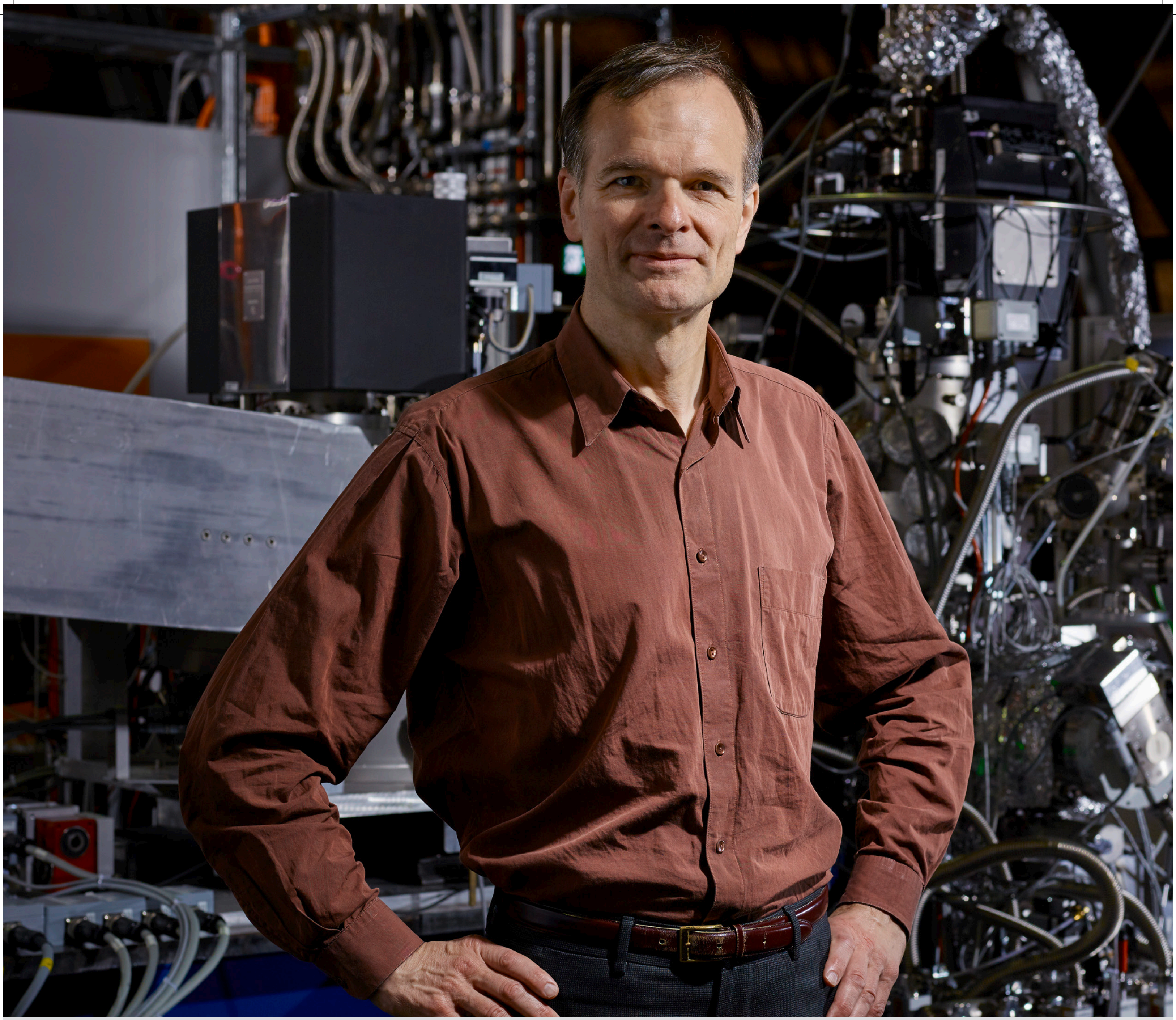
PAUL SCHERRER INSTITUT

Swiss Light Source

Solutions for Industry with Synchrotron Light



Gabriel Aeppli, Head of Synchrotron Radiation and Nanotechnology, Paul Scherrer Institute

