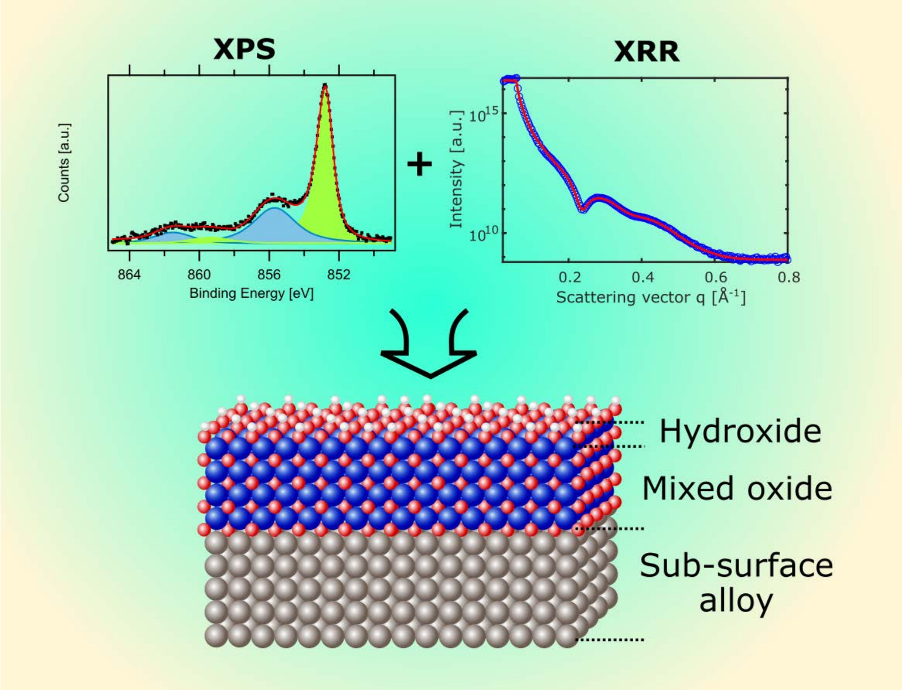


Science Highlights



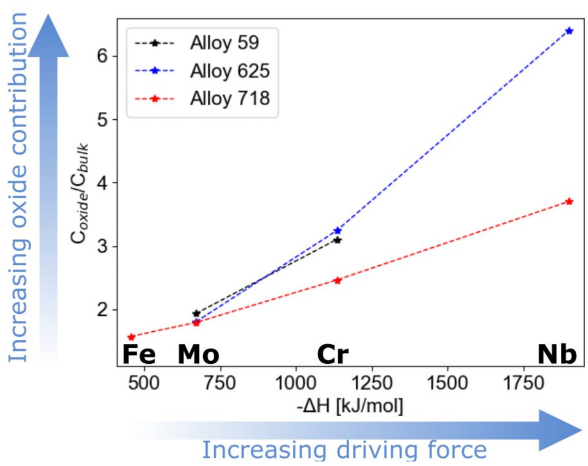
ADVANCED MATERIALS

Deciphering corrosion resistance of superalloys

To develop longer-lasting metallic materials for harsh operating conditions requires understanding of their surface composition, structure and properties. Researchers from KTH Royal Institute of Technology, Lund University, and Swedish steel company, Sandvik Materials Technology, investigated the surface chemistry and thickness of the protective native oxide layer of three industrial-grade, nickel superalloys with X-ray Photoelectron Spectroscopy (XPS) at MAX IV's FlexPES beamline and X-ray Reflectivity (XRR) at the Swedish Materials Science beamline P21.2 at PETRA III in Germany. The analysis revealed the differences between alloys, the preferential oxidation, and enrichment of alloying elements in the surface oxide films. This knowledge will further understanding of the differences in corrosion resistance between the alloys for potential design of more durable materials.

Publication

A. Larsson et al. Thickness and composition of native oxides and near-surface regions of Ni superalloys. *Journal Of Alloys And Compounds* 895 162657 (2022). DOI: 10.1016/j.jallcom.2021.162657



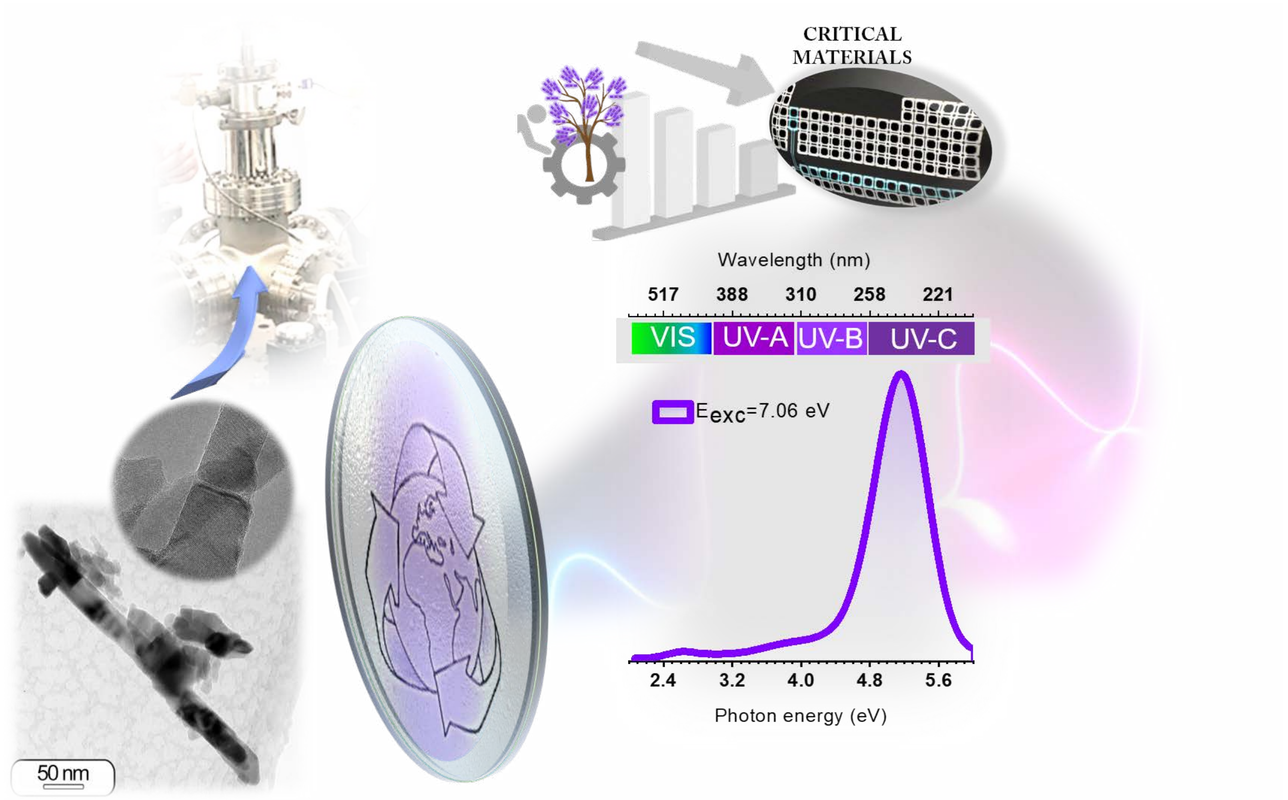
ADVANCED MATERIALS

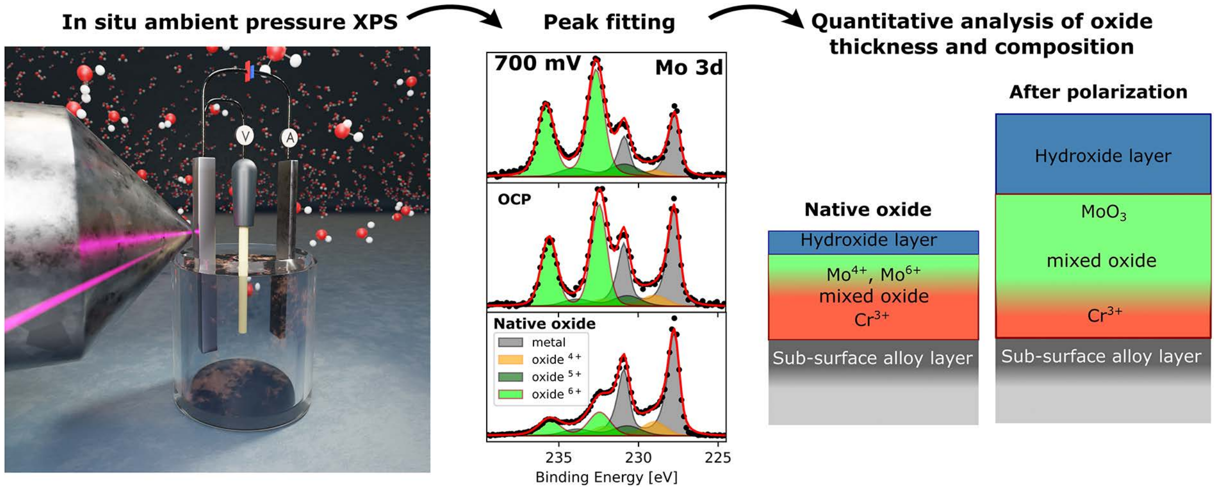
Non-toxic Deep UV phosphors for new sustainable technologies

A collaboration of Estonian and Spanish researchers has developed Deep-Ultraviolet (UV-C) emitters, known as phosphors, which do not contain rare earth elements, a depleting natural resource globally. Using a cost-effective wet chemistry method, the group synthesized undoped zinc aluminate ($ZnAl_2O_4$) nanofibers with a spectrally tuneable bright UV-C emission. The new phosphors represent a favourable alternative to conventional rare-earth doped phosphors utilised in tri-band-based white light-emitting diode (LED) technology, bio- and photochemistry and medical applications for disinfection or sterilization. The phosphor structure was analysed with X-ray absorption near-edge structure (XANES), extended X-ray absorption fine structure (EXAFS), X-ray diffraction (XRD), and Raman spectroscopy techniques. The luminescence properties were studied at MAX IV's FinEstBeAMS beamline. The work advances materials engineering aimed at creating non-toxic (mercury free) and non-critical (rare-earth free) materials, essential for the development of environmentally sustainable technologies.

Publication

R. E. Rojas-Hernandez et al. Deep-Ultraviolet Emitter: Rare-Earth-Free ZnAl2O4 Nanofibers via a Simple Wet Chemical Route, *Inorg. Chem.* 61, 11886 (2022). DOI: 10.1021/acs.inorgchem.2c01646





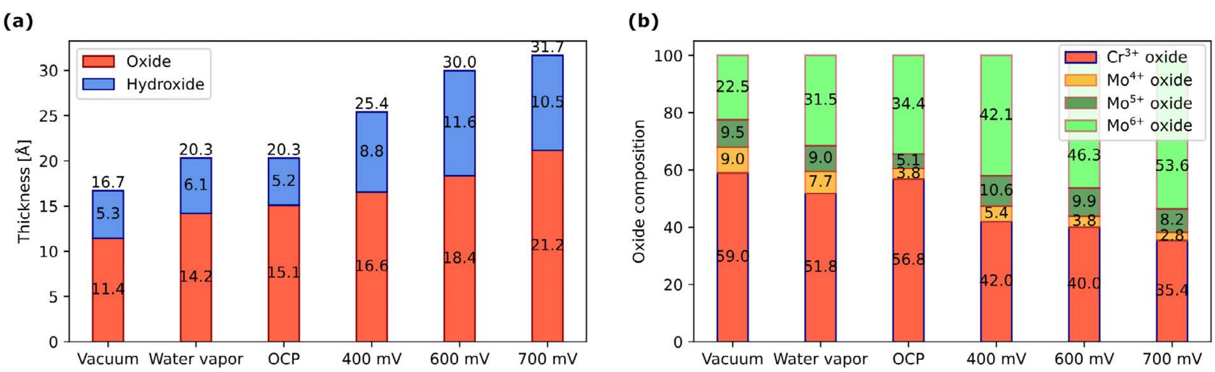
ADVANCED MATERIALS

Observations of the protective oxide film of metal surfaces

Researchers at Lund University and the Royal Institute of Technology (KTH) investigated the so-called passive film, a thin metal oxide layer which protects the surface of metals from oxidation and therefore, corrosion. The industrial alloy, nickel-chromium-molybdenum was tested under realistic conditions and pressures for the growth of the passive layer and its chemistry, thickness, and composition. The group used electrochemistry combined with ambient pressure X-ray photoelectron spectroscopy (AP-XPS) at MAX IV's HIPPIE beamline to observe the behaviour of the protective oxide films in solution, probing the oxide surface at the solid/liquid interface *in situ*. The study enhances knowledge of the surface chemistry of electrochemical systems, which is vital for the performance and degradation of batteries, fuel cells, and corrosion of industrial alloys. Understanding the mechanisms of corrosion and corrosion protection holds promise for the development of better alloys at reduced cost.

Publication

A. Larsson et al. In situ quantitative analysis of electrochemical oxide film development on metal surfaces using ambient pressure X-ray photoelectron spectroscopy: Industrial alloys. *Applied Surface Science* 611 (2023) 155714.
DOI: 10.1016/j.apsusc.2022.155714



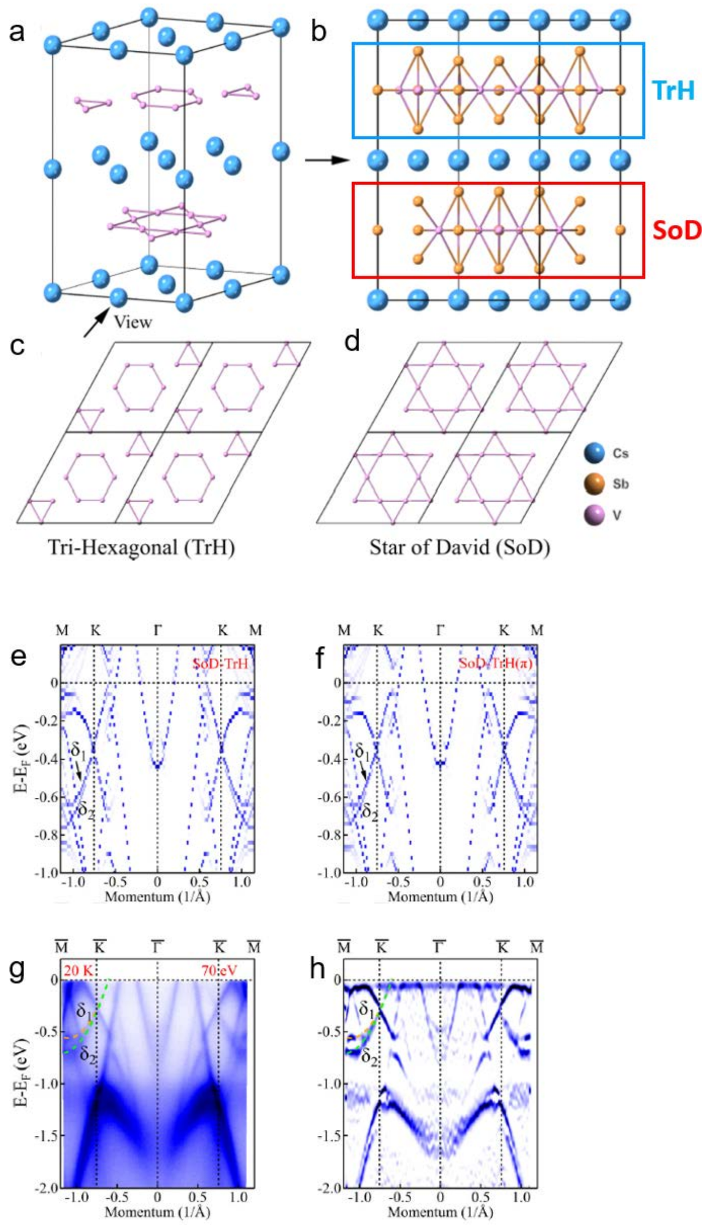
ADVANCED MATERIALS

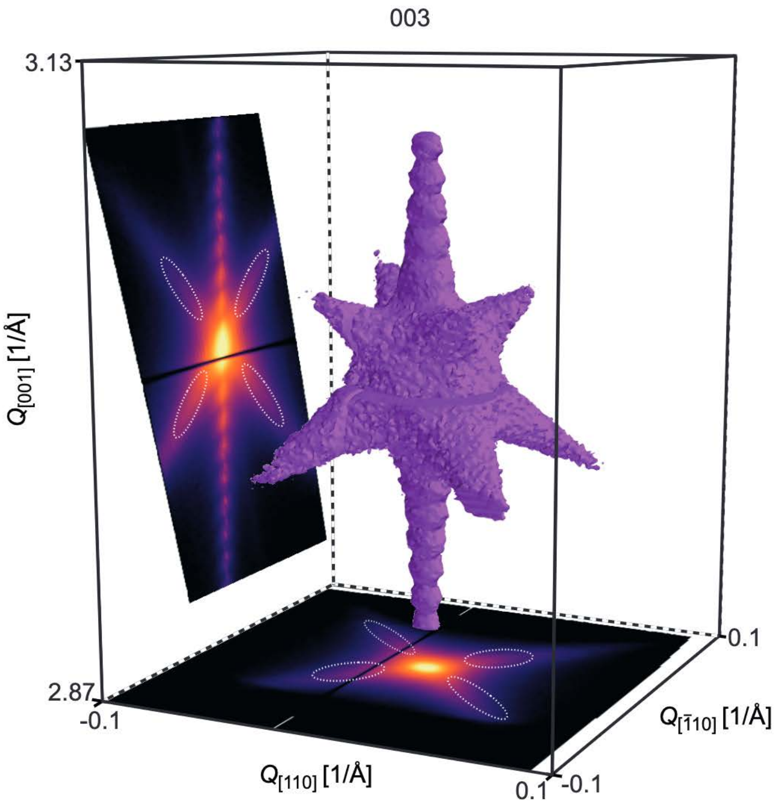
Coexistence of two intertwined charge density waves in a kagome system

An international research collaboration led by the Royal Institute of Technology (KTH) studied the charge density waves in a kagome system CsV_3Sb_5, a type of ferromagnetic quantum material, using angular resolved photoelectron spectroscopy (ARPES) at MAX IV's BLOCH beamline. They found that there are two intertwined charge density waves (CDW) coexist in CsV_3Sb_5. The "breathing" mode of a kagome lattice or triangular lattice can give rise to candidate distortions such as the Star of David (SoD) or its inverse structure, tri-hexagonal (TrH). However, to date, in most materials, only a single type of distortion has been observed at once. Utilising band calculations and ARPES measurements, the group reported the first strong evidence of the coexistence of two intertwined CDW order in kagome superconductor CsV_3Sb_5, which corresponding to the SoD and TrH distortions, and provide key insights into the 3D nature of the unconventional CDW in CsV_3Sb_5. The work offers a valuable window into the relationship between charge density waves and superconductivity.

Publication

C. Li et al. Coexistence of two intertwined charge density waves in a kagome system, *Phys Rev Research*. DOI: 10.1103/PhysRevResearch.4.033072





ADVANCED MATERIALS

Exploring the rich materials landscape of antiferromagnets

The pursuit for knowledge of the scaling limits of magnetic textures such as domain walls spans the full research field of magnetism from its physical fundamentals to applications in information technology. An international research group analysed the antiferromagnetic material CuMnAs with electron- and X-ray microscopy to illuminate the magnetic texture at nanoscale. They identified sharp (abrupt) crystal domain walls in the antiferromagnetic film corresponding to the Néel order reversal between two neighbouring atomic planes, opening a new research front of atomically sharp magnetic domain walls. Supporting measurements were performed with scanning X-ray diffraction microscopy at NanoMAX beamline at MAX IV. The study may enhance current understanding of electrical and ultrafast optical antiferromagnetic devices with magnetic field–insensitive neuromorphic functionalities. It opens another intriguing path into the rich materials landscape of antiferromagnets.

Publication

F. Krizek et al. Atomically sharp domain walls in an antiferromagnet. *Science Advances*. Apr 2022.
DOI: 10.1126/sciadv.abn3535

ADVANCED MATERIALS

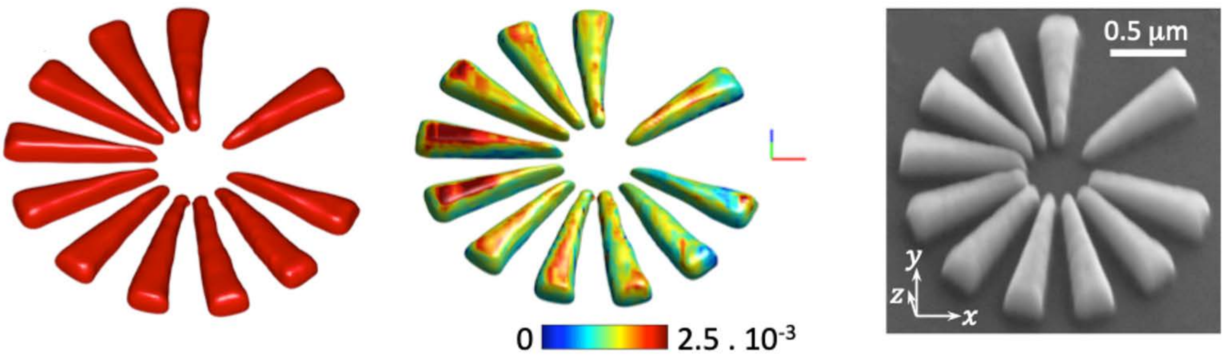
4th generation flux and nanoscale microscopy reveal clearest crystalline form

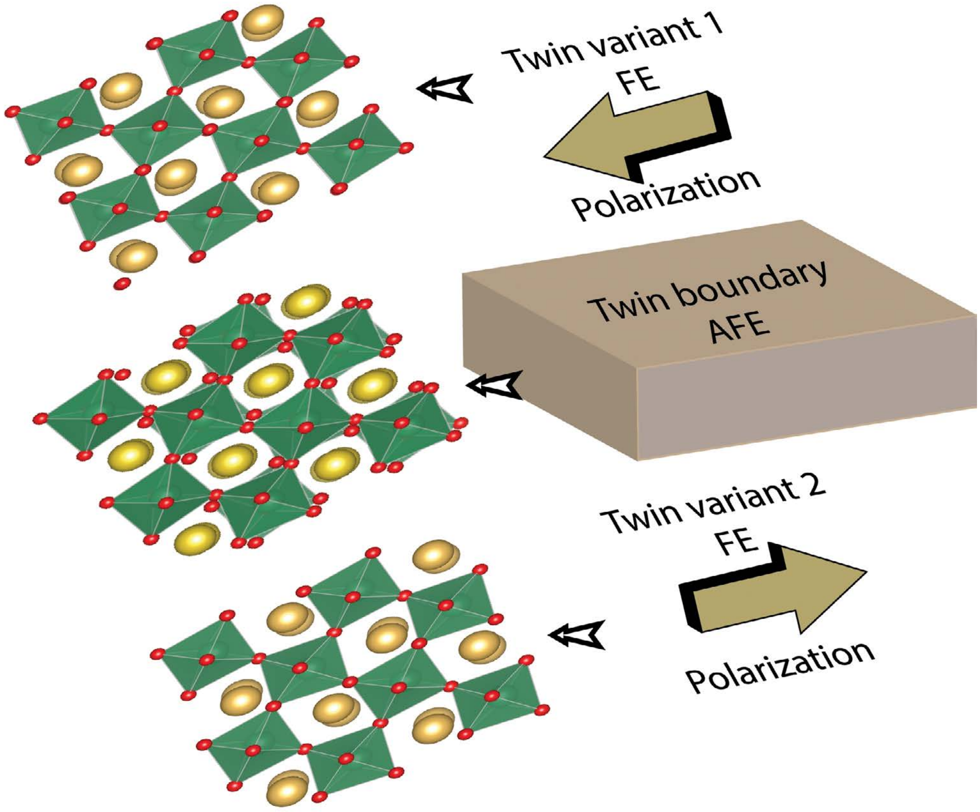
To capture extraordinary nanoscale details in crystals takes the powerful coherent flux of a 4th generation light source. An international research team has revealed 3D images of a complex crystalline star structure using Bragg ptychography, a technique based on the inversion of coherent data, at MAX IV's NanoMAX beamline. The study produced sharp, quantitative imagery primarily attributed to the unprecedented flux of coherent X-rays impinging on the silicon star (Si-star). Large datasets were generated with a quality enabling users to surpass experimental limitations such as positioning errors or poor scanning conditions to obtain a high-resolution image of the sample. 3D Bragg ptychography is valuable for investigation of crystal samples, from biominerals found in teeth, bones, shells and more, to a diversity of technologically relevant materials exhibiting magnetic, ferro-electric, or topological properties. The results demonstrate the possibility of unprecedented data quality beyond experimental limitations from new synchrotron sources.