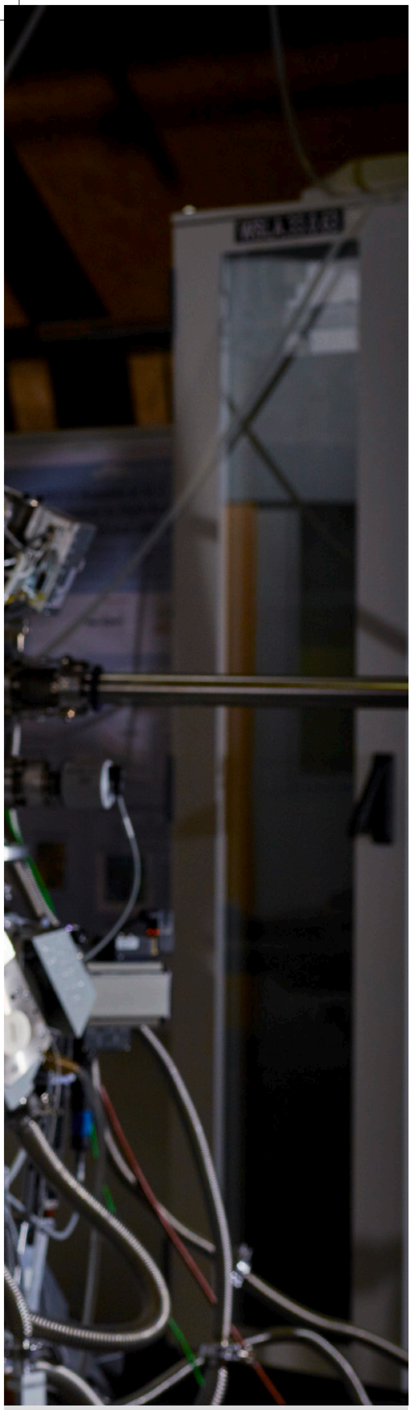
«The Paul Scherrer Institute is Switzerland's largest national laboratory for engineering and natural sciences, and we strongly welcome industry to make use of our research facilities, either directly, or in partnership with university research teams.

At the Swiss Light Source and Nanotechnology Laboratory, our intensely bright, pinpoint-sharp beams of x-rays and ultraviolet light are regularly used by industry to solve problems in materials science, medicine, food science, energy supply and the environment.

The examples in this booklet are just a few of the many where industry is using our state-of-the-art instruments to solve immediate problems, refine procedures for later use in product development and manufacturing, or build a full understanding of new materials.

We would be delighted to discuss with you how we can help your company.

Please get in touch.»



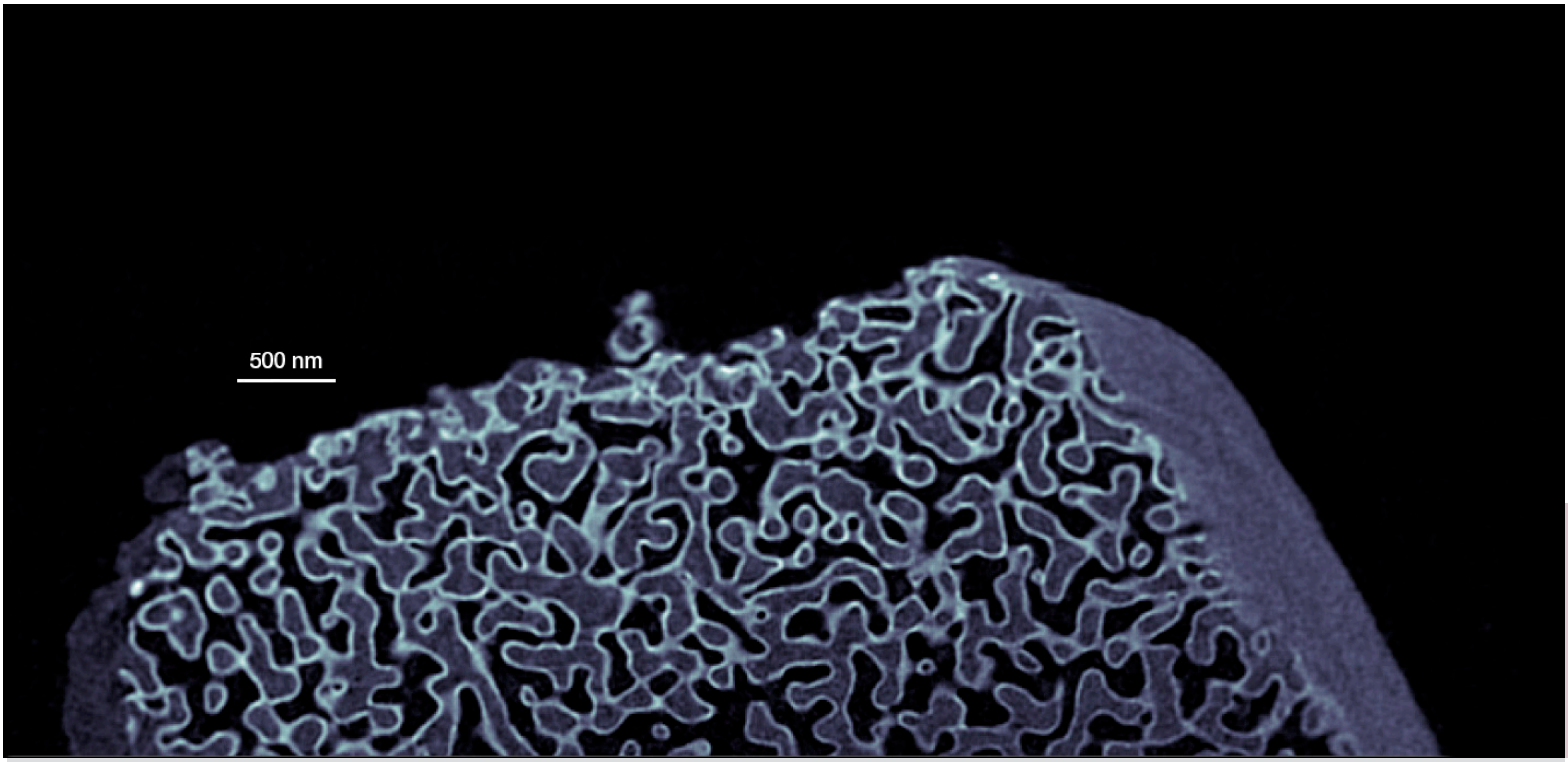
Content

3D mass density imaging

What is the nanostructure of a coated porous glass?