Publication

P. Li et al. 4th generation synchrotron source boosts crystalline imaging at the nanoscale. *Light Sci Appl* 11, 73 (2022).

DOI: 10.1038/s41377-022-00758-z





ADVANCED MATERIALS

Antiferroelectric-Ferroelectric switching in perovskite oxides key to sustainable technologies

Transition to green technologies require advances in power electronics that can enable fast switching and improved energy efficiency. Next-generation dielectric materials are needed to build advanced power electronics chips that can operate under high voltage, high transient currents, and high temperatures. Ceramic capacitors made with antiferroelectric materials have been considered for this purpose. However, most of the commercially available antiferroelectric materials contains lead and it is desirable to develop new more sustainable alternatives.

A research team from City University of Hong Kong have used the DanMAX beamline to study the antiferromagnetic properties of lead-free perovskite oxides. The research addresses the atomic scale mechanism for transformation between antiferroelectric (AFE) and ferroelectric (FE) phases in the material $NaNbO_3$. In order to design new materials with reversible AFE-FE transformation, it is essential to gain insight into the atomic scale mechanism for such phase transformation. The key result of the study is that the two different phases in $NaNbO_3$ are related to each other through a twinning operation of the same local structural units.

Publication

C. S. Htet et al. Atomic structural mechanism for ferroelectric-antiferroelectric transformation in perovskite $NaNbO_3$, *Physical Review* B 105, 174113 (2022).
DOI: 10.1103/PhysRevB.105.174113

ADVANCED MATERIALS

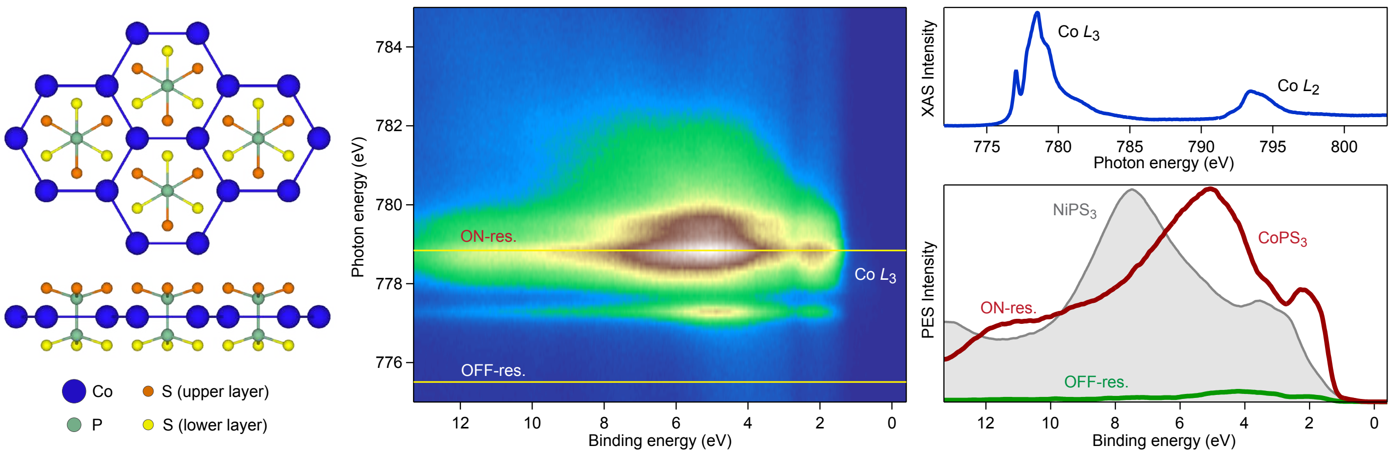
Layered van der Waals material show mixed character of insulating state

A group from Shanghai University has studied electronic structure of the antiferromagnetic van der Waals material $CoPS_3$. It belongs to the new class of layered materials called transition metal phosphorus trichalcogenides (MPTs) with the structural formula MPX_3 (M: transition metal ion; X: chalcogen, such as S or Se). The researchers used Near-Edge X-ray Absorption Fine Structure (NEXAFS) spectroscopy, and Resonant Photoelectron Spectroscopy (ResPES) at the FlexPES beamline.

According to theoretical analysis of their electronic structure, MPX_3 materials can be subdivided in two sub-classes – Mott-Hubbard and charge-transfer insulators. However, $CoPS_3$ should demonstrate the mixing character of the insulating state which the experimental data also confirm in very good agreement with theoretical calculations. The results show potential functionality of layered MPX_3 for applications such as opto-spintronics, sensing and catalysis. The next step is tailoring their electronic and magnetic properties to improve transport, optical and catalytic properties, and open for even more applications.

Publication

Y. Jin et al. Mixed Insulating State for van der Waals $CoPS_3$, *J. Phys. Chem. Lett*. 13, 10486 (2022).
DOI: 10.1021/acs.jpclett.2c02992



ADVANCED MATERIALS

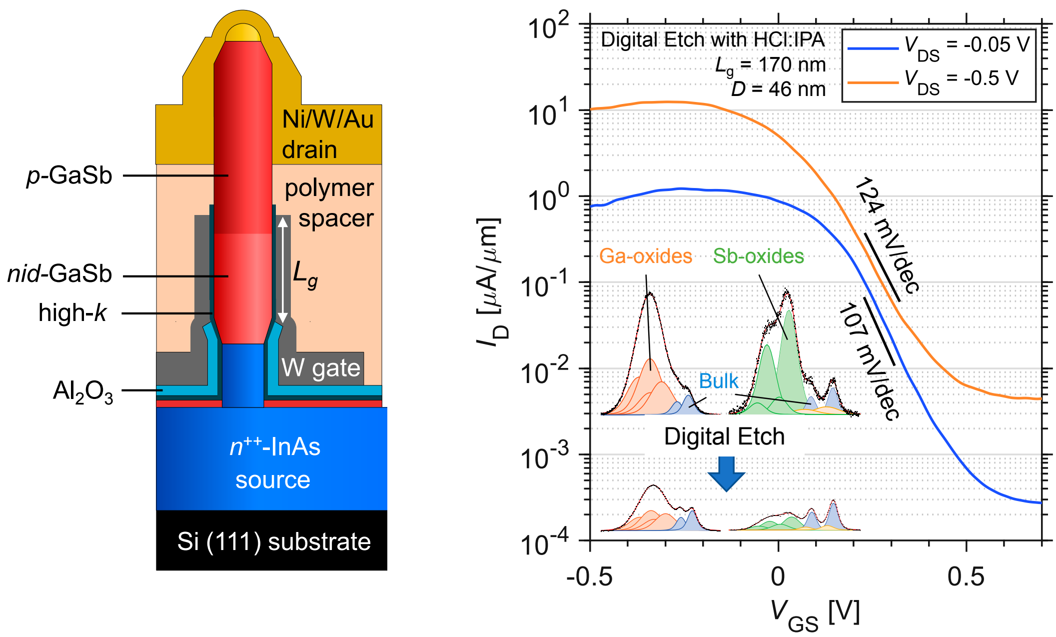
Better gate control for sustainable transistors

Sustainable electronics need to be energy efficient. A team from Lund University recently studied nanowire transistors to improve the electrostatic control of the gate. The material under study was gallium antimonide (GaSb) and the results indicate a high potential of antimony-based p-type devices for all-III-V CMOS technologies on silicon.

III-V compound semiconductors have high carrier mobility and thus a high potential to replace silicon as the channel material for transistors. Both n-type and p-type transistors are required to form complementary metal-oxide-semiconductors. Unlike III-V n-type counterparts which have already overperformed silicon transistors, the performance of III-V p-type transistors still need to be improved. The most important achievement of this study is that by improving the interface quality there is less voltage required to switch the transistor on and off. This means that we can lower the power consumption of using this type of electronic devices.

Publication

Z. Zhu et al. Improved Electrostatics through Digital Etch Schemes in Vertical GaSb Nanowire p-MOSFETs on Si, *ACS Appl. Electron. Mater.* 4, 531 (2022).
DOI: 10.1021/acsaelm.1c01134



ADVANCED MATERIALS

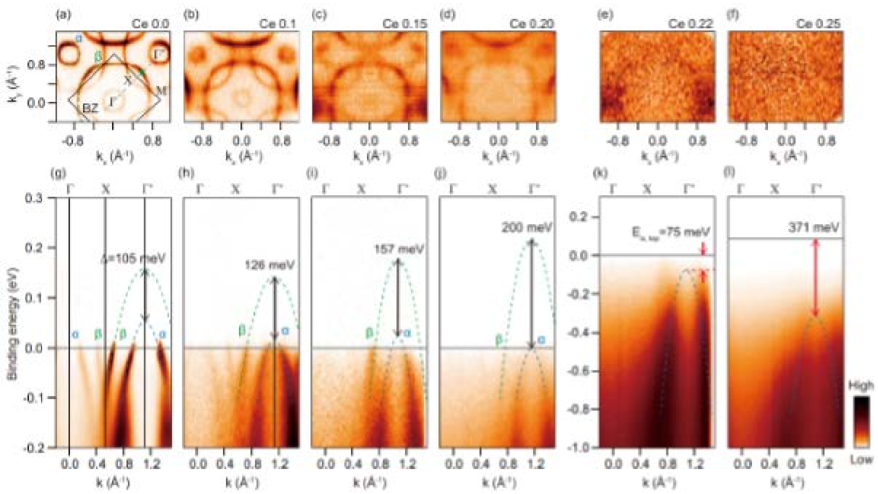
Novel metal-insulator transition in doped Sr_2RhO_4 associated with multi-band structures

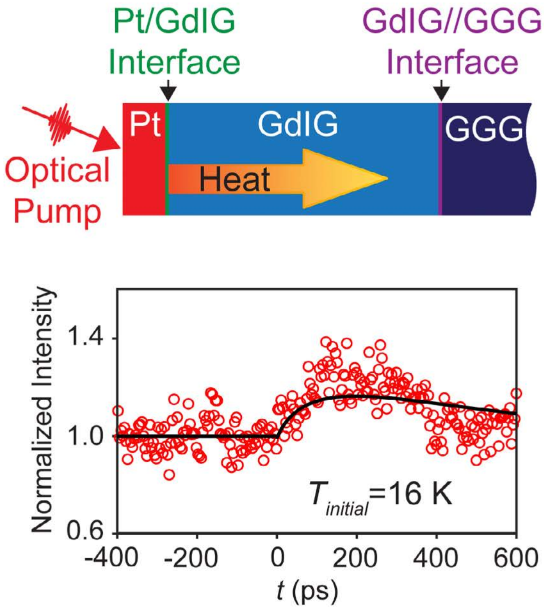
A team led by researchers from South Korea has studied the metal-insulator transition in La and Ce doped Sr_2RhO_4 systems. Intriguingly, both systems undergo a metal-insulator transition at a specific level of electron doping from the dopants, with a non-integer electron number. This implies that the system does not conform to a conventional insulator that could be explained by band theory or Mott physics.

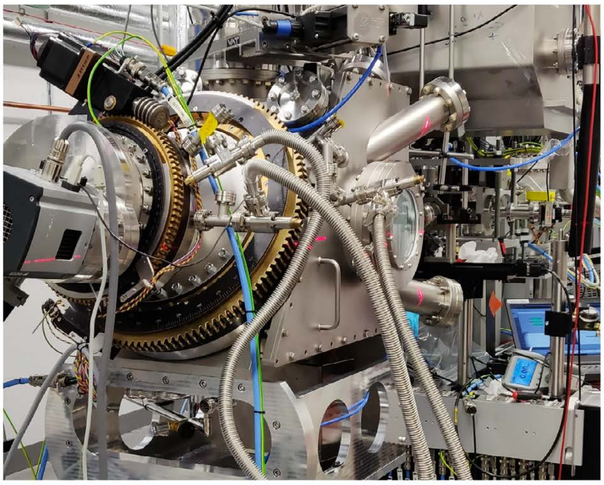
The team used Angle-Resolved Photoemission Spectroscopy (ARPES) to observe and analyse the evolution of each band of the systems in response to electron doping. They quantified the relative energy levels of each band and ascertained that the multi-band structure in the target systems was fundamentally formed by the effective spin-orbit coupling. By tracking the occupation of individual bands, they proposed a mechanism for the observed metal-insulator transition. Since multiple bands each has its distinct role in systems like Sr_2RhO_4, the findings demonstrate that electronic properties in systems do not occur merely due to the overall summation of electrons near the Fermi level. Instead, these properties are concealed within individual bands. The result opens the door to the possibility that other physical phenomena may be similarly hidden among multi-band structures.

Publication

K. Junyoung et al. Universality of charge doping driven metal-insulator transition in Sr_2RhO_4 and role of spin-orbit coupling, *Phys. Rev. B* 106, L241114 (2022). DOI: 10.1103/PhysRevB.106.L241114







ADVANCED MATERIALS

Understanding heat transport in nanostructures for future spin technologies

An international team of researchers has studied low-temperature nanoscale heat transport using the ultrafast time-resolved X-ray diffraction capabilities of MAX IV's FemtoMAX facility. This emerging area of condensed matter research ultimately involves understanding and exploiting the interaction between magnetism and heat flow at the nanoscale, an area termed spincaloritronics. The specific materials studied by the team consisted of magnetic oxide thin films with the garnet crystal structure with a platinum metal thin film at the surface. These systems combine spintronic and thermoelectric functionalities by facilitating the interconversion of spin, charge, and heat currents. It is particularly important to understanding how nanoscale magnetic properties depend on the temperature, and especially the gradient of the temperature, within these materials.

The experiments at FemtoMAX probed the flow of heat generated by an ultrafast optical pulse absorbed in the Pt layer into the magnetic garnet using ultrafast X-ray diffraction, an experiment that was possible at only a few facilities world-wide. The results will be beneficial to emerging technologies based on spincaloritronics, in which electrical currents are derived from heat transport in magnetic materials and in using rapidly heated and cooled magnetic materials to improve magnetic data storage.

Publication

D. Sri Gyan et al. Low-temperature nanoscale heat transport in a gadolinium iron garnet heterostructure probed by ultrafast x-ray diffraction. *Struct Dyn* 9, 045101 (2022).
DOI: 10.1063/4.0000154

ADVANCED MATERIALS

Trion study paves way for future optoelectronic devices

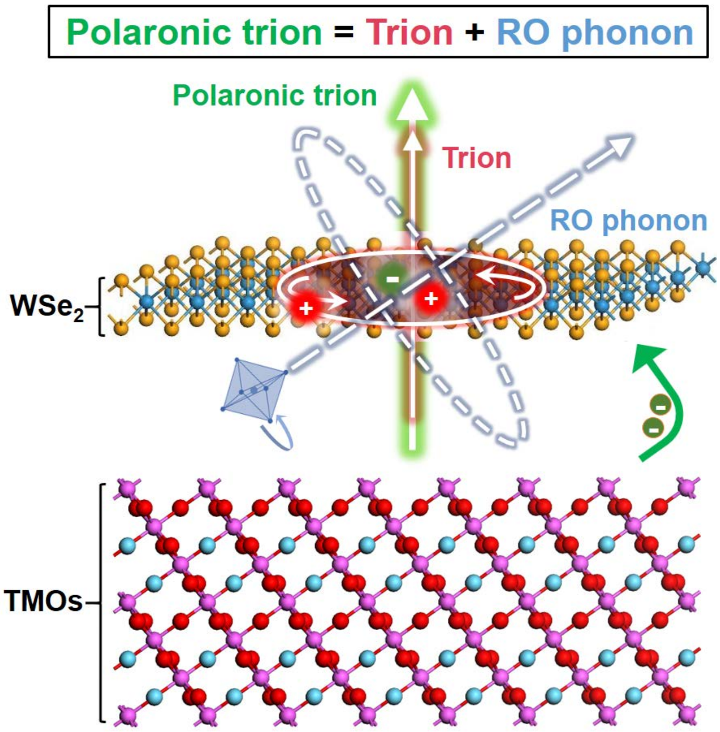
A team of researchers from the University of Science and Technology of China have realised a polaronic trion in the transition-metal dichalcogenide WSe_2. The team fabricated monolayer WSe2 and transition metal oxide (TMO) heterojunctions with clean interfaces. The strong interfacial coupling between WSe_2 and TMOs was confirmed by the X-ray photoelectron spectroscopy (XPS) and X-ray absorption spectroscopy (XAS) at the MAXPEEM beamline.

Tightly bound quasiparticles in a two-dimensional (2D) semiconducting transition metal dichalcogenides (TMDs), such as excitons and trions, have inspired attractive application prospects for exploring multi-body effects and engineering quantum techniques. Effective control of these quasiparticles is of prime importance for the development of quasiparticle-based optoelectronic applications driven by many-body effects. However, the demanding external field and complex device structures have greatly restricted their development.

The researchers have now fabricated monolayer WSe_2/TMOs heterostructures to introduce a strong interfacial coupling, as verified by their results from the study at MAXPEEM. This paves the way for the application of 2D TMDs in the future quasiparticle-based optoelectronic devices.

Publication

Y. Wang et al. Polaronic Trions Induced by Strong Interfacial Coupling in Monolayer WSe_2. *Adv. Electron. Mater.* 2023, 9, 2200852.
DOI: 10.1002/aelm.202200852



ADVANCED MATERIALS

Zinc in a link: A journey through ZnO_2 and ZnO local structure and lattice dynamics

Researchers from the EXAFS Spectroscopy Laboratory at the Institute of Solid State Physics (University of Latvia) investigated the decomposition of zinc peroxide (ZnO_2) to zinc oxide (ZnO) using in-situ X-ray absorption spectroscopy (XAS) combined with reverse Monte Carlo (RMC) simulations. The group reported a detailed understanding of the temperature-dependent local structure and lattice dynamics of two zinc oxide phases (hexagonal ZnO and cubic ZnO_2) and the mechanism of ZnO_2-to-ZnO decomposition.

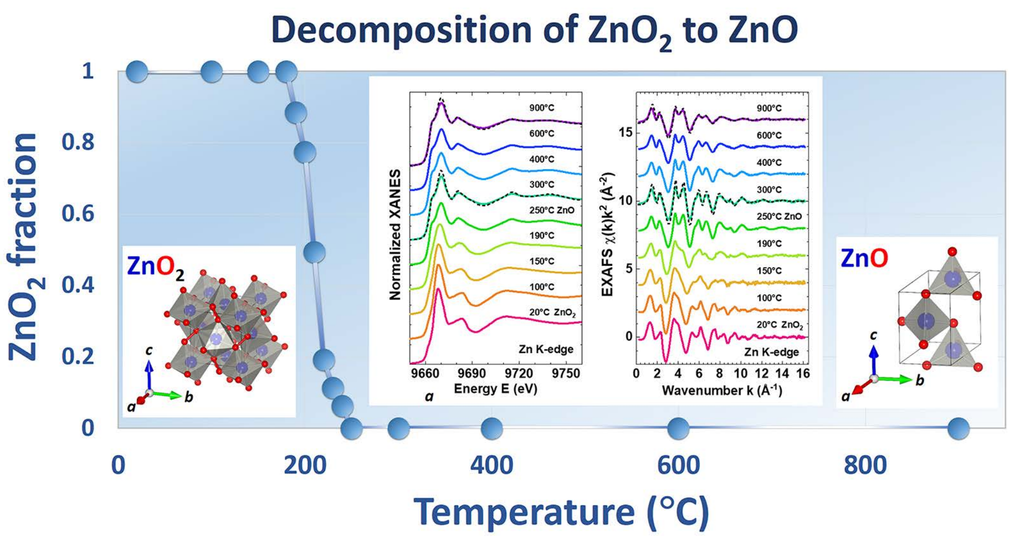
Zinc peroxide can be used as a precursor to producing ZnO nanoparticles and thin films with numerous applications, for example, in printed optoelectronics as a part of inkjet inks. Therefore, it is vital to understand how the decomposition of zinc peroxide proceeds and how zinc oxide nanoparticles grow upon further heating.

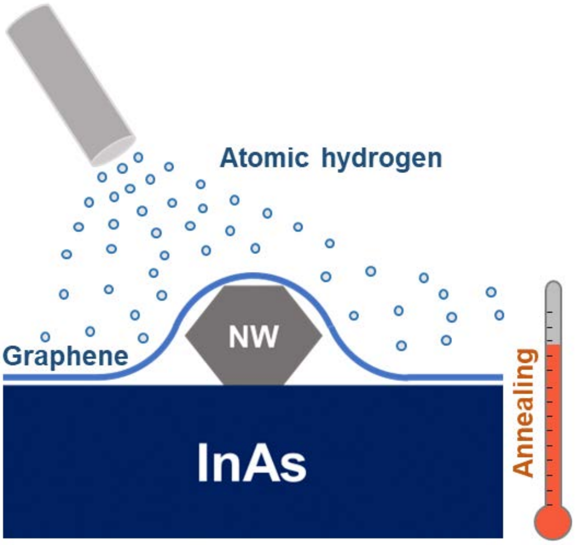
Based on the principal component and linear combination analyses of the extended X-ray absorption fine structure (EXAFS) spectra, scientists found that the ZnO_2 phase exists up to 180 °C. In contrast, only the nanocrystalline ZnO phase is present above 250 °C. After the RMC simulations, a substantial increase in disorder was found upon approaching the decomposition temperature.

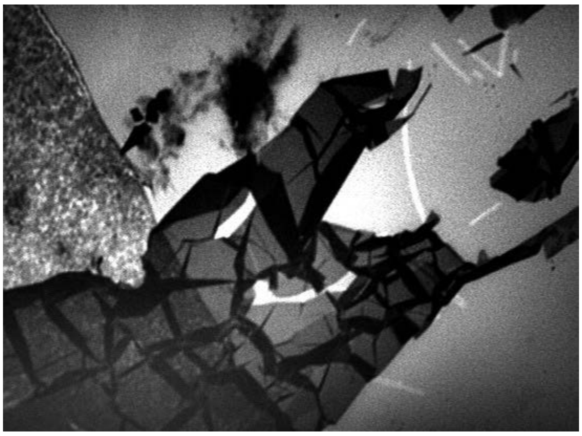
Here, researchers demonstrated that combining X-ray absorption spectroscopy at the Balder beamline with advanced data analysis is a powerful technique for in-situ and operando investigations of the local environment in materials with varying degrees of crystallinity and disorder. This combined technique is proposed to track phase transitions in complex compounds.