Ptychographic x-ray computed tomography can bridge the resolution gap between electron microscopy and other x-ray imaging techniques. A nano-porous glass used for the calibration of high pressure mercury porosimeters with a surface coating of Ta_2O_5 was used to test the limits of the technique.

A world record for tomography was achieved with 16 nanometre isotropic 3D resolution. The spatial distribution of pores, glass network and coating layer were distinctly resolved in the nano-porous glass.

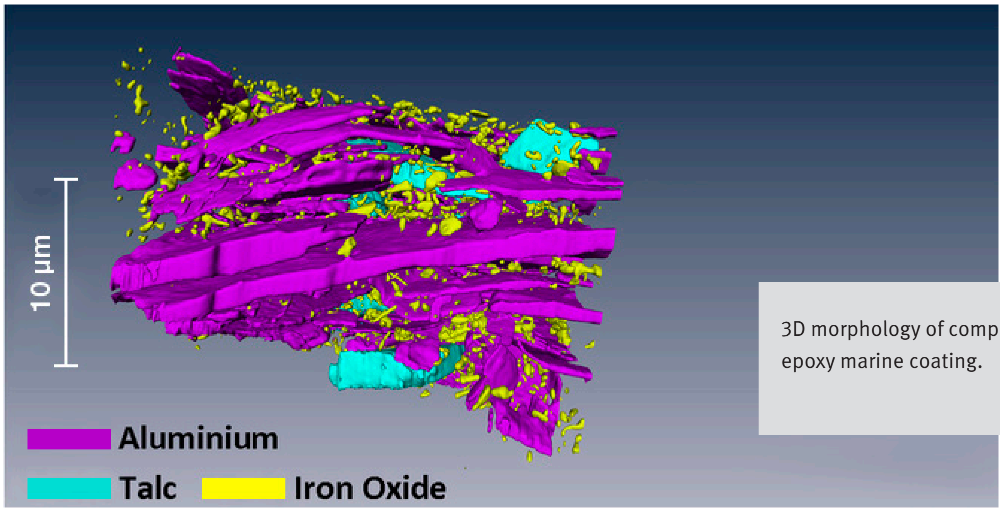


Section through nano-porous glass with three distinct grey levels for air (black), glass (grey), and a thin layer of Ta_2O_5 (white) coating the pores.

How is the performance of a marine coating linked to its nanostructure?

Epoxy coatings containing aluminium flakes have excellent anticorrosive properties and are widely applied to protect steel structures in the ocean. Ptychographic x-ray computed tomograph at the Swiss Light Source was used by a team from the London Centre for Nanotechnology, University College London and AkzoNobel to evaluate the connection between coating structure and performance.

An approach for validating mechanistic assumptions and modelling electrochemical processes was developed. It can be used to shorten the product research and development lifetime.

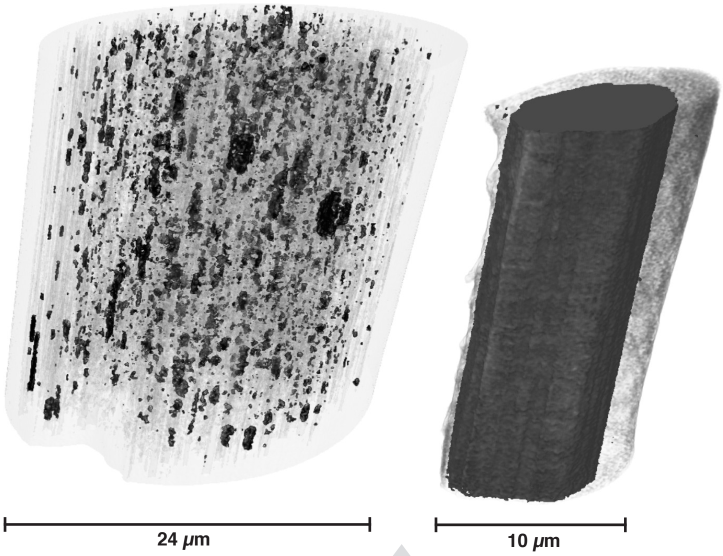


3D morphology of components in aluminium epoxy marine coating.

How is the nanostructure of a carbon fibre related to its tensile strength?

Manufacturing carbon fibre from polyacrylonitrile precursor is expensive. New lignin-based precursors, for carbon fibres with improved performance and reduced cost, are under development. Honda R&D Europe (Deutschland) wanted to relate mechanical strength to high-temperature heat treatment, graphitisation, porosity and density within the fibre.

With 100 nanometre resolution, highly resolved 3D mass density maps of entire sections of single carbon fibres revealed the porosity and spatial distribution of graphitisation. This can be directly related to standard laboratory characterisations.

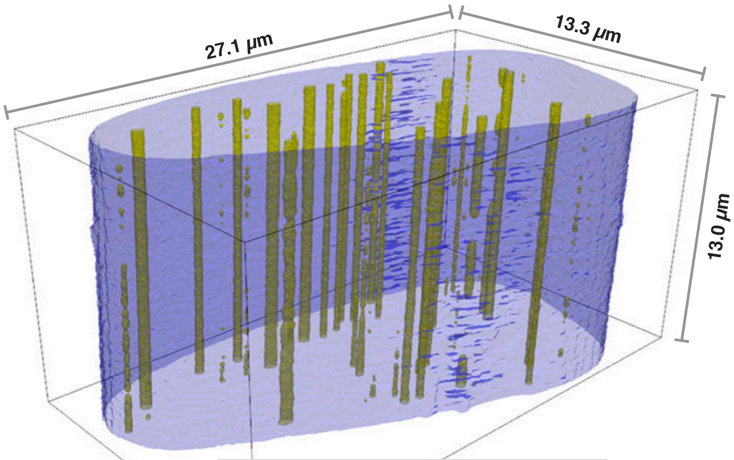


Ptychographic x-ray computed tomography observes the nano-morphology of 10 micrometre diameter carbon fibres.

How do silk fibres absorb water?

Nanopores in silk fibres influence the physical properties. For silk-based tissue engineering applications, it was unclear if the pores acted as water reservoirs when the silk became wet. The question is also important for using silk for organic electronics in textiles.

3D images of the interior of the silk fibre in dry and wet conditions revealed that the fibre swells anisotropically. Water molecules interacted with the silk protein rather than filling up the pores.

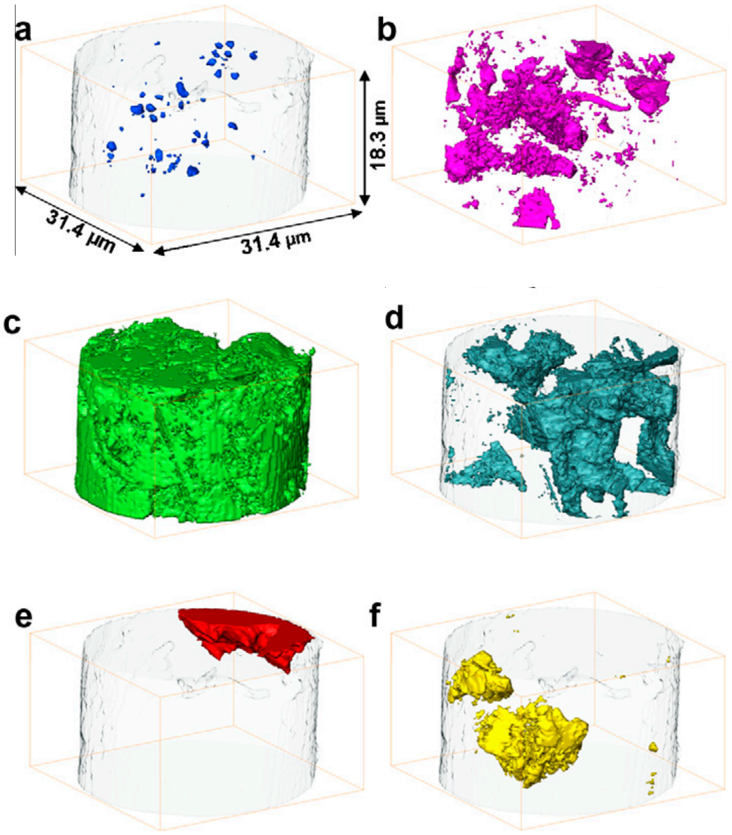


The pores inside a silk fibre are not filled with water when wet.

What is the mineral phase distribution in hardened cement paste?

Knowledge of the spatial distribution of mineral phases in hardened cements can aid modelling of weathering and durability in harsh environments. The spatial distribution of mineral phases in epoxy resin impregnated portland cement was required without sectioning the sample.

A 3D electron density map was derived by non-destructive ptychographic x-ray computed tomography. Six different material phases and their chemical compositions were identified with high spatial resolution.



Surface renderings of six phases in a portland cement sample:
(a) Unimpregnated / partially-impregnated porosity, (b) epoxy-impregnated porosity, (c) other hydrates (predominantly epoxy-impregnated calcium–silicate–hydrates), (d) calcium hydroxide, (e) calcium carbonate, (f) unhydrated / partially-hydrated clinker residues.