Publication

A. Kuzmin et al. In Situ Study of Zinc Peroxide Decomposition to Zinc Oxide by X-Ray Absorption Spectroscopy and Reverse Monte Carlo Simulations. *Phys. Status Solidi B*, 259: 2200001. DOI: 10.1002/pssb.202200001







ADVANCED MATERIALS

Graphene on InAs surfaces and nanowires: Establishing a 'clean interface' for future semiconductor technologies

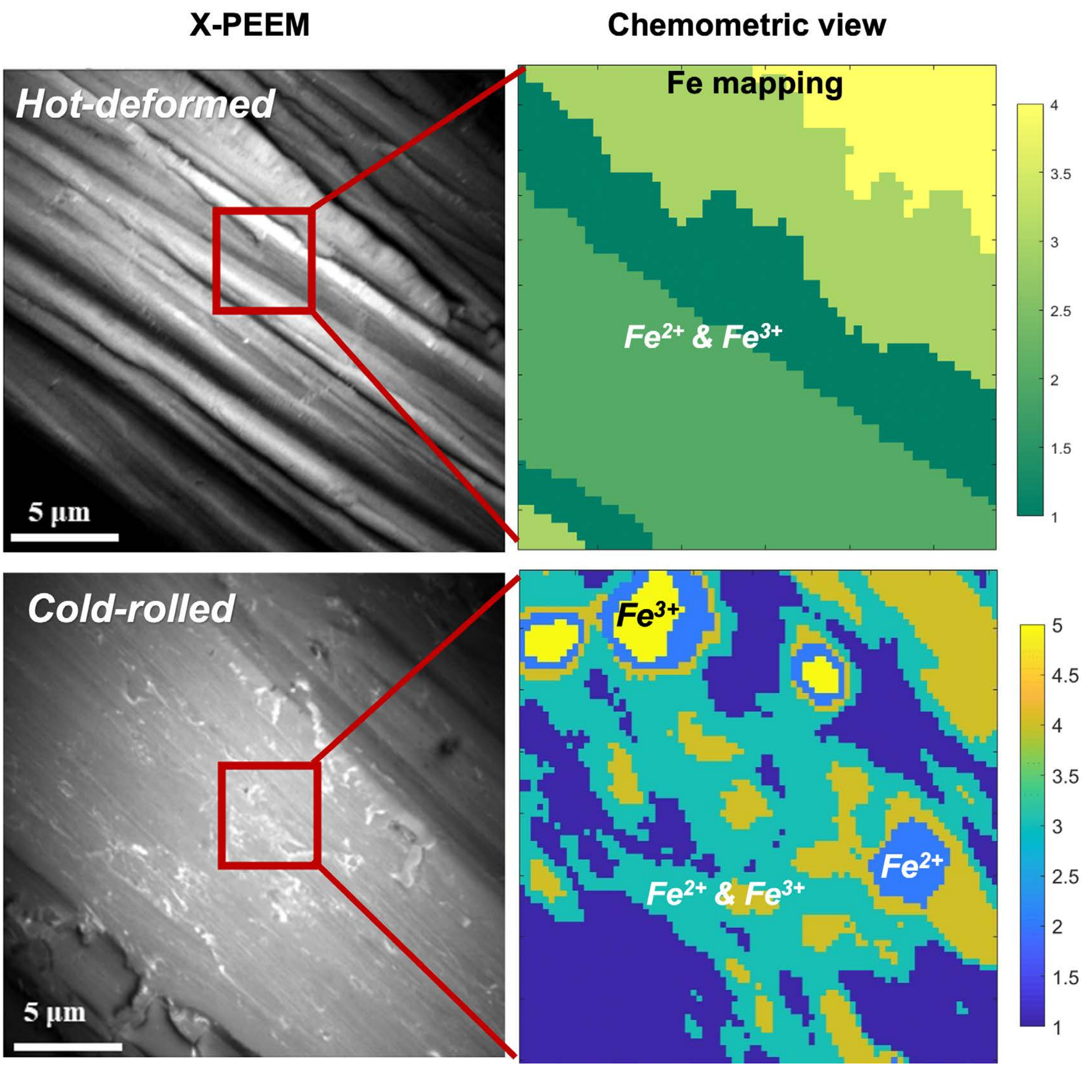
Highly conductive graphene and semiconducting III–V compound nanowires are two nanomaterials classes that promise optoelectronic, photovoltaic, and quantum technology applications. Its combination has resulted in exciting applications, such as transparent organic single-crystal field-effect transistors and high-responsivity heterojunction photodetectors, etc.

Folding two-dimensional graphene around one-dimensional InAs semiconductor nanowires could yield a new class of hybrid nanomaterials combining their excellent complementary properties. To achieve this, a 'clean interface' is crucial. In particular, removing the present native oxide on the InAs under the graphene is needed. Oxides can hinder the formation of ohmic contacts, and defects in the oxides can be detrimental to electrical and optical properties.

Scientists from NanoLund used LundNanoLab micro/nanofabrication facilities (part of the national Myfab infrastructure) together with MAX-PEEM beamline to answer the question on the potential of atomic hydrogen cleaning underneath graphene. They showed that the desired clean interface can be achieved by using atomic hydrogen annealing. They also found that the cleaning process would make the graphene fold tighter around the nanowire. The tight folding properties of the graphene and the clean interface open interesting prospects for using graphene as a wrap-around gate electrode for quantum technologies. Besides that, the hydrogen treatment for removal of the native oxide has been proven to work for a wide range of semiconductor materials, indicating that the present conclusions are not limited to InAs but extend more widely.

Publication

S. Fatemeh Mousavi et al. Atomic Hydrogen Annealing of Graphene on InAs Surfaces and Nanowires: Interface and Morphology Control for Optoelectronics and Quantum Technologies. *ACS Applied Nano Materials* 2022 5 (12), 17919-17927.
DOI: 10.1021/acsanm.2c03891



ADVANCED MATERIALS

Lighting the way towards stronger steel

Why the same steel, when treated differently, exhibits different corrosion behaviors? To answer this, an international collaboration led by NANOMO, University of Oulu conducted a study on the corrosion of hot-deformed and cold-rolled stainless steel. Researchers used advanced spectro-microscopy and chemometric analysis techniques to study the nanoscale differences in steel microstructures, taking advantage of MAX-PEEM beamline. Their research found that the hot deformed steel had a more uniform iron oxide composition. In contrast, the microstructure of the cold rolled steel was more heterogeneous, with distinct areas of varying di- and tri-valent iron compositions. This is a critical insight into the impact of nanoscale inhomogeneity on steel corrosion.

This understanding will lead to designing and processing steel with optimal properties, such as mechanical strength, elasticity, corrosion, and hydrogen resistance. Understanding the relationship between the nanoscale variation of steel microstructures and their performance will help to develop resilient steel.

Publication

H. Singh et al. Unveiling nano-scaled chemical inhomogeneity impacts on corrosion of Ce-modified 2507 super-duplex stainless steels. *npj Materials Degradation* 6, 54 (2022).
DOI: 10.1038/s41529-022-00263-z

ADVANCED MATERIALS

AP-XPS study settles ethanol VS ethoxy on Rutile TiO2(110)

The open question how ethanol adsorbs onto rutile TiO_2 ($rTiO_2$) and its stability is now settled. Researchers from the Department of Physics, Lund University, reported that whilst the ethoxies are slightly more stable, both molecular ethanol and dissociated ethoxy species coexist on the $rTiO_2$ surface at room temperature. They also reported for the first time that a multilayer of ethanol is formed at a higher pressure and at room temperature. Considering the simplicity of the ethanol/$rTiO_2$ system with its many applications in the field of energy, the lack of surface-sensitive ambient pressure studies was a missing key piece of understanding in the field.

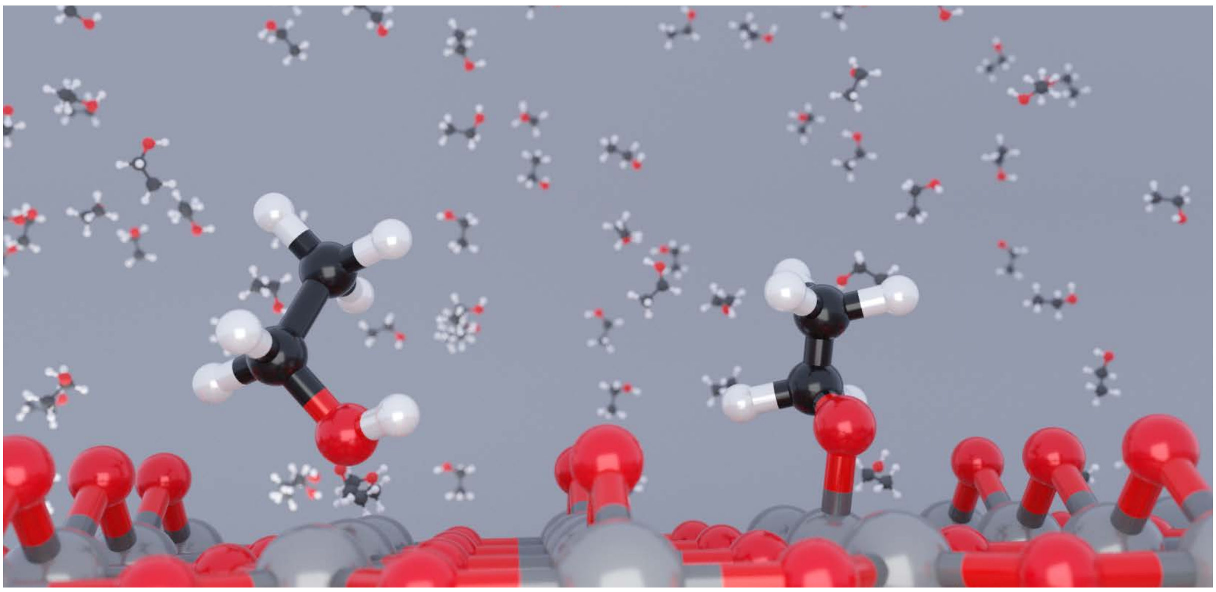
The fundamental study combined ambient pressure x-ray photoelectron spectroscopy (AP-XPS) at SPECIES beamline to bridge the pressure gap between idealised UHV measurements and 'messier' atmospheric pressures and conditions to differentiate between ethanol's two adsorption modes.

TiO_2 photoactivity is interesting for surface reactions. TiO_2 electrodes are active for hydrogen evolution production via water-splitting and ethanol degradation under ultraviolet (UV) illumination in electrochemical cells. An important question here is how ethanol adsorbs onto the TiO_2 surfaces.

This also provides a background for the scientists' studies of thin film growth on the solid surface from vapour precursors. Many of these precursors contain ethoxy ligands, which can react with the surface and surface-adsorbed protons. This research elucidates the reaction mechanisms of the precursors with TiO_2 surfaces and that near-ambient pressures can lead to considerable changes in adsorption behavior.

Publication

R. Jones et al. AP-XPS Study of Ethanol Adsorption on Rutile TiO_2(110). *The Journal of Physical Chemistry C* 2022 *126* (39), 16894-16902.
DOI: 10.1021/acs.jpcc.2c05389



CATALYSIS

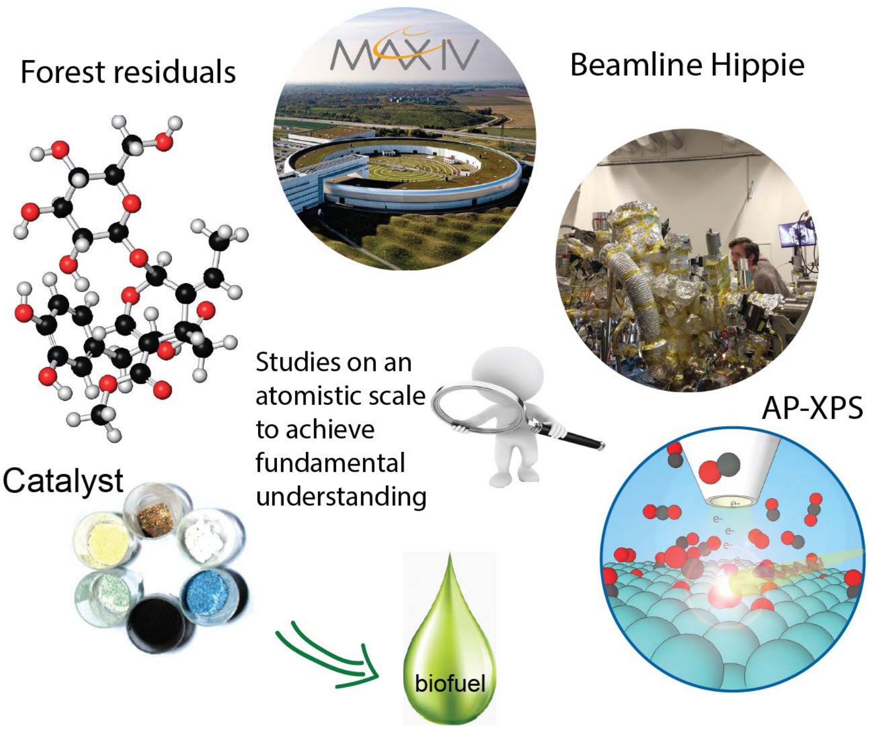
Catalytic action studied for upgrade of plant-based waste to biofuels

Molybdenum-based (Mo) catalysts improve the efficiency of chemical reactions in industrial hydrotreatment processes and are often used for removal of toxins in petroleum-based feedstocks for diesel and gasoline or valorisation of biomass. Researchers at Lund University and Malmö University studied the initial reduction of alumina supported nickel-molybdenum (NiMo) catalysts to understand how the composition of the catalysts influences their reduction and activation.

Results showed the aluminum support of the NiMo-catalysts strongly influences their reduction behavior, and the reduction temperature was tuneable by modifying the ratio of elements. Measurements at MAX IV's HIPPIE and Balder beamlines were taken with ambient-pressure X-ray photoelectron spectroscopy (APXPS) to decipher the catalysts' chemical surface properties, and X-ray absorption near-edge structure (XANES) to evaluate the oxidation state of the catalysts during activation. The work provides fundamental understanding of chemical and physical changes of the NiMo-catalyst, namely used to upgrade plant-based waste productions of the pulp and paper industry, such as lignin, to biofuels.

Publication

S. M. Gericke et al. In Situ H_2 Reduction of Al_2O_3-Supported Ni- and Mo-Based Catalysts. *Catalysts*
DOI: 10.3390/catal12070755



CATALYSIS

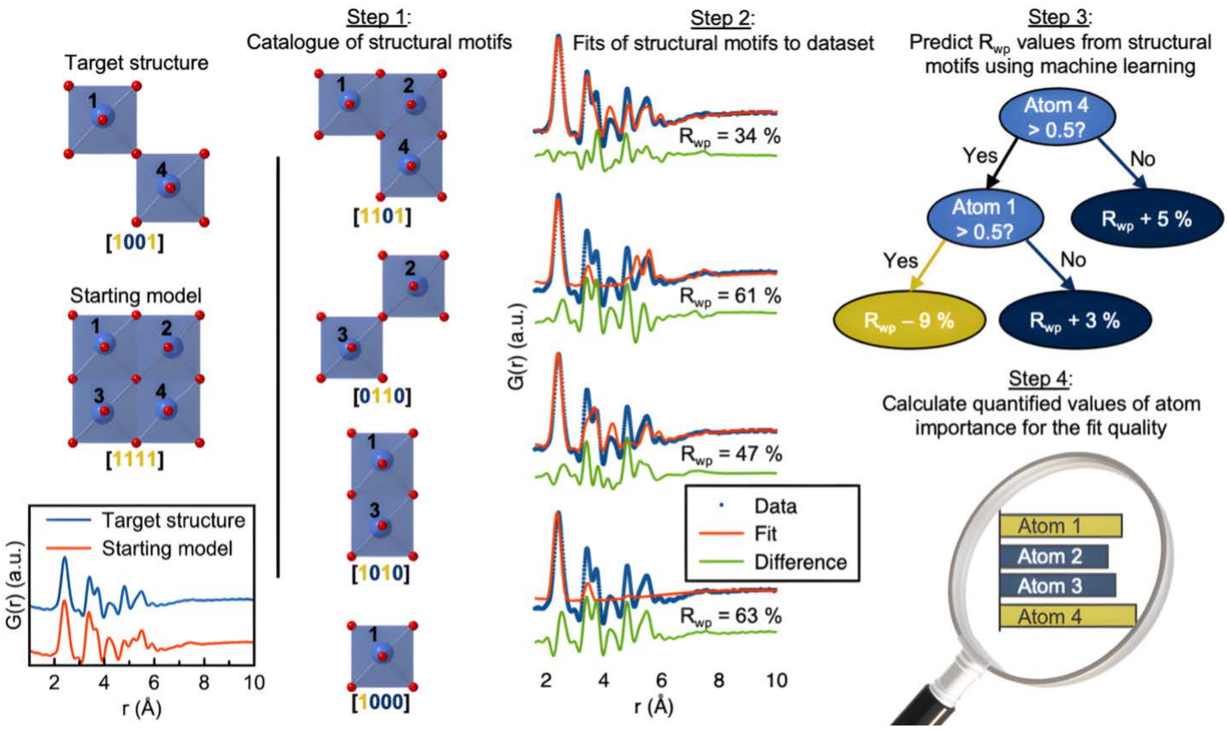
Catalyst essential for fuel cells studied in operando

Development of new catalysts is essential for the transition to zero-carbon technologies. One of the persistent problems in hydrogen fuel cell applications, is that small impurities can lead to deactivation of a fuel cell. This is especially relevant if the H_2 is supplied from biomass gasification. To eliminate impurities such as carbon monoxide, a common solution is to have a pre-treatment step (called PROX) which converts it into carbon dioxide.

A group from Aarhus University in Denmark used the HIPPIE beamline to study cobalt oxide films on platinum surfaces to catalyse the PROX reaction. They studied the material operando using Ambient Pressure X-ray PhotoElectron Spectroscopy (AP-XPS) The method let them follow evolution of the cobalt oxidation state as the catalyst becomes active in oxidation of carbon monoxide. Improvement of catalysts need to be based on a more detailed understanding of how they work and synchrotron experiments are likely to change our current knowledge dramatically.

Publication

E. Rattigan et al. The cobalt oxidation state in preferential CO oxidation on CoO_x/Pt(111) investigated by operando X-ray photoemission spectroscopy. *Phys. Chem. Chem. Phys.* 24, 9236 (2022).
DOI: 10.1039/D2CP00399F



INSTRUMENTATION

Nanomaterial structure revealed by explainable machine learning approach

A study led by researchers from the University of Copenhagen, focusing on using Explainable Machine Learning (XML), that is, Machine Learning with reasoning understandable to humans, to analyse X-ray total scattering data, was conducted at DanMAX. The analysis was made particularly in the context of nanomaterials with limited long-range order by analysing their pair distribution function, which is a way of describing distances between the atoms in the material. By refining a large number of structural motifs to the data and employing XML, the researchers can understand how each atom influences the goodness-of-fit. This process is called Machine Learning based Motif Extractor (ML-MotEx).

Conventional crystallographic methods face limitations when dealing with nanomaterials, for which the pair distribution function has proven to be a powerful tool. However, automated pair distribution function approaches are currently computationally expensive and time consuming. The ML-MotEx approach enables faster and more accurate analysis of nanomaterials and their structures by leveraging XML. The process can ultimately contribute to developing new materials with tailored properties.

Publication

A. S. Anker et al. Extracting structural motifs from pair distribution function data of nanostructures using explainable machine learning. *npj Comput Mater* 8, 213 (2022).
DOI: 10.1038/s41524-022-00896-3

The tool is available at
github.com/AndySAnker/ML-MotEx

INSTRUMENTATION

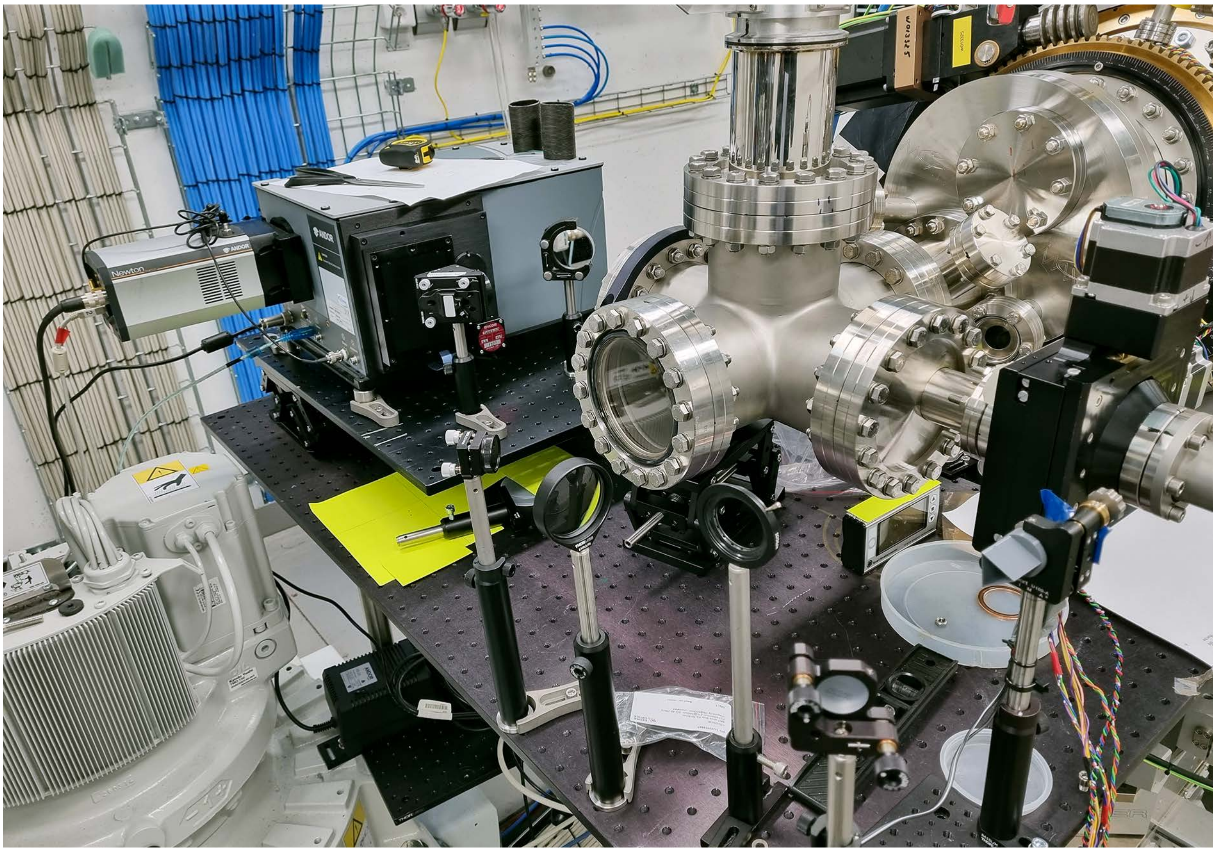
European time-resolved low-temperature luminescence spectroscopy in focus

Research toward novel scintillators and optical materials continues with the two permanent and two mobile luminescence stations at MAX IV and DESY, focusing on time-resolved techniques. Led by scientists from the University of Tartu, Estonia, a group of international researchers discussed and analysed the capabilities and challenges these infrastructures hold for research. MAX IV's FinEstBeAMS and Petra III's P66 beamlines have permanent time-resolved luminescence setups. FinEstBeAMS operates at 4.5–1300 eV in grazing incidence geometry, and P66 covers 4–40 eV in normal incidence geometry. On the other hand, mobile luminescence setups have been developed for studies at FemtoMAX and P23 beamlines. FinEstBeAMS, P66, and P23 provide time resolution from ~160 to 100 ps. FemtoMAX, however, provides an excellent time resolution of ~30 ps, limited by the time response of the photodetector.

Future research conducted at these stations hold promises for technological and new materials developments.

Publication

S. I. Omelkov et al. Recent advances in time-resolved luminescence spectroscopy at MAX IV and PETRA III storage rings. 2022. *J. Phys.: Conf. Ser.* 2380 012135. DOI: 10.1088/1742-6596/2380/1/012135



INSTRUMENTATION

Unfolding XPCS potentials at NanoMAX

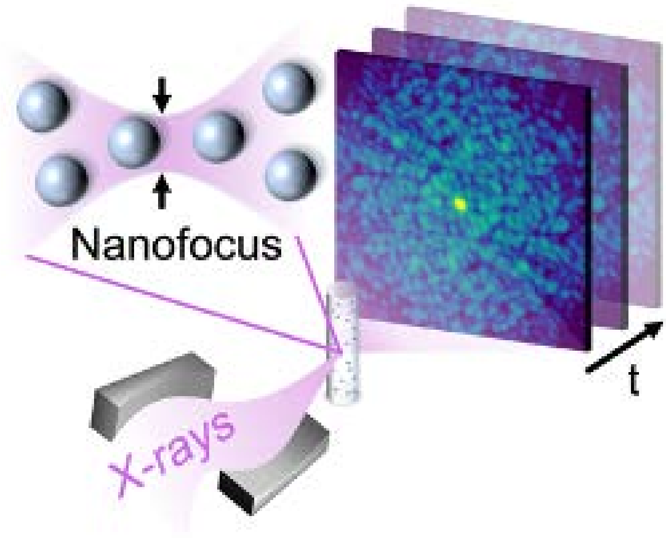
A close collaboration between MAX IV and the Department of Physics at Stockholm University resulted in the first proof-of-principle nano-focused X-ray photon correlation spectroscopy (XPCS) experiment. Researchers utilized the coherent properties and nano-focused X-ray beam at NanoMAX beamline to accurately measure structural dynamics in prototypical nanoparticle solutions by XPCS. This leads to a multifold increase in the speckle contrast, translating into a higher-to-noise ratio.