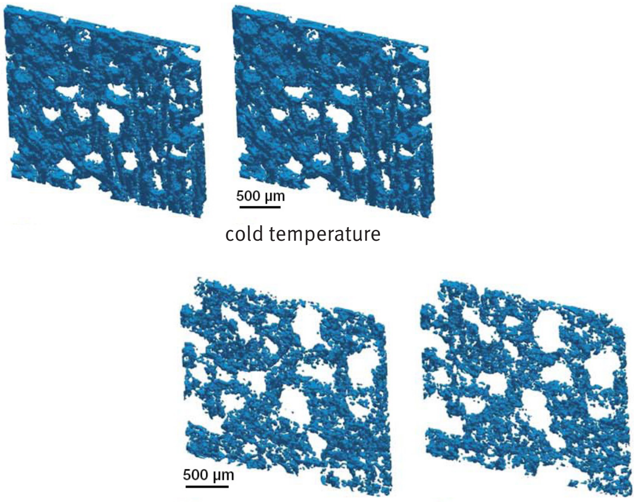
3D imaging

Real-time in situ studies

How does the texture of ice cream change with temperature?

The microstructure of food influences its perception during eating and is of crucial importance for manufacturing commercial food products. Ice cream has a microstructure that changes dramatically in response to temperature variations. Food scientists wanted to follow the impact of warming and cooling conditions, like those commonly found in commercial freezers.

A non-destructive x-ray time-lapse study of ice cream showed that the ice crystals and air bubble microstructure exhibited significant coarsening when repeatedly warmed and cooled. This method can also be applied to the general study of random three phase materials.



cold temperature

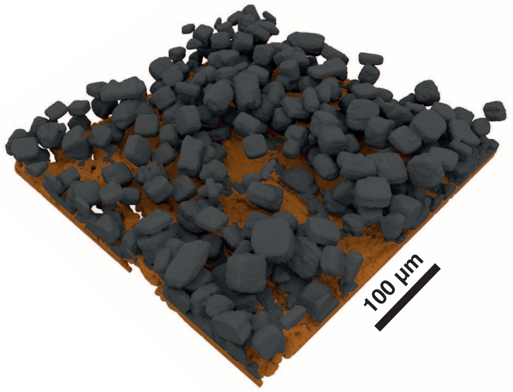
warm temperature

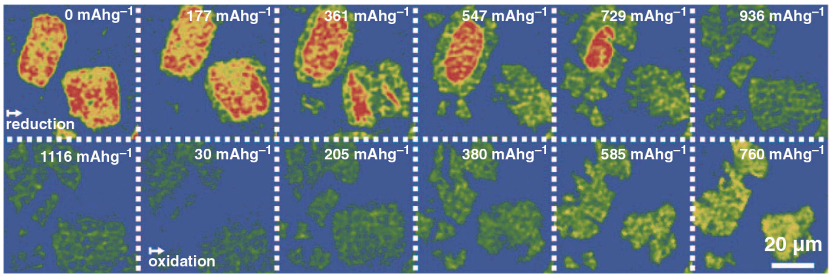
Changes in ice crystal structure in ice cream during a warming cycle.

How does a battery electrode change when operating?

Lithium-ion batteries could be improved by using high energy density materials. However, these materials have large volume changes during electrochemical operation leading to capacity loss and shorter battery lifetime. Using time resolved x-ray tomographic microscopy, engineers can relate the macroscopic battery electrochemical behaviour to structural and chemical changes in electrodes at the single-particle scale.

An electrochemical cell, transparent to x-rays, was built to study electrodes during battery operation in real time in a realistic environment. Structural and chemical changes in the electrode leading to electrochemical and mechanical degradation were tracked whilst the battery was charged and discharged.



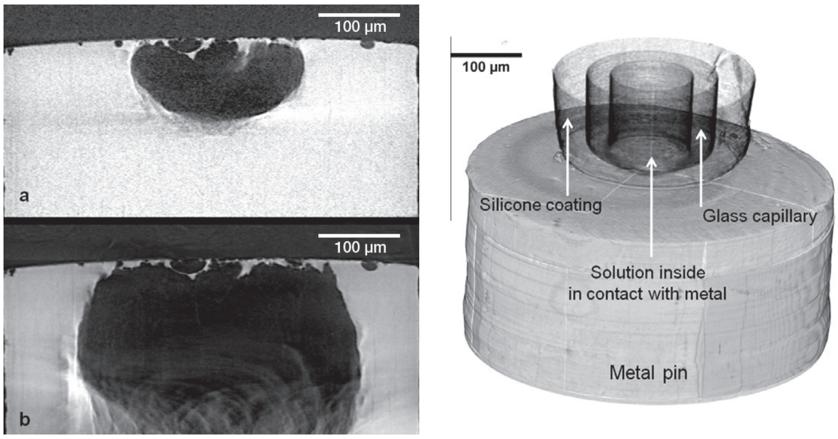


Two particles are tracked over time through their reactions. Nano-sized tin clusters form in a growing amorphous lithia matrix and alloy with Li to form Li_xSn. The particles expand and fracture under reduction.

How do chlorides corrode stainless steel?