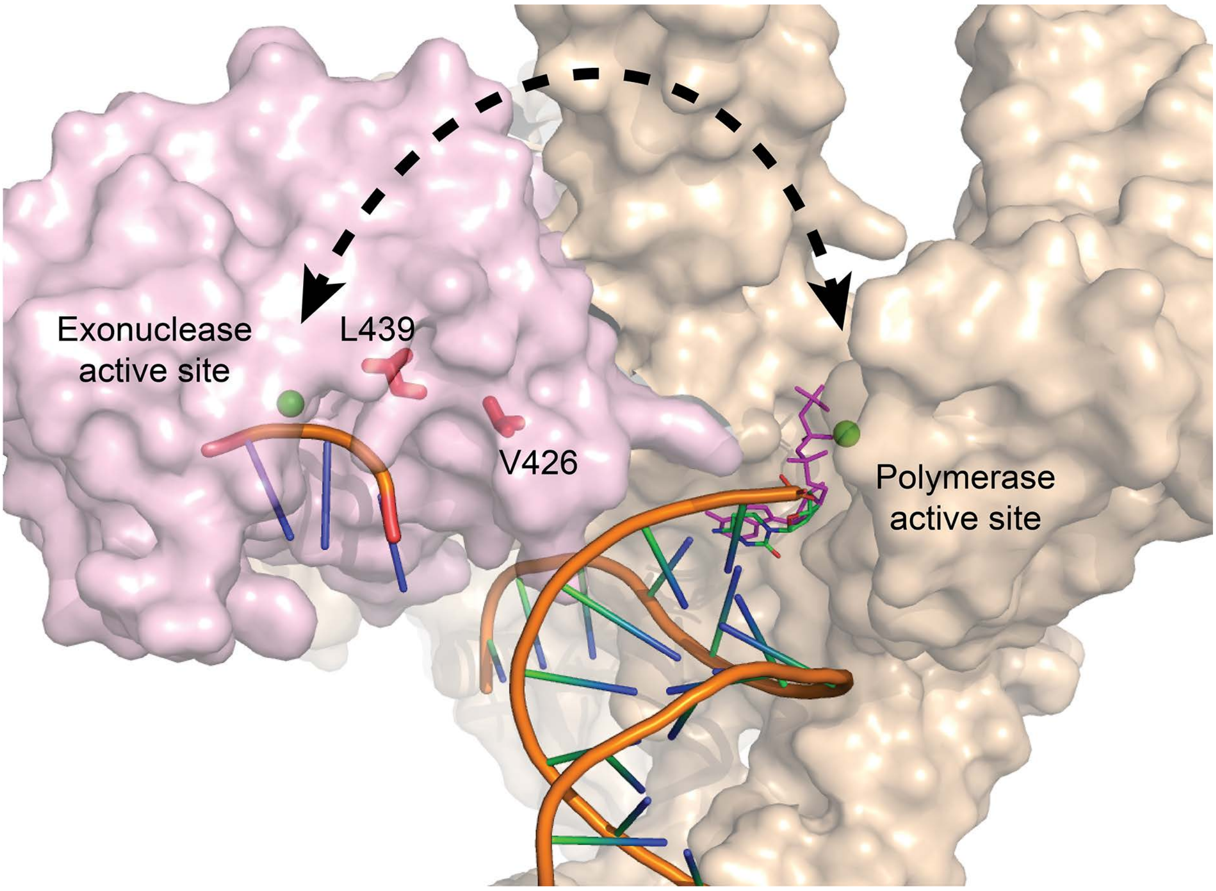
Tristan, a new state-of-the-art event-based X-ray detector, was used during the XPCS measurements. It allowed the researchers to capture the dynamics with a time resolution of microseconds (potential to reaching nanoseconds).

Combining the high spatial resolution of a nano-focus X-ray with the ability to measure structural dynamics by XPCS enables new access to such vastly unexplored phenomena. Nanofocused XPCS is a novel technique that can resolve the nanoscale fluctuations in a wide range of systems. It will help the understanding of complex physical mechanisms, for example, in liquid-liquid phase separation in biological solutions, which can help to develop new medical treatments for pathological disorders.

Publication

S. Berkowicz et al. Nanofocused x-ray photon correlation spectroscopy. *Phys. Rev. Research* 4, L032012, 25 July 2022. DOI: 10.1103/PhysRevResearch.4.L032012





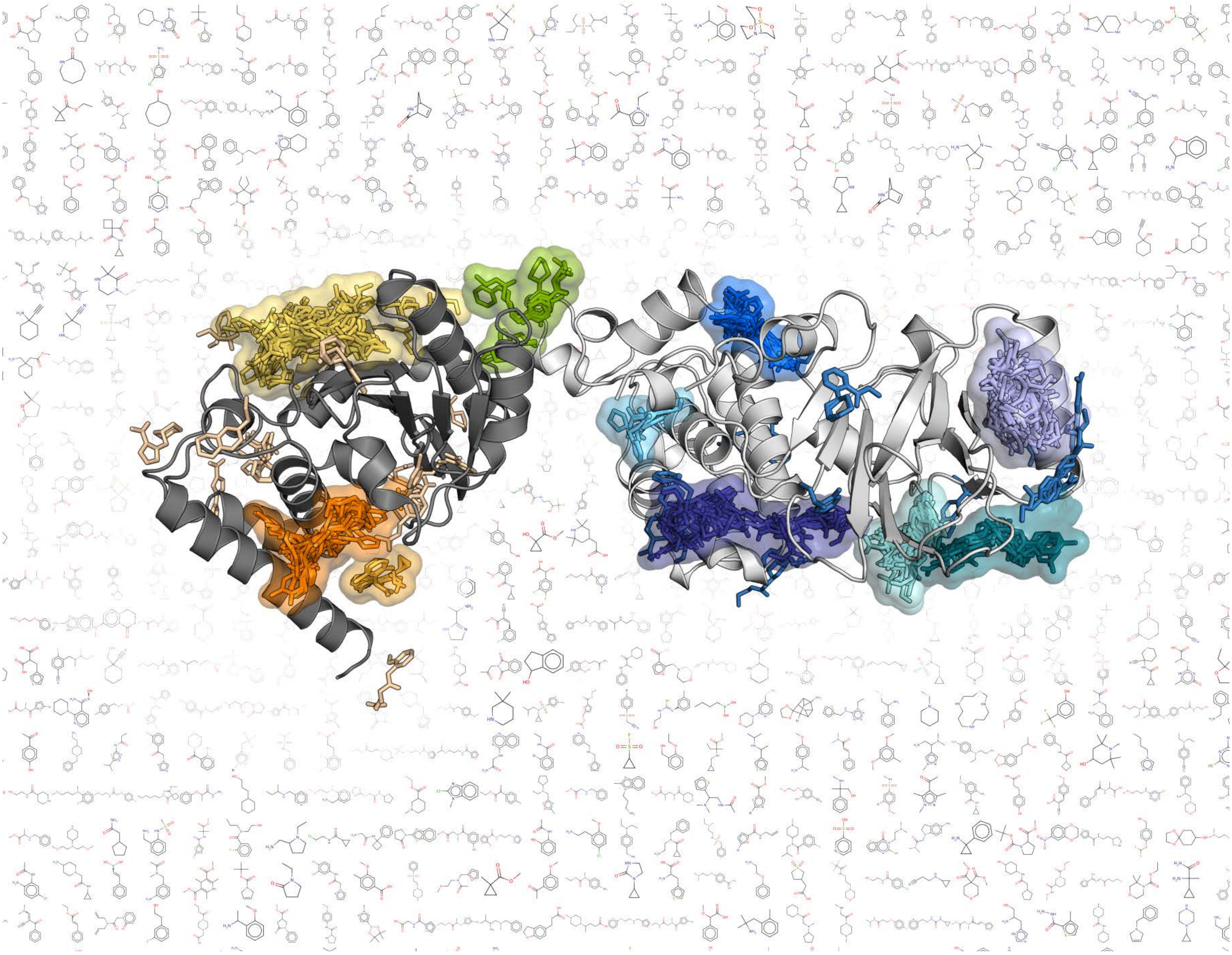
HEALTH AND MEDICINE

Increased DNA polymerase activity link in ultra-mutated cancer

Researchers from the University of Nebraska Medical Center and Umeå University investigated the biochemical and structural consequences of DNA polymerase epsilon mutation relating to human ultra-mutated cancers, which have tumours with extremely high mutation rates. They tested for the mechanism of mutation rate with sustained fitness. With yeast DNA, the scientists used substitutions mimicking human V411L, S459F, F367S, L424V and D275V, common cancer-associated DNA polymerase epsilon variants. Results showed all variants increase DNA polymerase activity, which may be the key to their pathogenicity. Additionally, they uncovered that hyperactive variants efficiently synthesize DNA at low nucleotide (dNTP) concentrations, which could promote an increase in cancer cell survival in early stages of tumour formation and replication during metabolic stress. Structural changes in the yeast were analysed at BioMAX beamline at MAX IV. The study enhances knowledge of DNA replication in cancer, with the possibilities of improved patient survival and response to immunotherapy.

Publication

S. Barbari et al. Enhanced polymerase activity permits efficient synthesis by cancer-associated DNA polymerase ϵ variants at low dNTP levels. *Nucleic Acids Research*, Volume 50, Issue 14, 12 August 2022, Pages 8023–8040.
DOI: 10.1093/nar/gkac602



HEALTH AND MEDICINE

Among the crowd: Extensive crystallographic fragment screening for new drug targets

Scientists screened the complete F2X-Universal Library with more than 1000 fragments and took the benefit of EasyAccess Frame, BioMAX beamline, and the fragment screening data analysis app, FragMAXapp, to identify hundreds of starting points for new drug targets exploration. The Macromolecular Crystallography Group (HZB), Germany, team focused on protein-protein interaction (PPI) modulators by targeting spliceosomal protein-protein complex of Prp8 and Aar2. This is the first study of the magnitude conducted at MAX IV.

Prp8 is a highly conserved protein, also referred to as the heart of the spliceosome, and is involved in several PPIs. Aar2 is a shuttling factor for parts of the spliceosome. Therefore, by screening this complex, several known PPI sites can be targeted on the surface of both proteins. After identifying the starting points, scientists investigated the functions of different proteins and spliceosome arrangements in more detail. During the study, the group also observed inherent binding mode validation and the implications on putative PPI sites.

The study shows how powerful the MAX IV facility is in handling crystallographic fragment screening of such magnitude.

Publication

T. Barthel et al. Large-Scale Crystallographic Fragment Screening Expedites Compound Optimization and Identifies Putative Protein–Protein Interaction Sites. *Journal of Medicinal Chemistry* 2022 *65* (21), 14630-14641. DOI: 10.1021/acs.jmedchem.2c01165

HEALTH AND MEDICINE

Anisotropic nanogels: How environment changes particle shapes

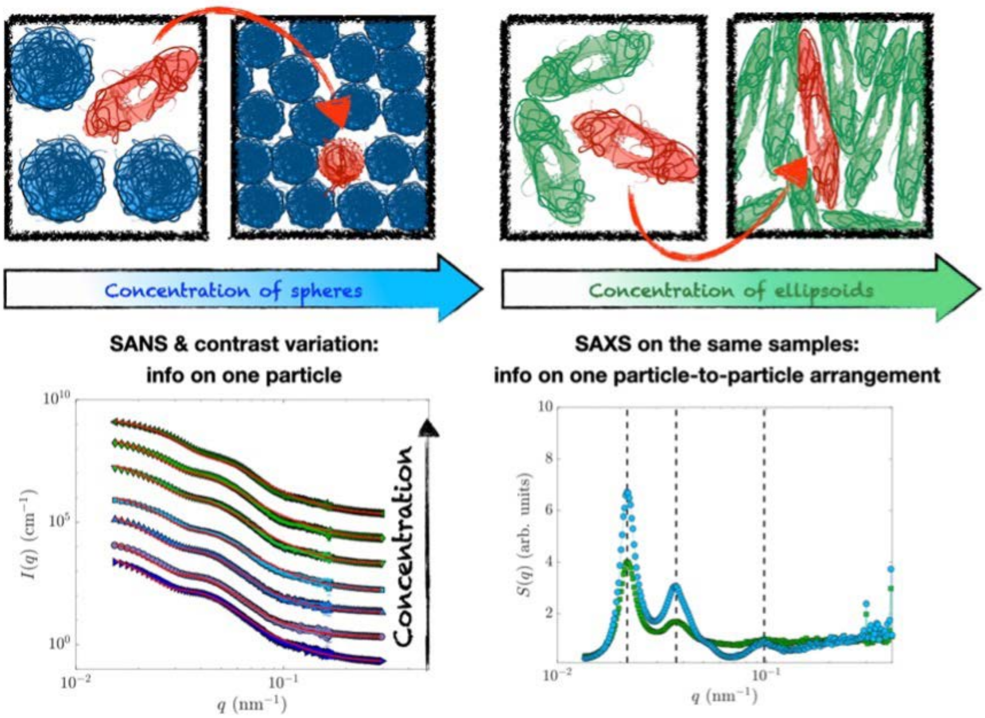
By combining small-angle neutron scattering and X-ray scattering at CoSAXS, RWTH Aachen-led researchers found that anisotropic ellipsoidal soft particles adjust and adapt their size and shape to the surrounding particles that make up the matrix they are embedded into. When spheres surround a soft ellipsoidal particle, they turn into a sphere. In contrast, if the soft elliposid is surrounded by other ellipsoidal particles, it enhances its anisotropy accordingly.

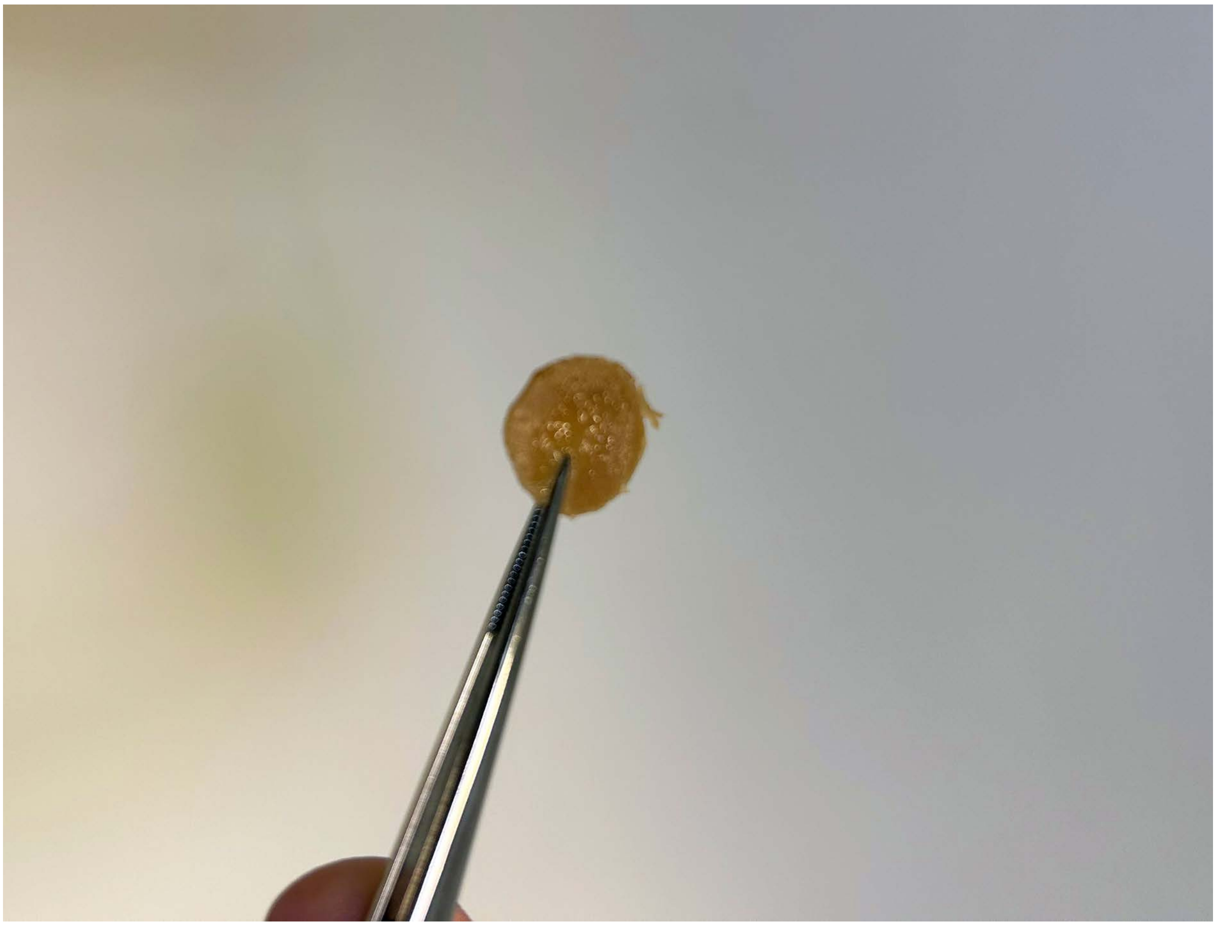
The techniques researchers used allowed them to see different parts of the samples, and their combination allowed them to gain information on the particles' individual shapes and how the particles were arranged in the sample. Monte Carlo simulations were also conducted to support the experiment data.

The effect of softness and anisotropy of individual particles is significant since it impacts how these particles are adsorbed at the interface. The soft ellipsoidal that researchers studied here can be used in the future for targeted drug delivery; therefore, understanding how they respond to crowding will allow them to conduct a rational design during formulation.

Publication

A. C. Nickel et al. Beyond simple self-healing: How anisotropic nanogels adapt their shape to their environment. *J. Chem. Phys*. 157, 194901 (2022);
DOI: 10.1063/5.0119527





HEALTH AND MEDICINE

Producing high-potential hydrogel from corn milling waste

Corn fibre, corn milling's abundant side streams, is rich in dietary fibres with potential antioxidant and prebiotic properties. Upgrading corn fibre into functional materials for biomedical and nutritional applications is a valuable opportunity to mitigate food waste and contribute to a circular food system. Researchers from KTH Food aim to upcycle this agricultural wastes and side streams into valuable food ingredients with health-promoting benefits.

In this experiment, researchers used enzymatic coupling, biochemical characterisation, in vitro cell viability, and oxidative stress performance assessment of the hydrogels in contact with human gut cells. They used CoSAXS beamline to understand the morphology and nanoscale structure of the polymeric networks that form the hydrogel. The experiments carried out at MAX IV were fundamental to controlling and understanding the structure of the hydrogels at the nanoscale and how it determines the properties and applicability of the hydrogels in advanced applications.

The experiment shows that the abundant food side stream (corn fibre) can be upgraded into a valuable hydrogel material for food and biomedical applications that could mitigate inflammatory processes. The hydrogels have excellent functional properties as biomaterials and can also reduce in vitro antioxidant processes in contact with gut cells subjected to oxidative stress.

Publication

S. Yilmaz-Turan et al. Hydrogels with protective effects against cellular oxidative stress via enzymatic crosslinking of feruloylated arabinoxylan from corn fibre. Green Chem., 2022, 24,9114.
DOI: 10.1039/d2gc03331c

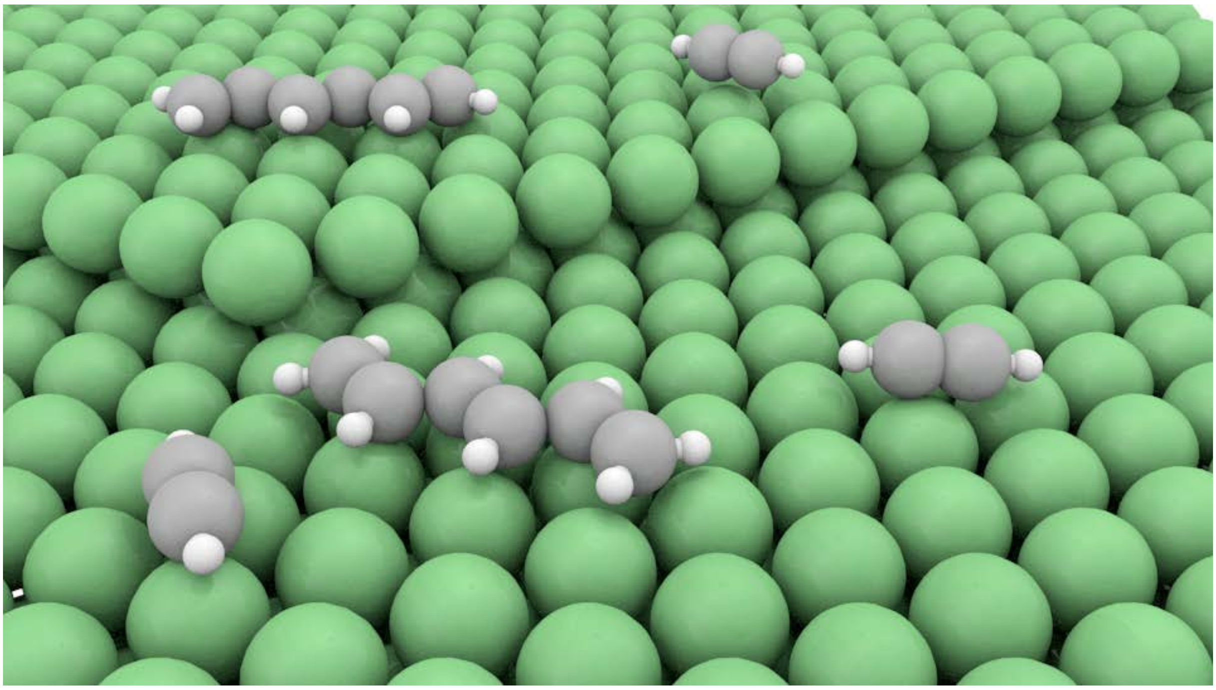
ENERGY AND FUELS

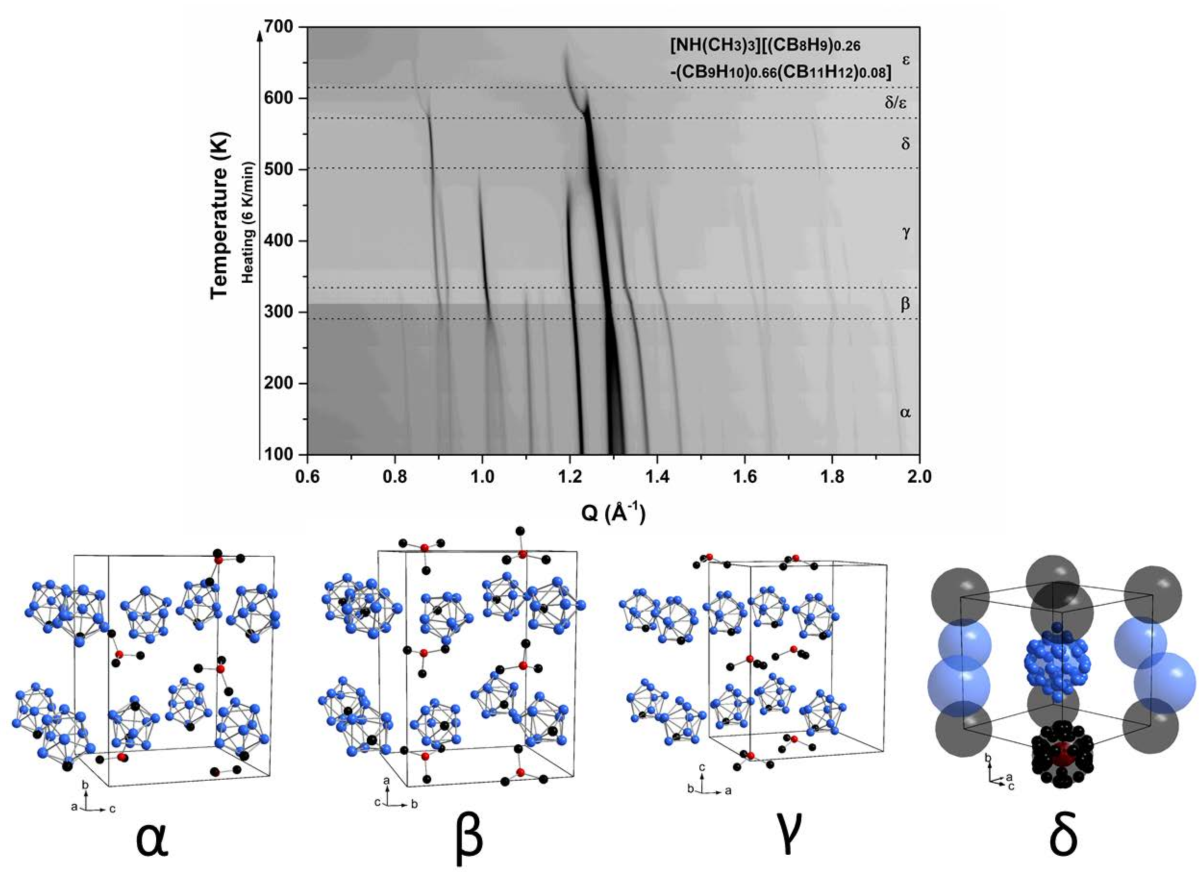
Charting growth of hydrocarbons chains for industrial processes

Small hydrocarbon molecules play a big role in the industrial production of present-day fuels and plastics. The mechanism of growth for hydrocarbon chains, however, is largely unknown. With X-ray photoelectron spectroscopy and *in situ* scanning tunneling microscopy, researchers from Austria, Italy and Germany analysed the formation of polyacetylene on a nickel model catalyst surface. The group found that active sites of hydrocarbon chains polymerization are the flat terrace sites for C-C coupling, not substrate step sites of the catalyst as previously thought and identified the intermediate species and reaction products. To test the growth process under comparable industrial conditions, the group used near-ambient pressure XPS (NAP-XPS) at SPECIES beamline at MAX IV. The study advances understanding of polymerization and reaction mechanisms of small hydrocarbons into long hydrocarbon chains for new or improved industrial processing.