Chloride-induced corrosion of stainless steel storage containers for radioactive waste stored in coastal locations over many decades is difficult to predict. The Radioactive Waste Management Directorate at the UK Nuclear Decommissioning Authority supported studies of pitting corrosion of stainless steel, with the aim of developing models of the corrosion damage that may occur on waste containers over long times.

A capillary microcell filled with NaCl solution was mounted on top of a stainless steel specimen. The growth of microscopic corrosion pits was imaged in situ and in real-time under electrochemical control. Corrosion pits followed the microstructure and rolling direction of the stainless steel.

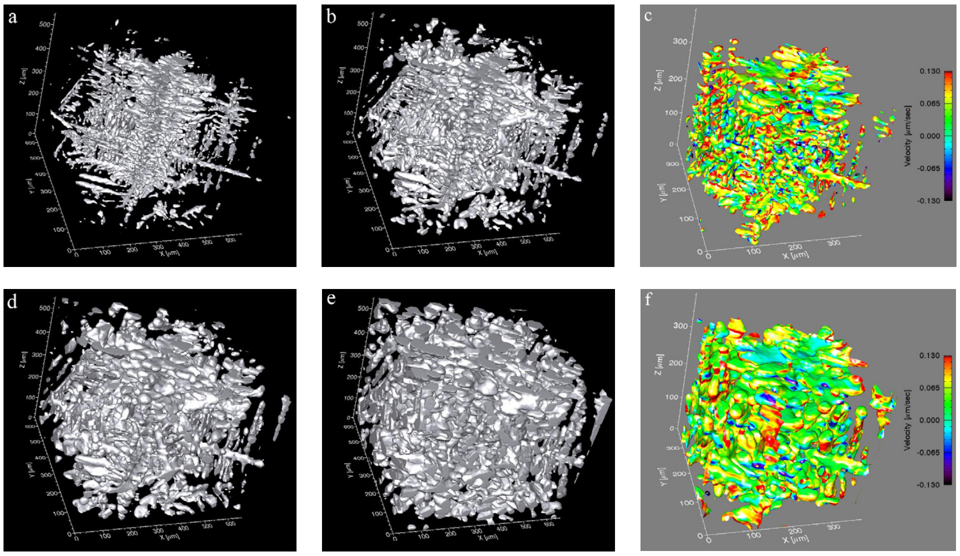


3D reconstructed view of an electrochemical capillary cell on top of a stainless steel pin, with cross-sections of a corrosion pit grown in 1 M NaCl at 50 µA for (a) 1 minute and (b) 6 minutes.

How fast do dendrites form in an aluminium-copper alloy?

Understanding the formation of materials at elevated temperatures is critical for determining their final properties. Real-time data on the solidification of an aluminium-copper alloy revealed the formation of the microstructure and how fast the solid-liquid evolution occurred.

A 300 W infra-red laser heating system was built to provide controlled localised heating up to 1800° C. As the alloy was cooled from above the melting point, ultrafast 3D x-ray tomographic microscopy captured the velocity of the evolving solid interface and dendrite growth with 1.1 µm pixel size.



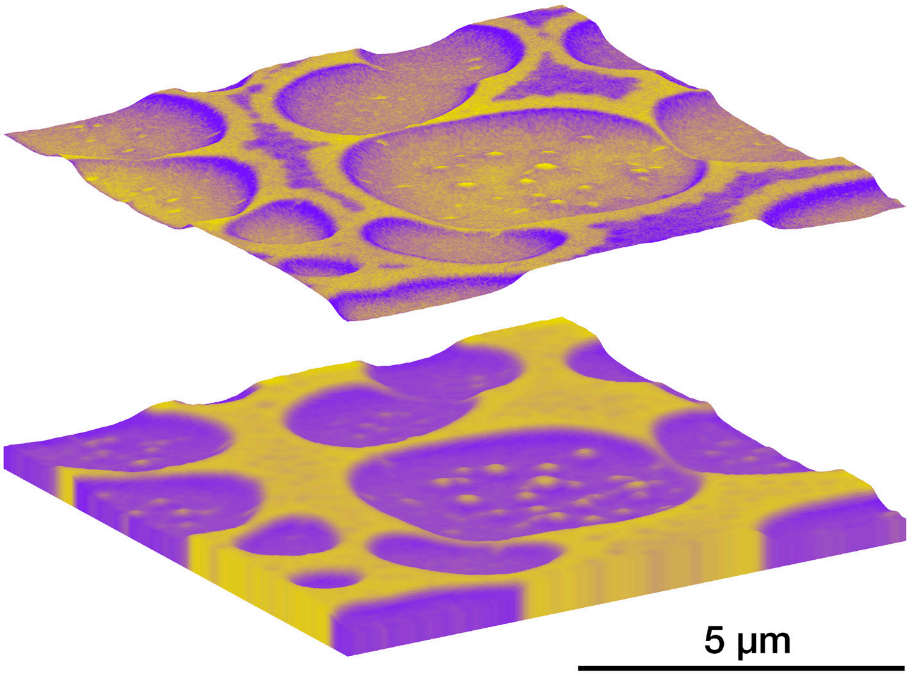
In situ dendrite evolution in an aluminium-copper alloy captured using ultrafast 3D tomography. Aluminium-rich dendrites are shown in (a, b, d, e). (c) and (f) show smaller portions of the solid–liquid interface of (a) and (d) coloured according to the velocity of the moving surface with warmer colours, eg red and yellow, indicating faster growth of the dendrites.