Publication

Z. Zou et al. In Situ Observation of C–C Coupling and Step Poisoning During the Growth of Hydrocarbon Chains on Ni(111). *Angewandte Chemie-International Edition* 62 (2023) e202213295.
DOI: 10.1002/anie.202213295





ENERGY AND FUELS

Developing precursor material for future batteries

Interest has increased over the last decade for research on boron-hydrogen based materials as electrolytes for all-solid-state batteries. Among these are metal *closo*-boranes, which exhibit high thermal and electrochemical stability, weak cation-anion interaction, and fast ionic conductivity. Scientists at Aarhus University report a new synthesis method for a novel mixed closo-carborane compound, trimethylammonium $[NH(CH_3)_3][CB_xH_y]$, a potential precursor material for a variety of electrolytes by simple cationic exchange. They investigated the crystal structures and polymorphism of both $[NH(CH_3)_3][CB_9H_{10}]$ and the isostructural compounds formed with a solid solution of $[CB_8H_9]$, $[CB_9H_{10}]^-$ and $[CB_{11}H_{12}]^-$ anions. Thermal stability was also evaluated, with $[CB_8H_9]^-$ found to be least stable. Measurements of *in situ* temperature variated powder X-ray diffraction were made at DanMAX beamline at MAX IV. The work defines new precursor materials for novel types of electrolytes and holds promise for the design of improved all-solid-state electrolytes for better batteries.

Publication

J. B. Grinderslev et al. Polymorphism and solid solutions of triethylammonium monocarboranes. *Dalton Transactions*, 2022, 51, 15806-15815. [IF: 4.569]
DOI: 10.1039/D2DT02513B

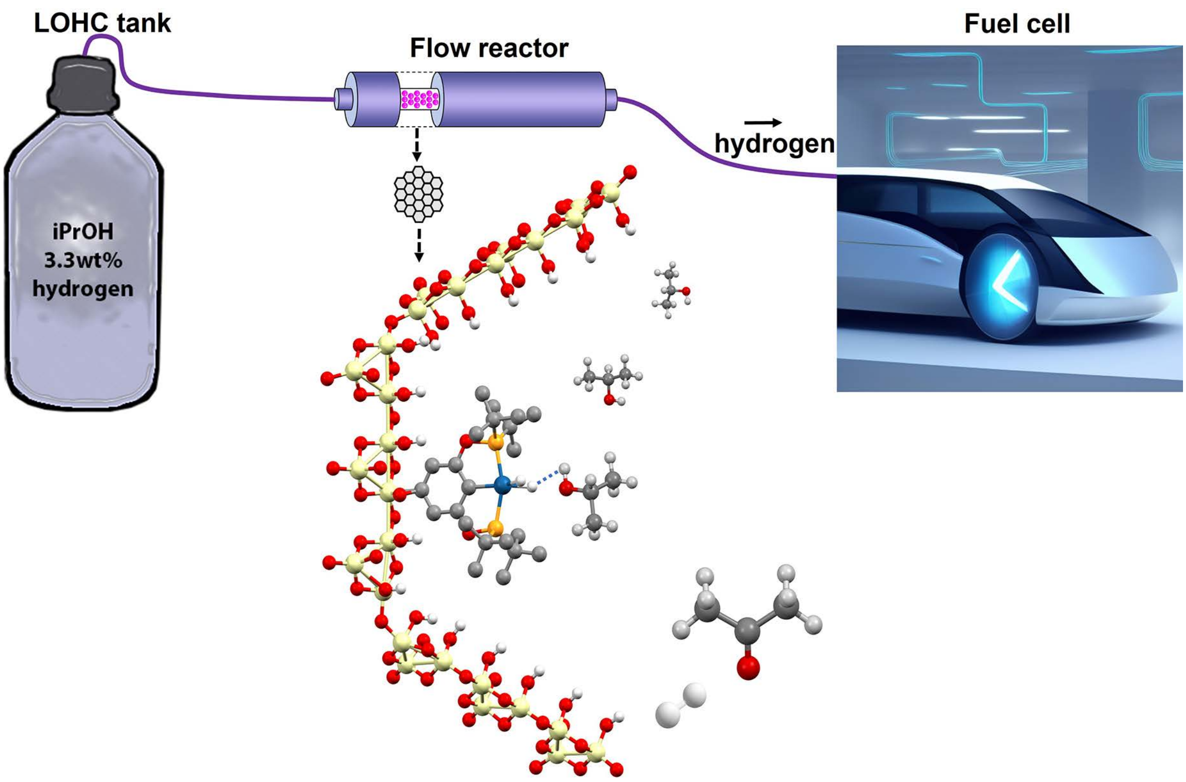
ENERGY AND FUELS

Heterogenized transition metal complexes for efficient hydrogen fuel production

Among the UN Sustainable Development Goals of 2030 is the use of clean energy. One burgeoning concept is the so-called hydrogen economy, where hydrogen is used as an energy carrier, which feeds fuel cells for on-demand electricity generation. A key challenge is solving hydrogen storage limitations. A potential solution are liquid organic hydrogen carriers (LOHC), which generate hydrogen *in situ* and are noteworthy for their safety and compatibility with existing infrastructure. Still, available methods of hydrogen extrusion from LOHCs remains too slow for on-board application. Scientists at Lund University explored the use of heterogenized transition metal complexes for dehydrogenation of an acyclic heteroatom-rich LOHC in a continuous flow reactor. Utilizing non-toxic substrate isopropanol, they achieved excellent selectivity and activity of the transition metal complexes with the high operational temperatures and stability of standard heterogeneous catalysts. The results translate to fast, efficient hydrogen production compatible with potential applications in fuel-cell powered vehicles. X-ray absorption near-edge structure (XANES) spectroscopy performed at MAX IV's Balder beamline provided insights into resting states and decomposition mechanisms of heterogenized catalysts. The work advances knowledge related to on-board hydrogen storage and implementation of the hydrogen economy.

Publication

A. Polukeev et al. Iridium-Catalyzed Dehydrogenation in a Continuous Flow Reactor for Practical On-Board Hydrogen Generation From Liquid Organic Hydrogen Carriers. *Chemsuschem*. DOI: 10.1002/cssc.202200085



ENERGY AND FUELS

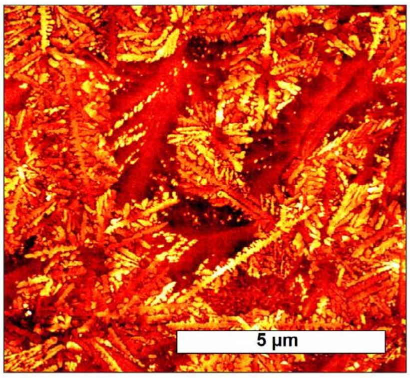
Towards stable electrodes for artificial photosynthesis

Photoelectrochemical reactors can convert solar energy into chemical form and store it as hydrocarbons, using water and carbon dioxide as inputs. Highly efficient solar-to-hydrocarbon conversion can be realised in a photoelectrochemical reactor based on solar cell materials. However, these materials are not chemically stable in the reactor without protective coating. Insufficient long-term stability is the biggest challenge for the technology.

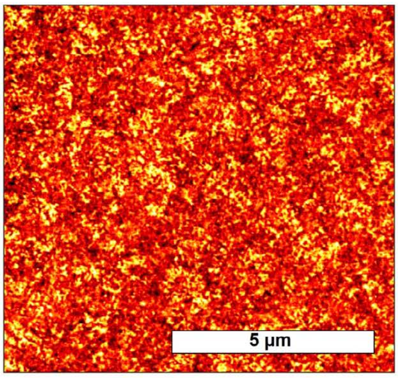
A group from Tampere University, Finland, has used the FinEstBeAMS beamline to study the properties of TiO_2 thin film coatings grown by atomic layer deposition as a function of growth and post-deposition annealing parameters. The experiments show that amorphous TiO_2 thin films containing low amount of precursor traces and high degree of Ti^{3+} defects form a nanocrystalline rutile coating during vacuum annealing. Unlike common microcrystalline anatase-TiO_2 coatings, the nanocrystalline rutile coating has exceptionally high resistance to pinhole mediated corrosion under alkaline conditions. This makes the coating a promising candidate for providing long-term stability to photoelectrodes in photoelectrochemical (PEC) devices.

Publication

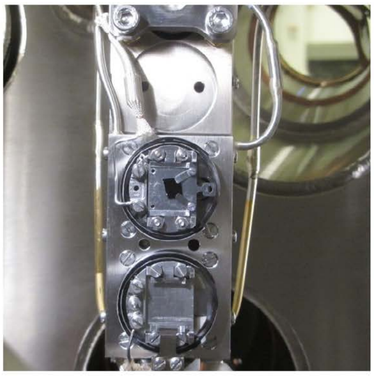
L. Palmolahti et al. Pinhole-resistant nanocrystalline rutile TiO_2 photoelectrode coatings. *Acta Materialia* 239 (2022). DOI: 10.1016/j.actamat.2022.118257

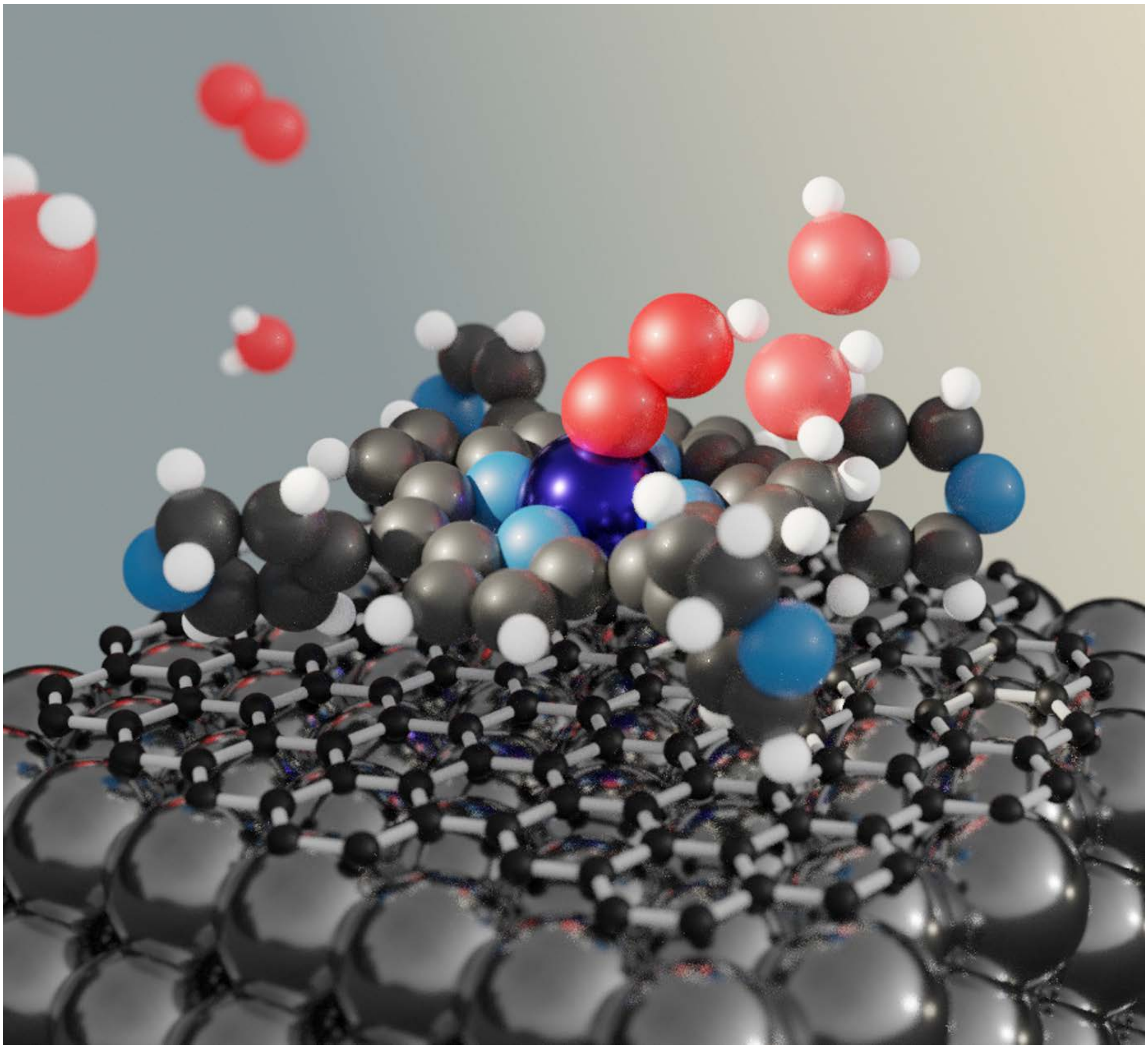


Anatase



Nanorutile





ENERGY AND FUELS

First report on stable O_2H to build ground for future rechargeable batteries

The first *in situ* observation of the most important Oxygen Reduction Reaction (ORR) intermediate, the hydroperoxyl molecule (O_2H) in a complex with water, was reported by the IR-Vis SFG laser spectroscopy group at the University of Trieste. The elusive intermediate was stabilised thanks to molecular water clusters that form at single Co atoms trapped in the porphyrin's tetra-coordination environment. The interplay between charge transfer, dipole and H-bonding, and water solvation determines the hydroperoxyl-water complex stability and the Co-OOH bonding geometry.

The experiment combined several experimental and theoretical tools, such as IR-Vis Sum-Frequency Generation laser spectroscopy, DFT calculations, and AP-XPS at the HIPPIE beamline.

This fundamental comprehension will contribute towards ORR pathways selectivity engineered control and the design of novel (bi-)functional metalorganic 2D materials as best electrodes for future rechargeable batteries.

Publication

F. Armillotta et al. Single Metal Atom Catalysts and ORR: H-Bonding, Solvation, and the Elusive Hydroperoxyl Intermediate. *ACS Catalysis* 2022 12 (13), 7950-7959.
DOI: 10.1021/acscatal.2c02029

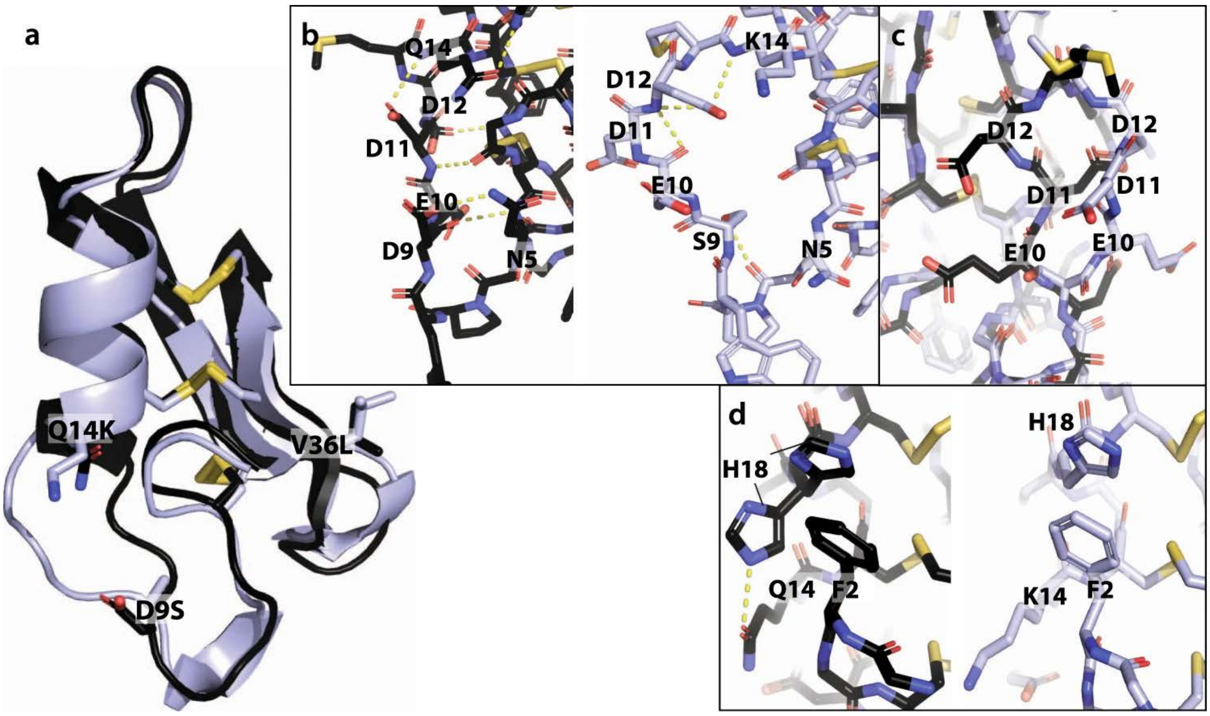
STRUCTURAL BIOLOGY

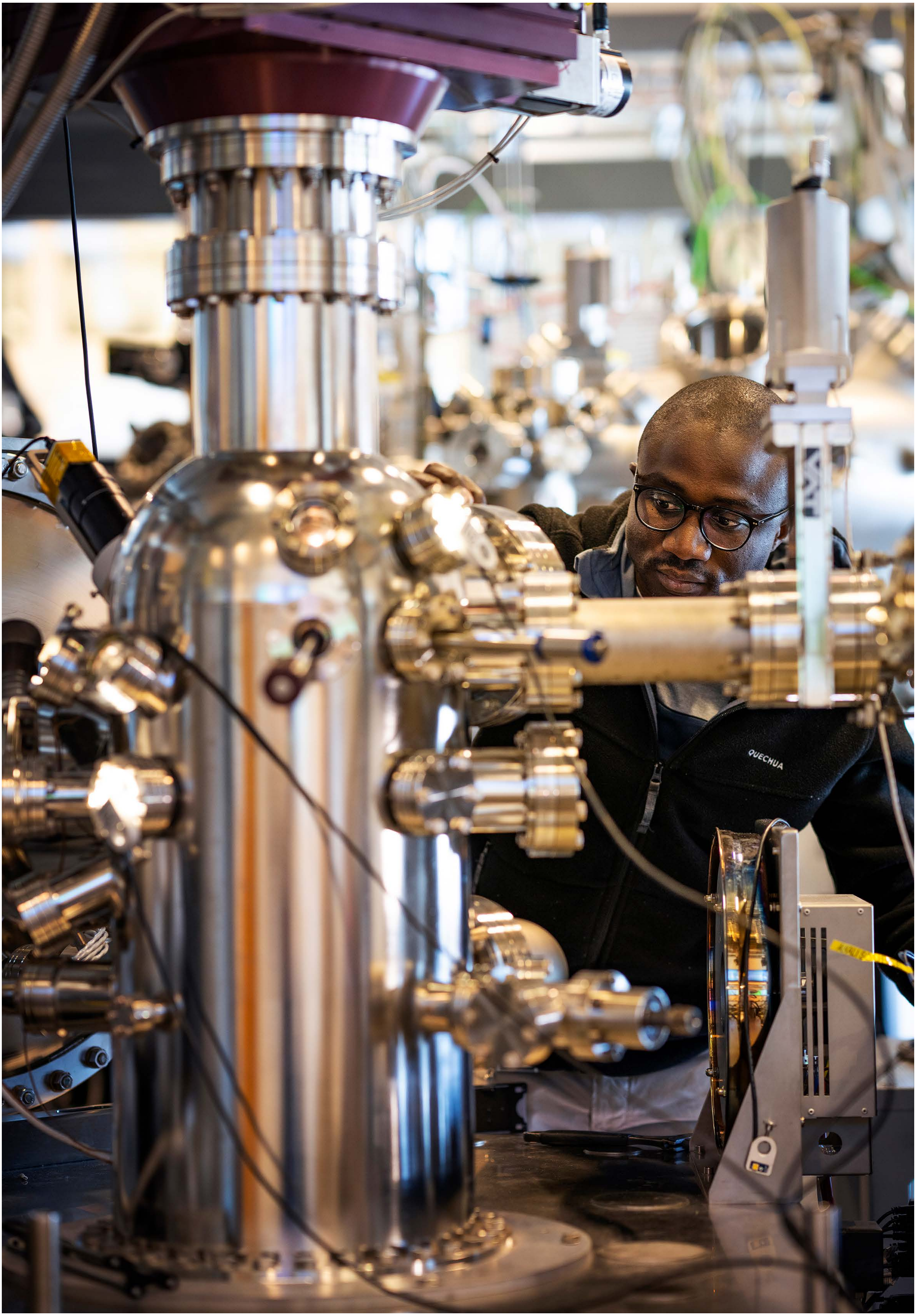
Protein self-assembly of anti-microbial peptide for biomaterials

An international team of scientists characterised the mutant, anti-microbial peptide plectasin (AMP: PP142) and its ability to self-assemble into helical non-amyloid fibrils. Results revealed that fibril structural formation, including a reversibility with lowered pH and stoichiometric effects with varied protein concentration and pH. They found the mutant variant gains anti-microbial potency against *Staphylococci* with the introduction of mutations. Structural data of PP142 was obtained at MAX IV's BioMAX beamline. The peptide plectasin is a favoured research entity for its low toxicity, high serum stability and long in vivo half-life, and is considered a likely drug candidate against bacterial resistance. The study furthers knowledge of protein self-assembly and potentially, the design of new functional biological materials and AMPs.

Publication

C. Pohl et al. pH- and concentration-dependent supramolecular assembly of a fungal defensin plectasin variant into helical non-amyloid fibrils. *Nature Communications* (2022) 13:3162.
DOI: 10.1038/s41467-022-30462-w




QUECHUA

ForMAX – a new beamline for multiscale and multimodal structural characterization

MAX IV opened its fifteenth beamline for user operation in winter 2022. ForMAX provides multiscale and multimodal structural characterization from nanometres to millimetres length scales by combining small- and wide-angle X-ray scattering (SWAXS) with full-field microtomographic imaging (µCT). The beamline is specially designed for the development of new materials from renewable forest resources, but also very well suited for studies within, for example, food or life sciences.

Many natural and synthetic materials are hierarchic, meaning that they show important structure at many different length scales that govern the material properties. Wood is an archetypical example (shown in Figure 1), cellulose bundles into fibrils, in turn forming ordered nanostructures in a matrix of other biopolymers, that form the building blocks of the cell walls, in turn making up the cellular structure. It is this ordering of cellulose at different length scales that governs the mechanical properties of wood. To improve our understanding of the structure-function relationship in hierarchical materials requires the means to probe their multiscale structure. Moreover, one need sufficient temporal resolution to study how the structure evolves in situ while processing the material or exerting an external stimulus.