Separate surface from bulk properties

Is the surface layer of a blended polymer film well-connected to the bulk?

Cambridge Display Technology Ltd are developing blends of semiconducting polymers to be placed between two electrodes in prototype organic solar cells. Electrical performance is directly linked to the thin film microstructure and its connection to the electrodes. Common imaging techniques such as atomic force microscopy and electron microscopy lack the chemical sensitivity to decouple surface layer structure and composition from the bulk of the film.

Scanning transmission x-ray microscopy can simultaneously generate images of the surface and bulk structure, even for materials with no difference in electron density or constituent atoms. The phase-separated layered structure of the thin film could be uniquely imaged. Polymer droplets with different compositions were found to connect through the layer structure giving good electrical connectivity.



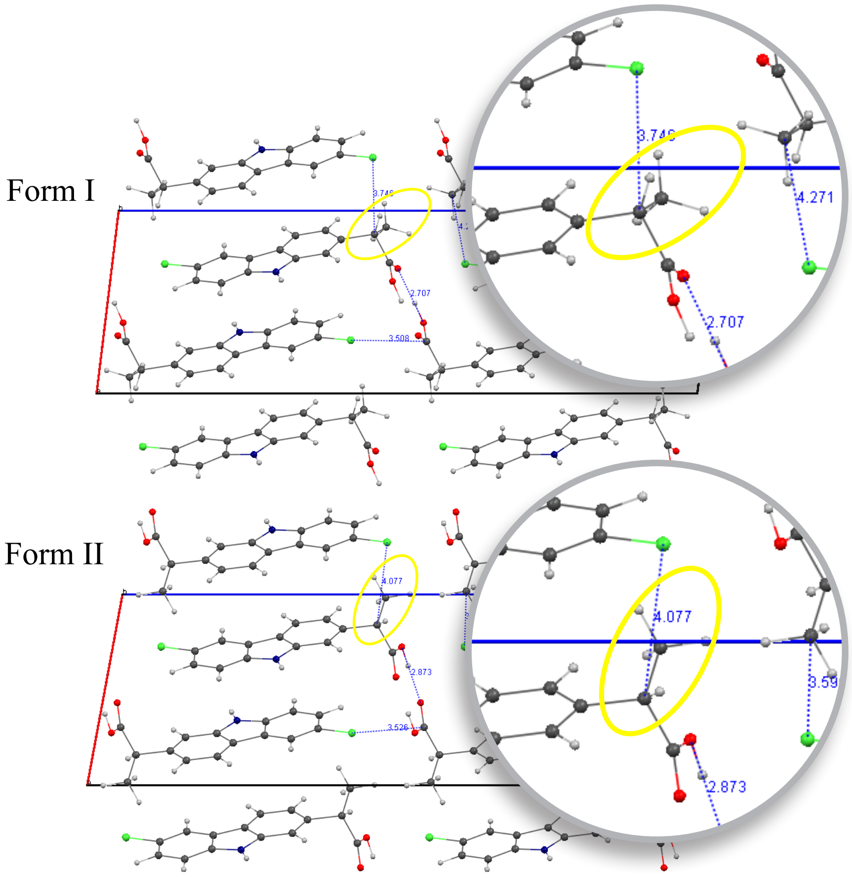
Thickness and composition variations in a 10 x 10 micrometre area of a 150 nanometre thick film of semiconducting polymers.

Detect impurities and polymorphs in chemicals and drugs

Do two forms of carprofen have the same structure?

Polymorphism can have a significant impact on the quality and performance of pharmaceutical and chemical products. Carprofen is a non-steroidal anti-inflammatory drug used in veterinary medicine. Characterisation of possible polymorphs by laboratory x-ray powder diffraction could only indicate that carprofen might exist in two different forms.

With high-resolution synchrotron powder diffraction, carprofen I (thermodynamically stable form) and carprofen II (kinetically stable form) were found to be conformational polymorphs differing only by the torsional angle of a methyl group.



Carprofen polymorphs differ only by the torsional angle of the methyl group.

Fast, high-resolution synchrotron x-ray powder diffraction is also a powerful tool for quantitative phase analysis of pharmaceuticals, both pure active pharmaceutical ingredients and formulated drugs.

Excelsus Structural Solutions, a PSI spin-off company that provides analytical services to the pharmaceutical industry, demonstrated the ability to directly detect and quantify minority phases smaller than 0.05%wt in pharmaceutical mixtures reaching unprecedented Level of Detection (LOD) and Level of Quantification (LOQ).

What is synchrotron light?

Synchrotron light is routinely over a billion times brighter than a laboratory x-ray source. Materials can be studied in incredible detail with significant advantages over standard laboratory x-ray or ultraviolet light sources.