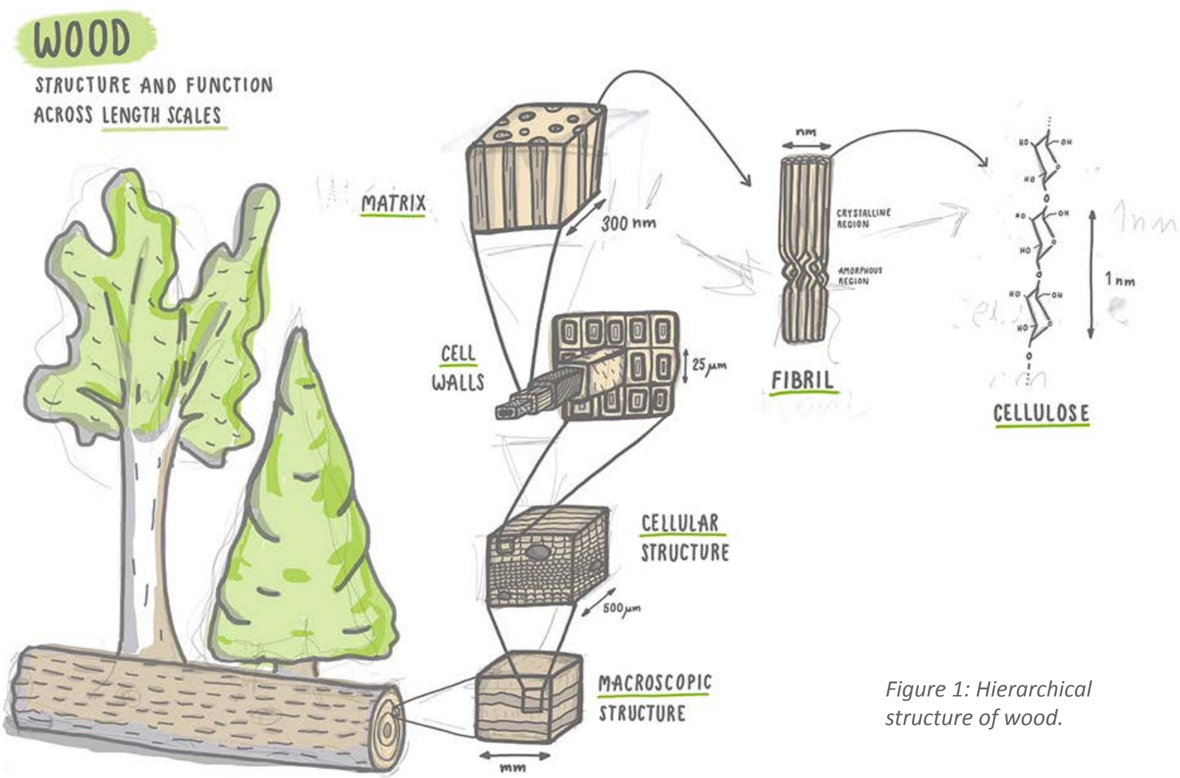


Figure 1: Hierarchical structure of wood.

More than 70 % of Sweden's land area is forest, with a majority of it cultivated. As a consequence, forestry has been a cornerstone of the Swedish economy for a century. Currently, there is significant interest within both academia and industry to develop new, sustainable usage of renewable forest resources. One example of such collaborative effort is Treesearch (www.treesearch.se), a research platform with almost twenty academic and commercial partners that collaborate on the development of new materials and specialty chemicals from forest resources.

ForMAX is the result of an effort to support the development of new materials from the forest. The construction of the beamline was funded by the Knut and Alice Wallenberg Foundation, whereas the operation costs for ten years are covered by Swedish industry via Treesearch. With this investment, 50 % of user time at ForMAX is reserved for Treesearch members, whilst the other 50 % is available to general users.

Multiscale and multimodal structural characterization

ForMAX beamline provides multiscale structural characterization of materials from nanometres to millimetres length scales. Moreover, the beamline enables in situ studies of real, heterogeneous materials during processing or external stimulus. ForMAX combines two complementary techniques into a single instrument:

- Structural features on the micrometre to millimetre length scales are probed by full-field microtomographic imaging (μCT). This technique provides a 3D image of the density distribution within the sample, with a sub-second temporal resolution.
- Nanoscopic structural features, in turn, are probed by small- and wide-angle X-ray scattering (SWAXS). SWAXS probes size, shape, and orientation of nanoscale structural features within the sample, with a temporal resolution in the millisecond or tens of millisecond range depending on the sample. ForMAX is particularly well suited for mapping nanoscale structural features in heterogeneous materials by so-called scanning SWAXS imaging, by scanning the sample through a focused X-ray beam and using the local SWAXS signal as contrast for imaging.

Both of these techniques are applicable to a wide range of materials and are relatively flexible with respect to sample preparation and sample environments.

ForMAX beamline

ForMAX beamline provides μCT and SWAXS in the 8–25 keV energy range. The beamline is equipped with two monochromators, a standard crystal monochromator and a "high-flux" multilayer monochromator for imaging experiments requiring high temporal resolution, providing a flux in the order of 10^{13}–10^{14} photons /second. The X-ray beam size at the sample position can be varied in the range from 15×50 μm^2 to 1.3×1.5 mm^2, depending on the monochromator. Secondary optics will allow further focusing down to approximately 1×1 μm^2 and expanding the beam to about 5×5 mm^2.

The combination of μCT and SAXS poses technical challenges. In μCT one monitors the transmitted X-ray beam directly downstream of the sample, i.e., at zero scattering angle, whilst in SAXS one measures X-ray scattering at very small angles. As a result, the tomography microscope blocks the SAXS signal. The approach at ForMAX is to mount the microscope on a motorized detector gantry (shown in Figure 2), allowing easy movement of the microscope in and out of the X-ray beam path. This enables users at ForMAX to collect μCT and SAXS data in a sequential manner.

SWAXS mapping experiments of fibrous materials, such as cellulose-based materials, pose another technical challenge. On the one hand, diffraction from fibrils, fibres or filaments is anisotropic, so in mapping experiments one needs

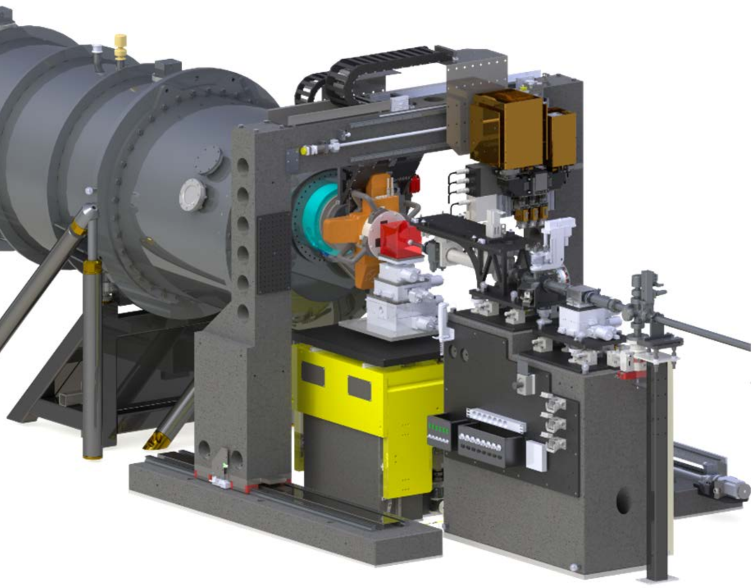


Figure 2: The end station at ForMAX. The main components include the yellow experimental table, where users build their experimental setup, the detector gantry onto which the tomography microscope and WAXS detectors are mounted, allowing easy movement of these in and out of the beam, and the evacuated flight tube that hosts the SAXS detector.

to collect WAXS data in all directions of the scattering plane. On the other hand, one needs to collect SAXS data simultaneously. The solution at ForMAX is a custom WAXS detector with a hole in the centre that allows users to collect WAXS data while the SAXS signal simultaneously passing through onto a SAXS detector further downstream.

In addition, there are several ongoing, community-driven projects to develop dedicated sample environments for ForMAX. These include, for example, load devices with integrated humidity control and a setup for combined rheology and SWAXS experiments.

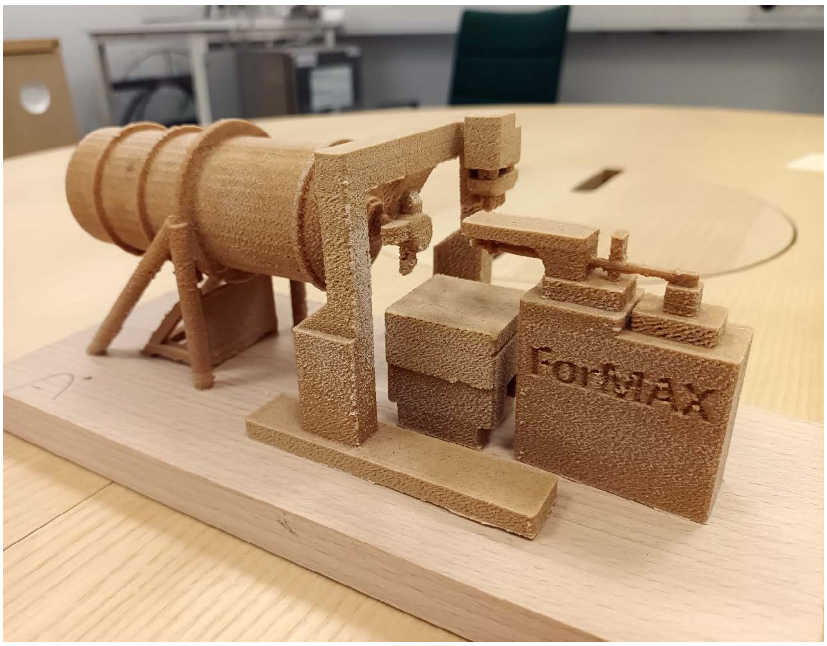


Figure 3: 3D printed impression of ForMAX beamline.

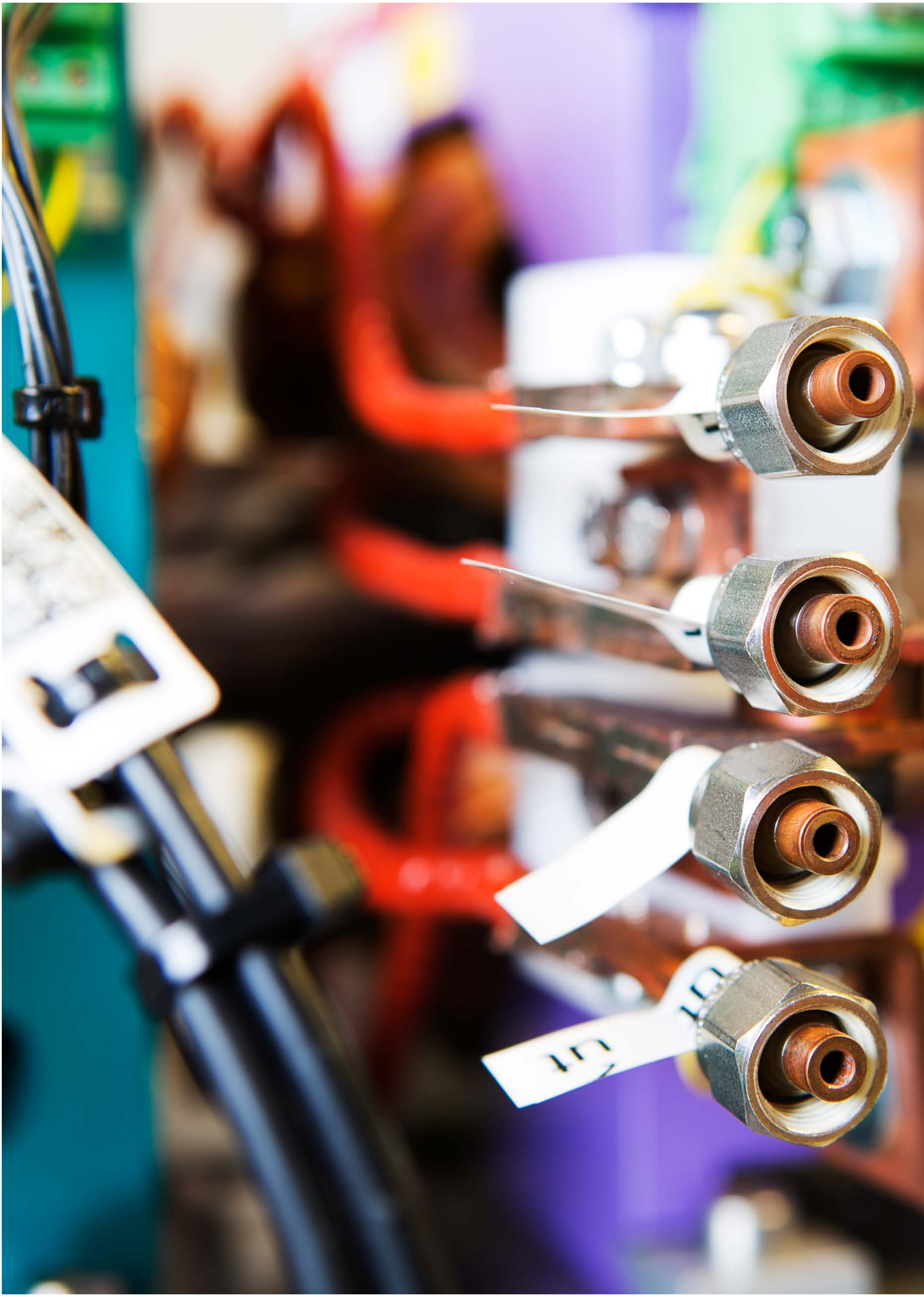
Future outlook

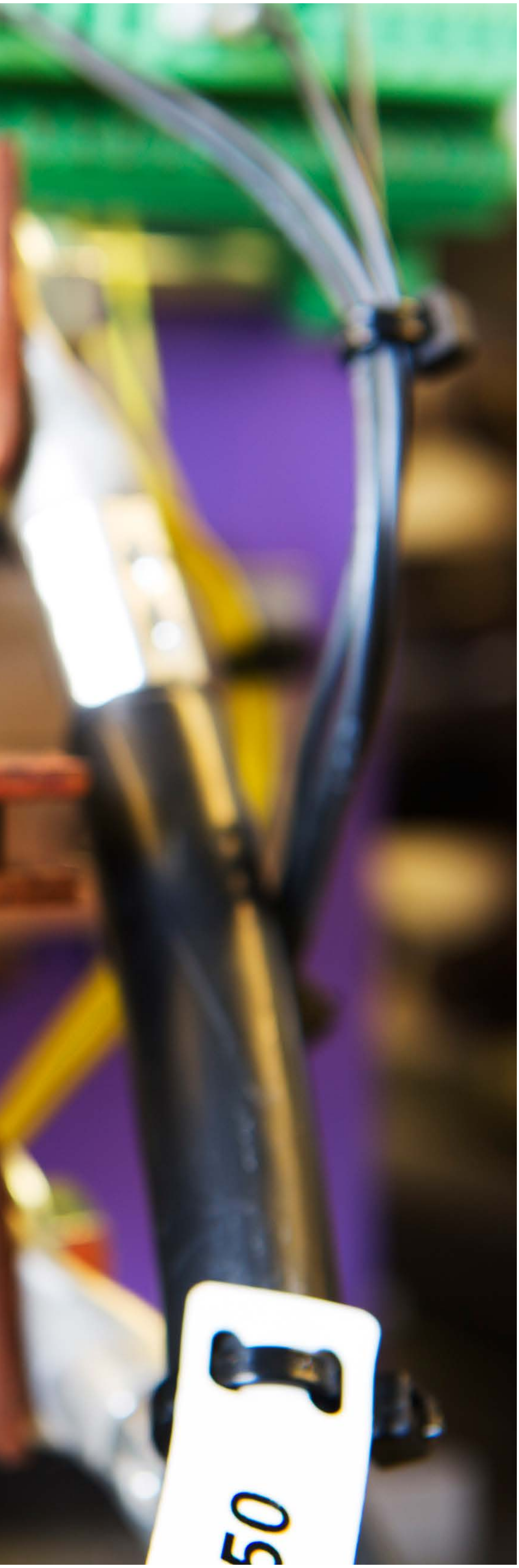
Although ForMAX is still in its early days of user operation, it has already shown promise for both academic and industrial research. Early examples of research on novel wood-based materials include the assembly of nanocellulose into fibres with extraordinary mechanical properties, the mechanical properties of cellulose-based foams, and wicking in sustainable paper straws. We look forward to interesting research along these lines in the future.

The multiscale and multimodal structural characterization provided by ForMAX beamline is also valuable for samples beyond novel, forest-based materials. Early examples range from energy materials to bone implants, highlighting the breadth of research that we can foresee at ForMAX.

Funding

Funding for the construction of ForMAX beamline was provided by the Knut and Alice Wallenberg Foundation, and the operational costs for ten years are covered by Swedish industry via Treesearch – a national research platform for the development of new materials and specialty chemicals from the forest. Access is provided to both Treesearch members and general users through Proposal calls.





Accelerator Operations and Development

Accelerator Operations in 2022

The year 2022 was marked by almost full recovery in delivered beam hours compared to the previous year, which was severely affected by the COVID-19 pandemic. The recovery was not yet at the levels of 2020, when accelerator operations achieved the international golden standard of roughly 5000 hours of delivery. This was partly due to remaining side effects of the pandemic, in particular, the installation of two new beamline insertion devices in the 3 GeV ring, which was postponed from summer 2021 to winter 2022. Moreover, additional time in summer 2022 shutdown was planned to allow for an investigation of potentially serious arcing in the linac RF system. Finally, the last operations week of the year in the 3 GeV ring converted to a maintenance week, originally upon request from two beamlines who needed to replace leaking fixed masks in their front-ends and later used to remove a leaking harmonic cavity. The resulting scheduled beamline hours for 2022 were 4464, 4848, and 4272 for the 3 GeV ring, 1.5 GeV ring, and Short Pulse Facility (SPF), respectively. These figures fall short of the planned goals for the year by roughly 20% for the storage rings and about 30% for the Short Pulse Facility. The evolution of scheduled beamline hours (Figure 1) is charted since the start of User Operation in 2017.

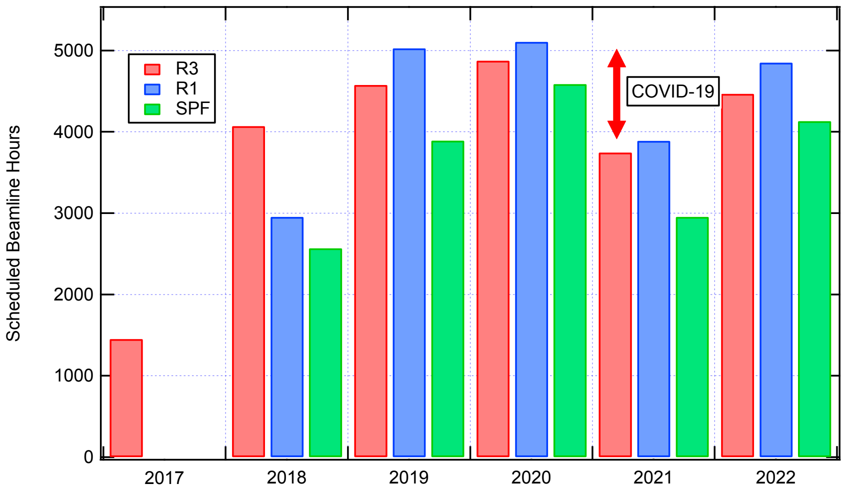
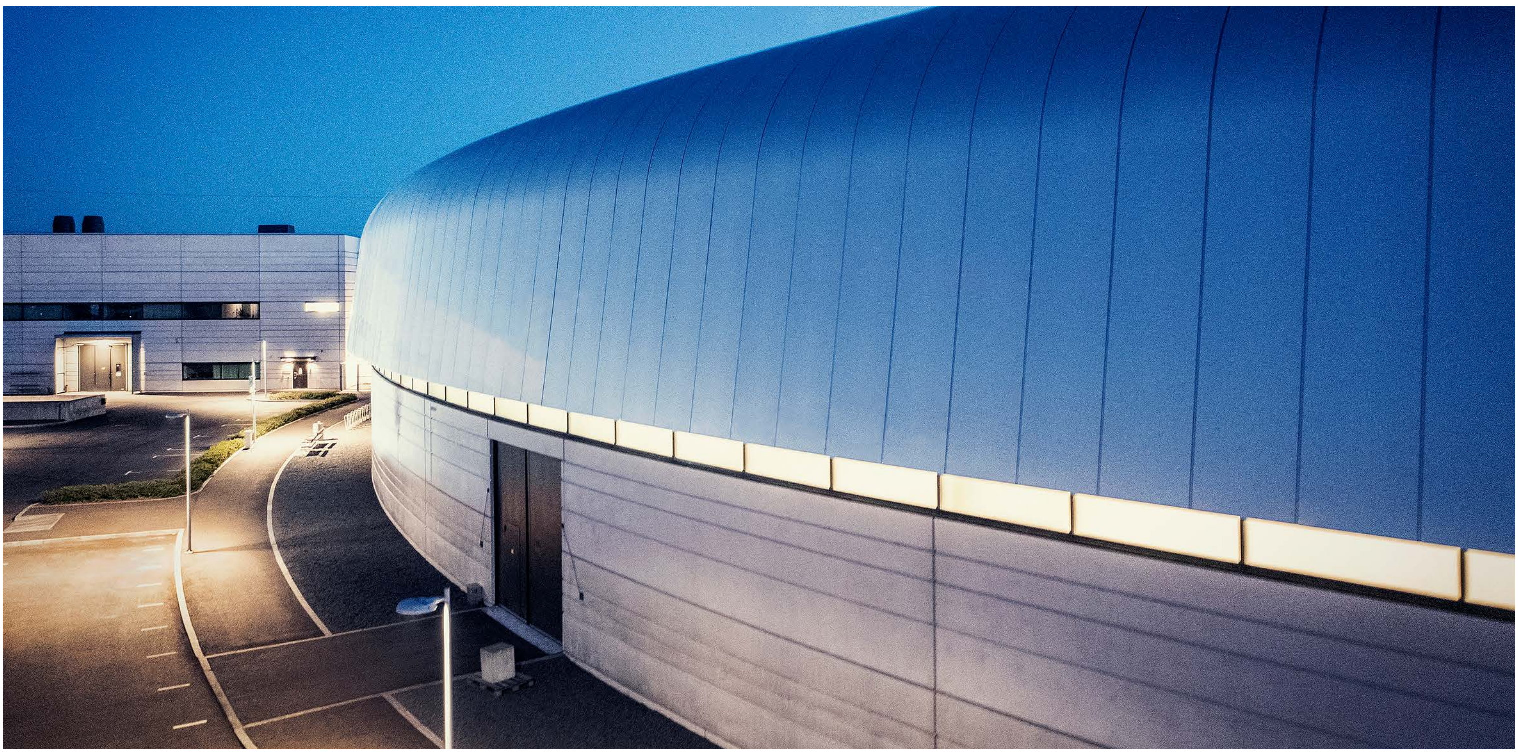


Figure 1: Number of scheduled beamline hours per year since the start of User Operations in 2017. By 2020, all accelerators were approaching 5000 hours. The significant reduction in 2021 is a direct result of the COVID-19 pandemic and 2022 display a welcome recovery toward the 2020 levels.



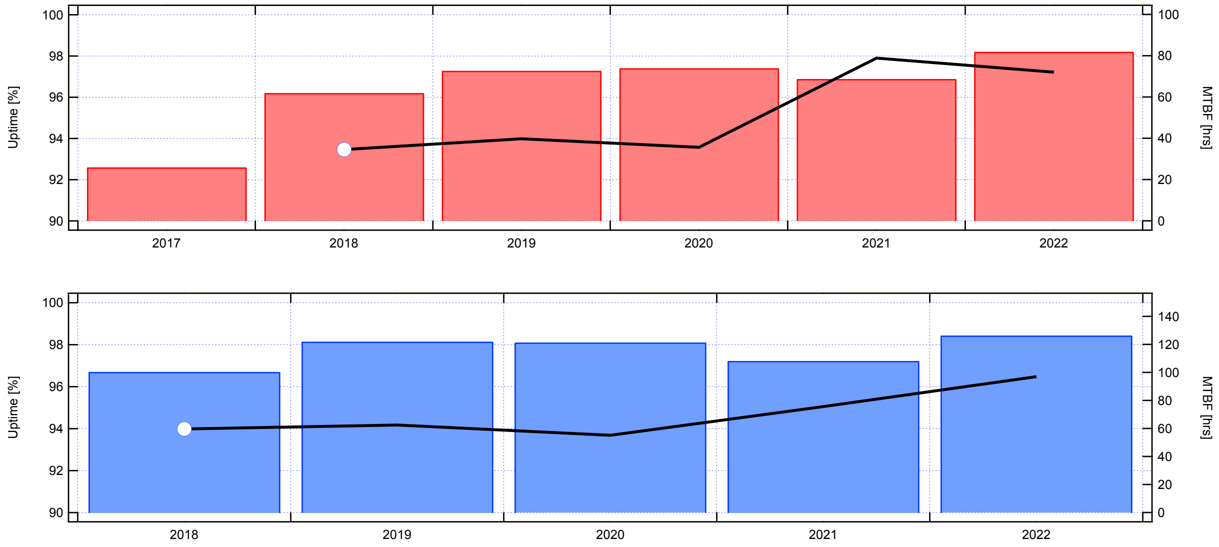


Figure 2: Uptime and Mean Time Between Failures (MTBF) for the 3 GeV (Red) and 1.5 GeV (Blue) storage rings since the start of User Operations.

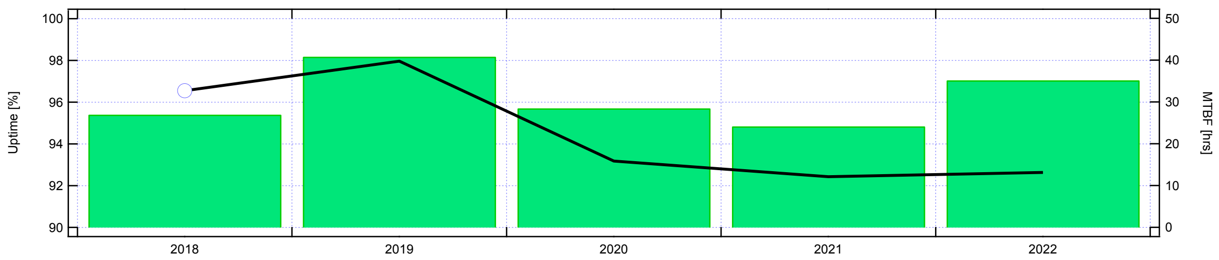


Figure 3: Uptime and MTBF for the Short Pulse Facility.

The accelerator uptime in 2022 (Figure 3) was the greatest in both storage rings since the start of User Operations, achieving 98.2 % in the 3 GeV ring and 98.4% in the 1.5 GeV ring. The SPF (Figure 4) shows a much-improved uptime (97%) compared to the past two years, a welcome result of the linac reliability improvement programme launched in 2022.

Concurrently, the mean time between failures (MTBF) continued at excellent levels for the storage rings. The goal of 72 hours MTBF was met for the 3 GeV ring and exceeded for the 1.5 GeV ring, which achieved 97 hours. The MTBF for the SPF in 2021 (13 hours) was slightly better than the previous year, but still below our 24 hour goal. However, the programme launched in 2022 to automate the recovery of modulator/klystron trips provided a very significant reduction in the frequency of long-duration downtimes (Figure 4).

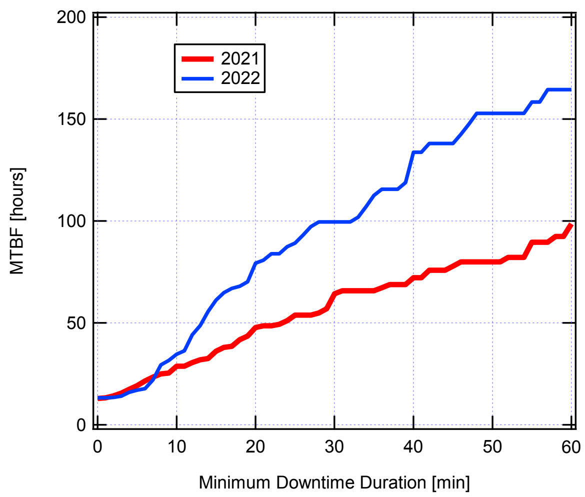


Figure 4: MTBF for the Short Pulse Facility as a function of downtime duration – only the downtimes with duration larger than a given value are counted. A significant reduction in long downtimes is clear from 2021 to 2022.

Accelerator Division Highlights

Transparent Injection into the 1.5 GeV ring

A multipole injection kicker (MIK) system was built, installed and successfully commissioned in the 1.5 GeV ring. This device follows the same design principles of a similar system already in operation in the 3 GeV ring for several years, and which has provided world-record low perturbations to the stored beam during top-ups. The original system was developed within the scope of collaboration with synchrotron SOLEIL in France and was based on a concept proposed at BESSY in Berlin. It consists of eight current-carrying wires arranged in such a geometry that a deflecting magnetic field is produced at the position of the injected beam, while the field seen by the stored beam is ideally zero.

Commissioning of the new system in the 1.5 GeV ring was extremely fast and once installation completed in late November, high efficiency injection was achieved within the first 15 minutes of operation (Figure 5). There was outstanding reduction in perturbations to the stored beam as compared to the previously used conventional injection system (Figure 6). While the dipole kicker remains a fallback, the MIK became the standard mechanism for injection during beam delivery to beamlines already the day following its first operation.

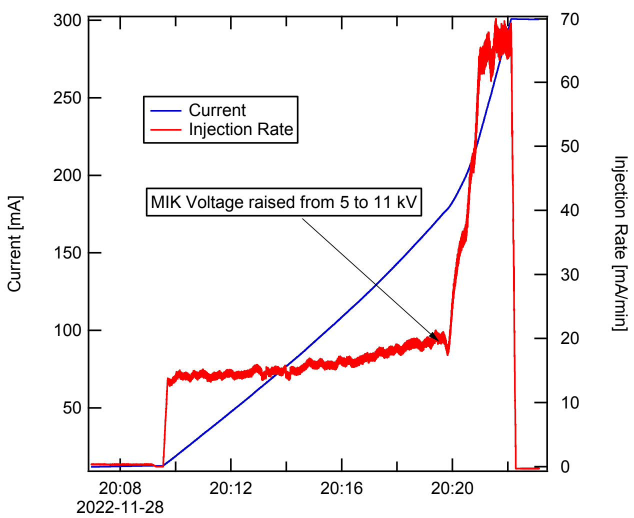


Figure 5: First injection realized with the Multipole Injection Kicker in the 1.5 GeV ring.

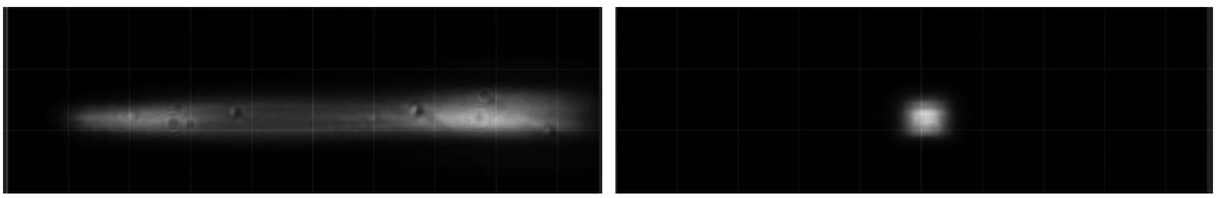
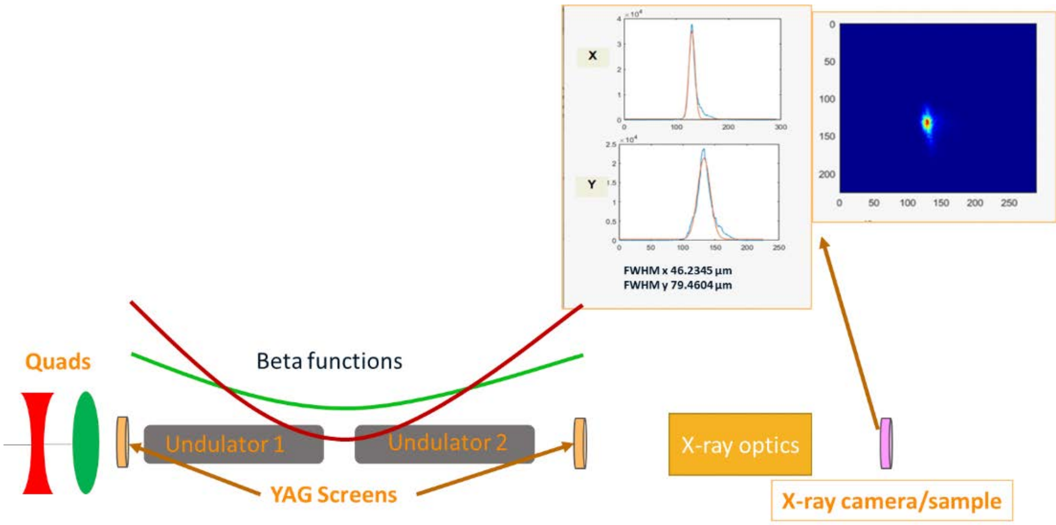


Figure 6: Transverse profile of the electron beam in the 1.5 GeV as observed at a diagnostic beamline ring during a top-up injection. (Left side) injection with the conventional injection system. (Right side) injection with the new Multipole Injection Kicker.

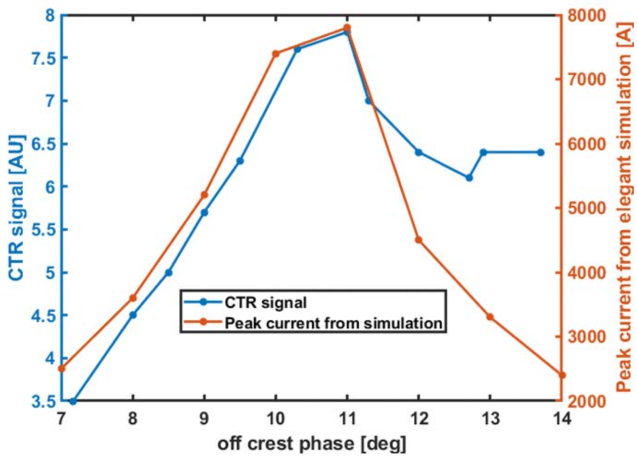
Understanding the effect of focussing in the undulator on the spot size at FemtoMAX

Recent improvements of the X-ray beam size are partially a result of the much-improved electron beam emittance. However, much effort has also been put into understanding the effect of beam focus inside the FemtoMAX beamline undulators. One can optimise the electron beam focus inside the undulators by minimising the X-ray spot size, as seen in the image from the FemtoMAX X-ray camera located at the sample point. Using this method, you see that a tighter focus in the centre of the undulator is favoured compared to a larger waist with a smaller average beam through the undulators. Further studies are planned to optimise the point of focus for the X-ray optics and electron beam.

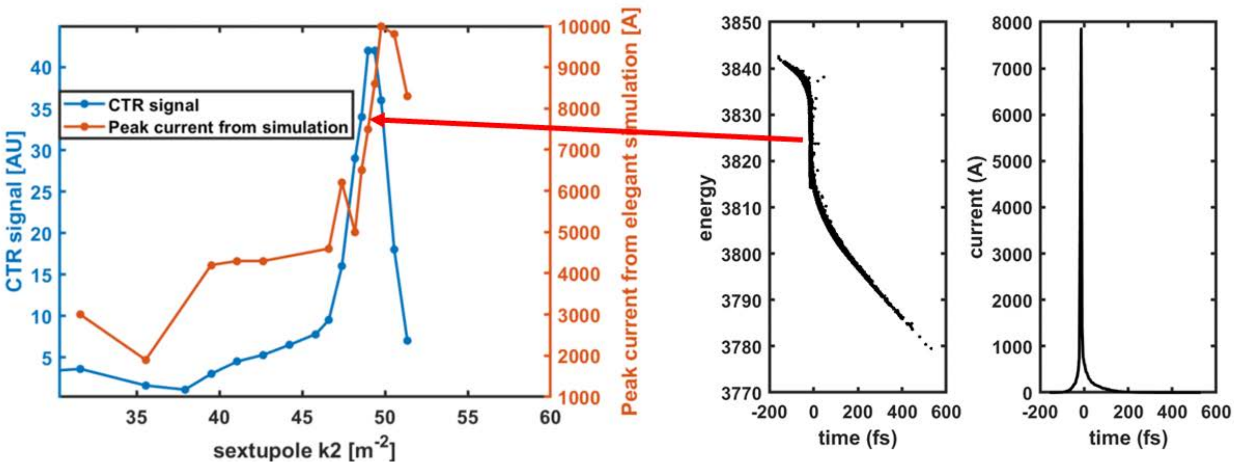


Bunch length investigations

With the addition and commissioning of a Coherent Transition Radiation (CTR) bunch length diagnostics tool, new studies of bunch length and sextupole influence on longitudinal phase space have been performed. For the first study the phase in the main linac (before BC2) was scanned around the maximum CTR signal. The peak intensity of the CTR signal was recorded (plotted blue line). This was found to agree with the elegant simulation of peak current (plotted orange line). According to the simulation, this phase sweep corresponds to a bunch length between 18 and 6 fs full width at half maximum (fwhm) parameter.



In another study, the sextupoles in BC1 were scanned, and the peak intensity of the CTR signal (plotted blue line) was recorded. The plotted orange line represents the peak current elegantly simulated. The simulation results were in good agreement with the peak. The beam was very short during this scan, with a bunch length ranging from 16 to 5 fs in the simulation. This study clearly confirms the unique linearization method of the MAX IV bunch compressors.



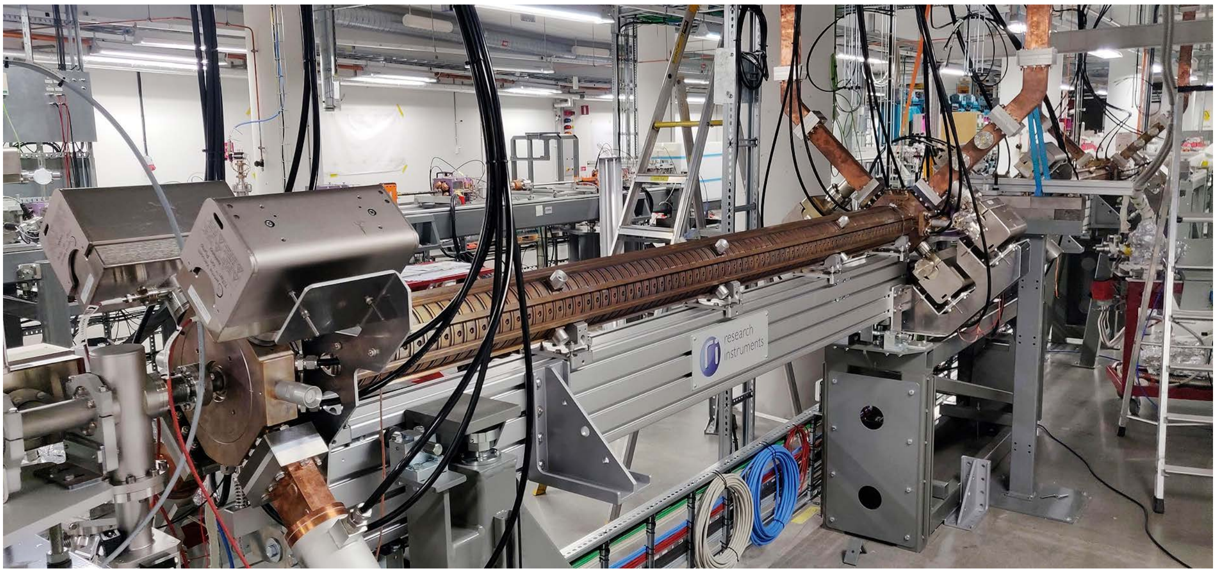


Figure 7: One of two transverse deflecting cavity structures installed in the Short Pulse Facility hall in autumn 2022.

Transverse Deflecting Cavity System at the Linear Accelerator

The suite of diagnostic systems used to characterize the properties of the electron beam produced by the MAX IV linear accelerator received a major boost with the assembly of a transverse deflecting cavity (TDC) line. The TDC will allow the measurement of various properties of the very short electron bunches (nominally down to 100 fs). In particular, the first direct measurement of the bunch lengths as well as knowledge of the variation of beam properties along the bunches (so called “slice parameters”) will be possible down to 1 fs time resolution. This will give an important insight into the complex dynamics of the short bunches with benefits, for example an understanding of the causes of emittance growth along the linear accelerator. In total, this will provide more robust delivery of short X-Ray pulses to FemtoMAX beamline and paves the way for the future production of even shorter pulses and operation of the linac as a driver to a soft X Ray free-electron laser.

The radiation safety permit will be obtained in the first quarter of the coming year. It will allow for RF conditioning of the structures and beam commissioning.

Transverse Resonant Island Buckets (TRIBs) in the 1.5 GeV ring

To satisfy the needs of scientific experiments requiring customized time structures of synchrotron radiation, the 1.5 GeV ring is today run in “single-bunch” mode for a few weeks every year. While this mode enables outstanding science, it also reduces total intensity to the detriment of other experiments, which are typically impossible under such circumstances. One scheme that could potentially allow the simultaneous use of the source by both communities thus increasing the availability of the source for time-resolved science, was pioneered at BESSY – transverse resonant island buckets (TRIBs). In this operation mode, the optics in the storage ring is brought close to a third-order resonance causing transverse phase space to split into a number of “islands” which constitute a spatially separated source of light. The population of these islands can be controlled on a bunch-by-bunch basis so that beamlines that select only light from the islands can effectively observe a light pulse repetition rate that is different from that observed by beamlines that select the light from the core of the beam. In this way, multi-bunch and single-bunch experiments can be run simultaneously.

In 2022, the scheme was successfully tested in experiments at the MAX IV 1.5 GeV ring at currents up to 450 mA. Tests in which beamlines could select the light from the islands and undulator gaps were moved while top-up injection carried out were also successful (Figure 8). These allowed us to start addressing a number of critical challenges, including storing enough charge in the islands and, in particular, maintaining stable transverse tunes while insertion device gaps and phases change.

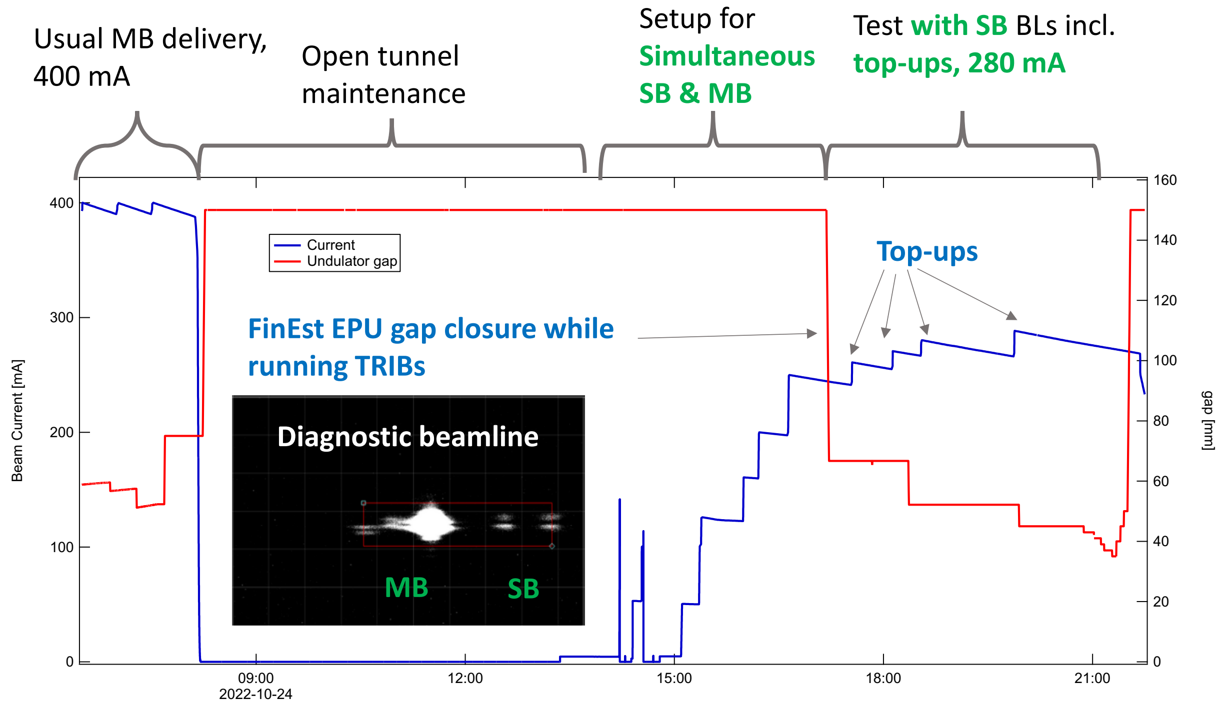


Figure 8: Beam current and FinEstBeAMS beamline undulator gap during tests with TRIBs. Once the undulator gap was closed while in TRIBs mode at 280 mA, light from the islands was selected at the beamline and top-ups were realized. The insert shows the transverse profile of the electron beam as observed at a diagnostic beamline with both the core (multi-bunch) beam and the island (single-bunch) beam.

1.5 GeV ring Fast Orbit Feedback coverage expanded

As in the 3 GeV ring, the 1.5 GeV electron beam is passively stable during delivery, and active orbit feedback with a high closed-loop bandwidth is still required to suppress perturbations arising from the movement of insertion device gaps and phases. Due to delays in power supply deliveries during the COVID pandemic the system testing prior to 2022 only included stabilization of the FinEstBeAMS long straight. Once the power supplies were received, commissioning of the full Fast Orbit Feedback system commenced during the autumn of 2022. The end result was the stabilization of the orbit in the remaining EPU straight sections, leaving only the FlexPES beamline straight. Performance in terms of attenuation is in line with the design.

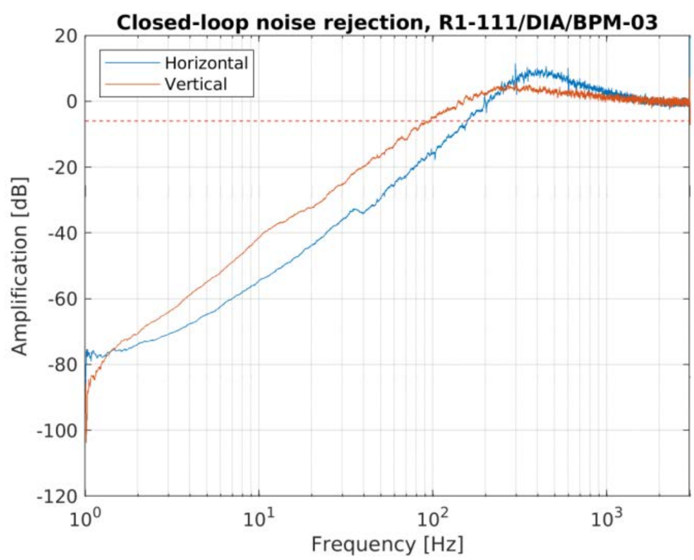
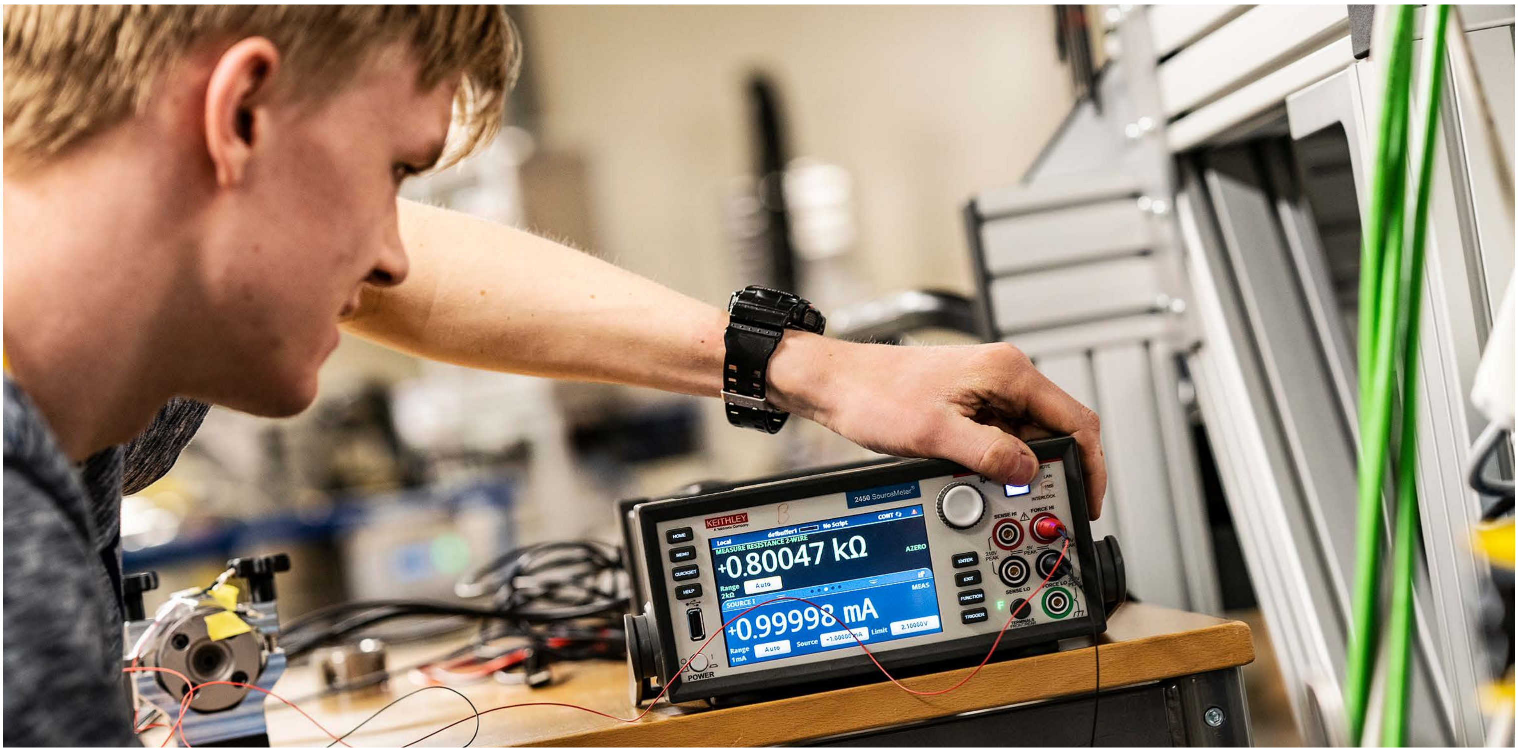


Figure 9: Orbit noise attenuation with the Fast Orbit Feedback active as measured in a BPM flanking the FinEstBeAMS beamline insertion device.



Active shims trialled in 1.5 GeV ring

During the winter shutdown 2021-2022 active shims were installed on the FinEstBeAMS beamline straight section as part of the effort to compensate for the effect the insertion device has on the ring magnet optics. After an initial verification of the hardware installation, the system went into beam commissioning during spring 2022. The active shim system consists of two thin sheet cables with eight leads each, attached to the top and bottom of the ID vacuum chamber. When powered and properly calibrated in a feed-forward loop they allow for the dynamic compensation of the integrated ID field components *in situ*, achieving a significantly higher degree of transparency.

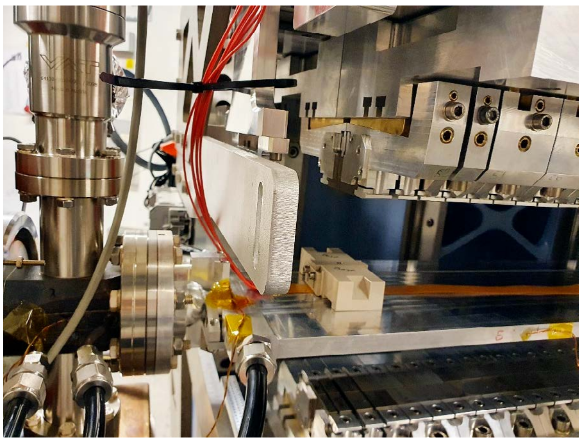


Figure 10: Active shims as installed in the FinEstBeAMS EPU. The upper sheet cable containing the shim leads is found on top of the vacuum chamber.

After calibrating the FinEstBeAMS active shims using empirical methods, based on cancelling the ID focusing and Amplitude-Dependent Tune Shift (ADTS) impact, they were integrated into operation during spring 2022. A further set of active shims has since also been installed for the insertion device at BLOCH beamline.

Lower Emittance Optics in the 3 GeV ring

Theoretical and experimental studies aimed at reducing the electron beam emittance and thereby increasing the photon beam brightness in the 3 GeV ring initiated in 2022. This is the first step toward the ultimate goal of achieving the diffraction limit at hard X-ray photon wavelengths. In 2022, stronger focussing optics were implemented that can be achieved without any hardware changes to the ring, namely maintaining the same magnets and power supplies, but simply readjusting their excitations. The optics are designed to provide a bare-lattice emittance of 270 pmrad, particularly a 20% reduction with respect to the 328 pmrad of the nominal lattice (Figure 9). Beam threading algorithms were implemented to realize the first few turns in the new lattice and capture of a low current could be successfully demonstrated. This allowed verification and correction of linear optics parameters such as the dispersion function and beta beats. Achieving enough dynamic aperture to allow for beam accumulation is yet to be demonstrated.

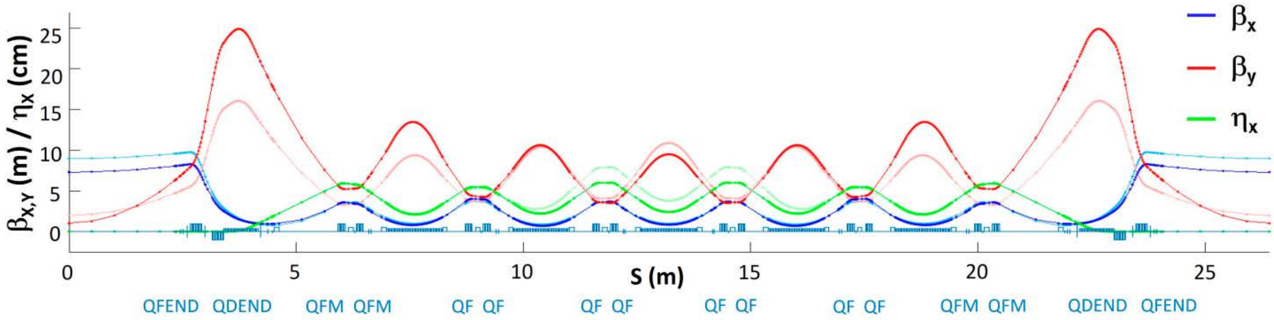


Figure 11: Twiss parameters along one achromat of the MAX IV 3 GeV ring. Light colours present lattice at 328 pmrad, dark colours (new lattice at 270 pmrad).

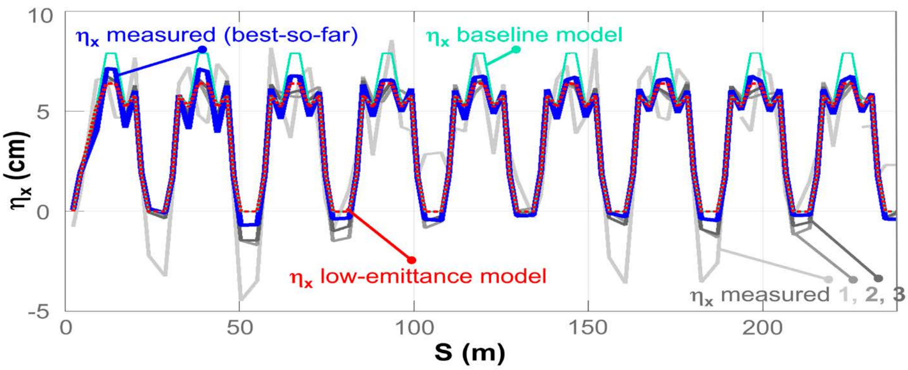
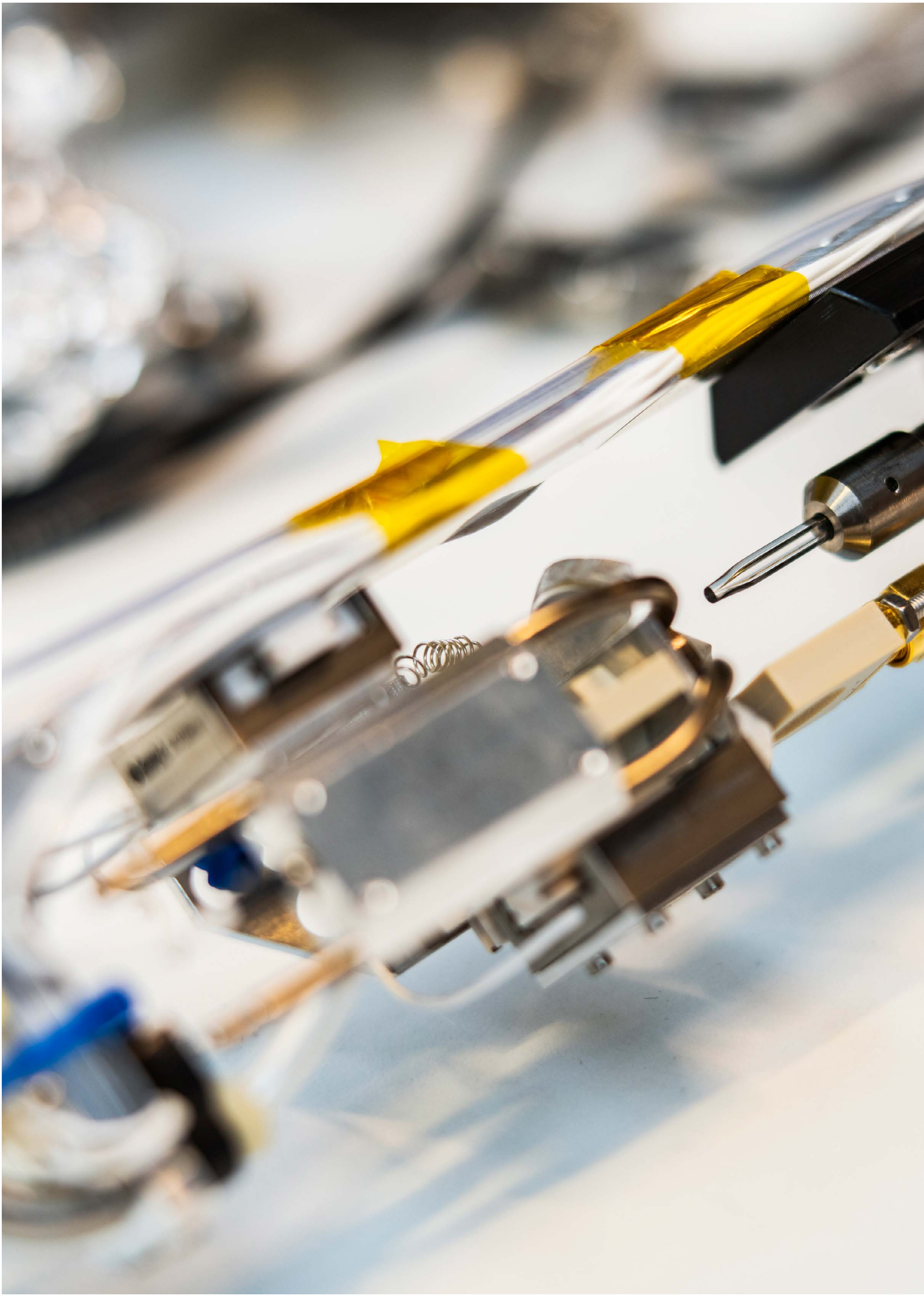


Figure 12: Dispersion function measurement for the new lower emittance lattice.

HarmonLIP 2022 Workshop

October 10-12, 2022, a workshop on the use of Harmonic Cavity Systems at fourth and future generation lightsources was organized at MAX IV. The workshop was sponsored by LEAPS (League of European Accelerator-based Photon Sources) and had 50 registered participants from 13 Institutions and 10 countries. This is the first in a series of events that result from the LEAPS internal project HarmonLIP initially proposed by MAX IV to the LEAPS Working Group 2 in autumn 2021. Hosting the event at MAX IV highlights the fact that even though harmonic RF systems have been successfully in use at third generation lightsources for many years, they were often added to those sources as part of upgrade programs while they are part of the baseline design of most fourth generation lightsources, particularly in the intermediate energy range of a few GeV where intrabeam scattering (IBS) is most severe. For future light sources aiming to achieve even lower emittances than the present suite of existing and planned sources, good control of the bunch length will be even more critical, and one may envisage the need to achieve considerably larger lengthening ratios than those achieved today (which vary from 2 to 5). Combining different harmonic systems may be a mechanism to provide such extremely long bunches, but its feasibility remains to be demonstrated experimentally.

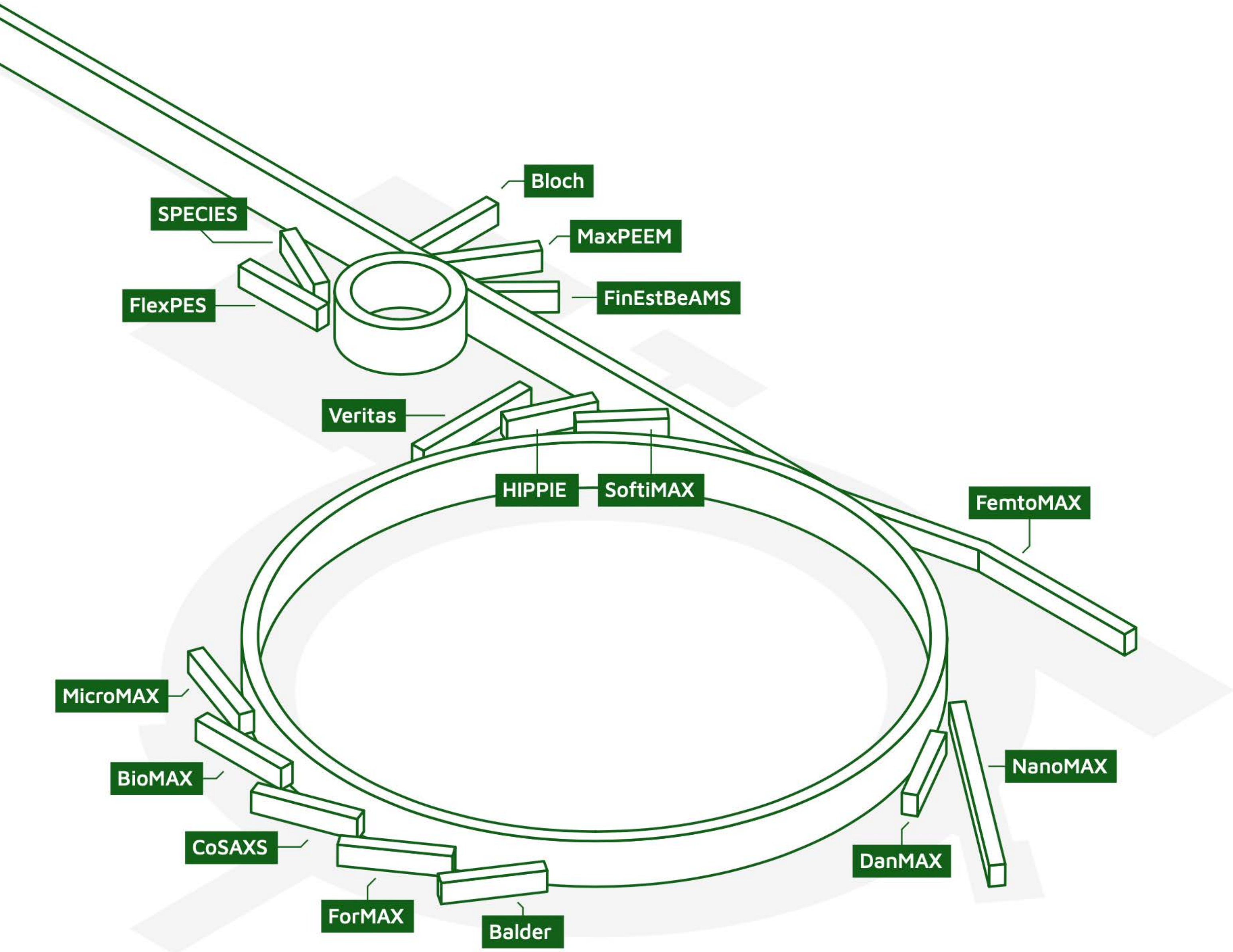
Figure 13: Some of the HarmonLIP participants during a tour of the MAX IV facility.



Facts and Figures

MAX IV beamlines

Beamline	Methods
Balder	Hard X-ray absorption and emission spectroscopy (XAS, XES) and X-ray diffraction (XRD) with emphasis on in-situ and time resolved studies
BioMAX	Macromolecular crystallography with a high degree of automation and remote user access
Bloch	Angle-resolved photoelectron spectroscopy (ARPES) including spin resolution (spin-ARPES) for studies of the electronic structure of solids and surfaces
CoSAXS	Small and wide angle X-ray scattering (SAXS, WAXS) and coherent techniques for soft matter and biomaterials
DanMAX	Powder diffraction (PXRD) and full-field tomographic imaging of hard (energy) materials
FemtoMAX	Time-resolved hard X-ray scattering (XRD) and spectroscopy (XAS) methods for studies of ultrafast processes
FinEstBeAMS	Electron and ion spectroscopies, and luminescence methods for studies of low density matter and solids
FlexPES	Soft X-ray spectroscopies for studies of low density matter and solids
ForMAX	Full-field tomography, SAXS/WAXS, scanning and imaging
HIPPIE	Ambient Pressure Photoelectron Spectroscopy (AP-XPS) on solids and liquids
MAXPEEM	Photoelectron microscopy for investigation of surfaces and interfaces
MicroMAX	Macromolecular crystallography, serial crystallography, time-resolved crystallography
NanoMAX	Imaging with spectroscopic and structural contrast techniques on the nano scale
SoftiMAX	Scanning transmission X-ray microscopy (STXM) and coherent imaging methods
SPECIES	Resonant inelastic X-ray scattering (RIXS) and Ambient Pressure Photoelectron Spectroscopy (AP-XPS)
Veritas	Resonant inelastic X-ray scattering (RIXS) with a unique resolving power and high spatial resolution

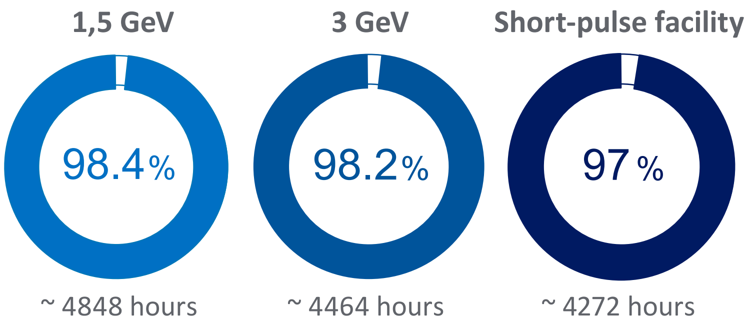

Bloch
SPECIES
MaxPEEM
FlexPES
FinEstBeAMS
Veritas
HIPPIE
SoftiMAX
FemtoMAX
MicroMAX
NanoMAX
BioMAX
CoSAXS
DanMAX
ForMAX
Balder

MAX IV in Numbers 2022

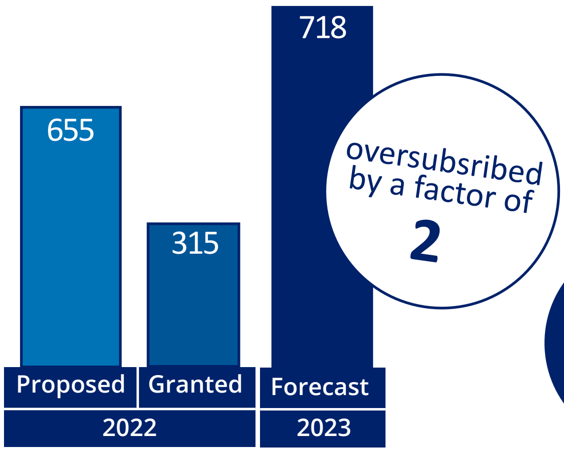
BEAMLINES

In 2022, the last two funded MAX IV beamlines, **MicroMAX** and **ForMAX**, received their **first X-ray light**. This bring the MAX IV beamline portfolio to **16 beamlines,** which will provide more research opportunities in the areas of structural biology and sustainable materials.

ACCELERATORS UPTIME



USER SCIENTIFIC PROJECTS

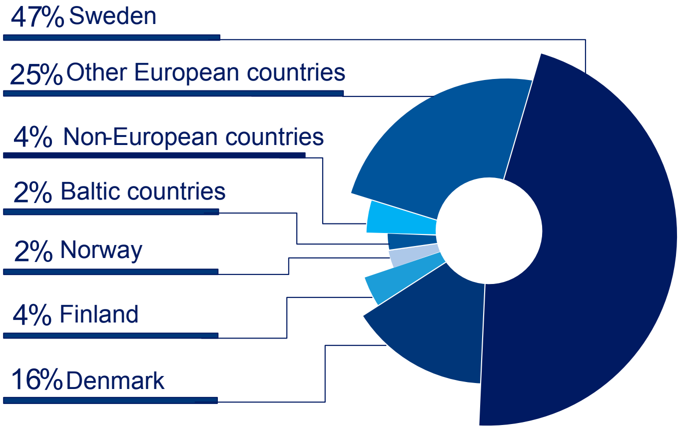


~ 41000 hours

of beamtime for user scientific projects across all MAX IV beamlines

USERS

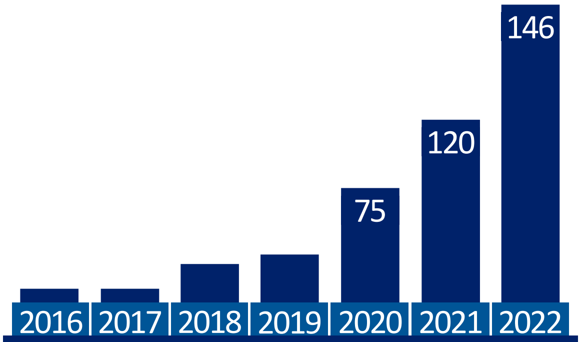
1400 users from **140** institutions across **32** countries



1 in 3

MAX IV users is a female scientist

PUBLICATIONS



Publications per year

430 publications from 2016–2022

+22% increase of publications compared to 2021

RESEARCH AREAS

- Physics and astronomy
- Chemistry
- Chemical engineering
- Materials science
- Engineering
- Energy
- Life and health sciences

INDUSTRY

5678 hours of user scientific projects connected with industry

694 paid proprietary hours*

33 proprietary user groups*

81% proprietary industry users are from Sweden

*33% of all proprietary hours were funded by Vinnova through Industrial Pilot Projects

FULL-TIME EMPLOYEES

300 full-time employees

75% | 25%

MAX IV International Collaborations

ExPaNDS – European Open Science Cloud Photon and Neutron Data Services

A collaboration that aims to deliver standardised, interoperable, and integrated data sources and data analysis services for photon and neutron facilities.

HALRIC

EU-supported initiative to increase companies and hospitals engagement in research collaborations with research infrastructures in the Öresund-Kattegat-Skagerrak (ÖKS)-Hamburg area.

nffa.eu research infrastructure

NFFA-Europe is a center for research infrastructure that integrates nanofoundries.

MAX IV

ReMade @ARI

A central hub for circular economy research – network of 48 facilities supporting recyclable material science.

LEAPS INNOVATION

European hub working with long-term partnerships between industry and European light sources, synchrotrons and free-electron lasers.

iNEXT DISCOVERY

The consortium enables access to structural biology research infrastructures for all European researchers, including non-experts in structural biology.

PRISMAS

PhD Research and Innovation in Synchrotron Methods and Applications in Sweden – is a doctoral programme that trains the next generation of leading synchrotron experts.

Read more about national and international collaboration projects MAX IV is involved in via: maxiv.lu.se/collaboration

* European projects running in 2022.

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