The Swiss Light Source at the Paul Scherrer Institute generates intensely bright beams of synchrotron light by accelerating electrons to near light-speed and circulating them in a 288 metre circumference storage ring to produce synchrotron light.

Unique characteristics of synchrotron light

Brightness
Synchrotron light sources provide a photon flux 12 orders of magnitude brighter than the best available laboratory x-ray sources.

Wavelength
The incoming wavelength of synchrotron light, from infrared to hard x-rays, can be varied over a wide range and tuned to the requirements of the experiment.

Polarisation
Synchrotron light can be produced with a variety of polarisation states, including linear and circular polarisation.

Time structure
A synchrotron light source produces photon flashes at megahertz frequency allowing time-resolved studies to be carried out.

Coherence
Fully coherent x-ray beams allow lensless imaging and retrieval of an object's real space structure.

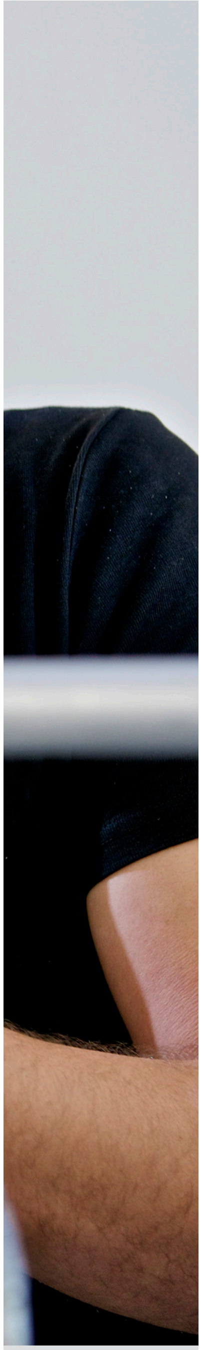
See http://www.psi.ch/sls/
for detailed information.

Inside the Swiss Light Source. Intensely bright beams of x-ray and ultraviolet light emerge from the storage ring tunnel structure running around the middle of the hall to be used by experiments located in the outer area.

Jens Wenzel Andreasen,
Senior Researcher DTU Energy,
Denmark

«A lot of attention is given to the user experience and accessibility at the Swiss Light Source. You can be busy taking data just a few minutes after starting your experiment time.
The combination of very high flux and efficient detectors allows for time-resolved experiments down to milliseconds. These properties provide ample scope for simulating realistic industry processing relevant for production.
I believe that many industrial users can get a fast answer to their most challenging ideas by using the Swiss Light Source.»

Benefits of using the Swiss Light Source



- Gold standard synchrotron light source for reliability and efficiency.
- Long tradition of building world-class high performance research infrastructures supplying beams of x-rays, neutrons, muons and protons for industrial and academic research.
- Experienced in handling commercial confidentiality and other industry requirements.
- Excellent support of your team from initial enquiry, through to sample preparation, experiments and data analysis using the best equipment for your application.
- Dedicated technology transfer team at the Paul Scherrer Institute has been supporting partnerships between the Swiss Light Source and industry for many years.
- Single point of contact for all enquiries for easy communication between the Swiss Light Source, the Paul Scherrer Institute and your company.
- More than 70 expert scientists to work with you on experiments to maximise the performance of the x-ray instruments
- State-of-the art experimental facilities, high levels of automation and on-site sample preparation laboratories.
- Remote control and monitoring of experiments
- Rapid turnaround using our flexible access procedures.
- On-site guest house and restaurants for use during experiments

Technical references

What is the nanostructure of a coated porous glass?
X-ray ptychographic computed tomography at 16 nm isotropic 3D resolution
M Holler et al, Sci. Rep. 4 (2014) 3857;
doi: 10.1038/srep03857

How is the performance of a marine coating linked to its nanostructure?
Three-dimensional structure analysis and percolation properties of a barrier marine coating
B Chen et al, Sci. Rep. 3 (2013) 1177;
doi: 10.1038/srep01177

How is the nanostructure of a carbon fibre related to its tensile strength?
Characterization of carbon fibers using x-ray phase nanotomography
A Diaz et al, Carbon 67 (2014) 98;
doi: 10.1016/j.carbon.2013.09.066
Partial financing of this work through the ColdWear project from industy partners Statoil ASA, Total E&P Norge AS, Janus Holdings AS, Weenaas AS, and Swix Sport AS.

How do silk fibres absorb water?
Ptychographic x-ray tomography of silk fiber hydration
M Esmaeili et al, Macromolecules 46 (2013) 434;
doi: 10.1021/ma3021163

What is the mineral phase distribution in hardened cement paste?
Density mapping of hardened cement paste using ptychographic x-ray computed tomography
P Trtik et al, Cement & Concrete Composites 36 (2013) 71;
doi: 10.1016/j.cemconcomp.2012.06.001

How does the texture of ice cream change with temperature?
3D-characterization of three-phase systems using x-ray tomography: tracking the microstructural evolution in ice cream
B Pinzer et al, Soft Matter 8 (2012) 4584;
doi: 10.1039/c2sm00034b

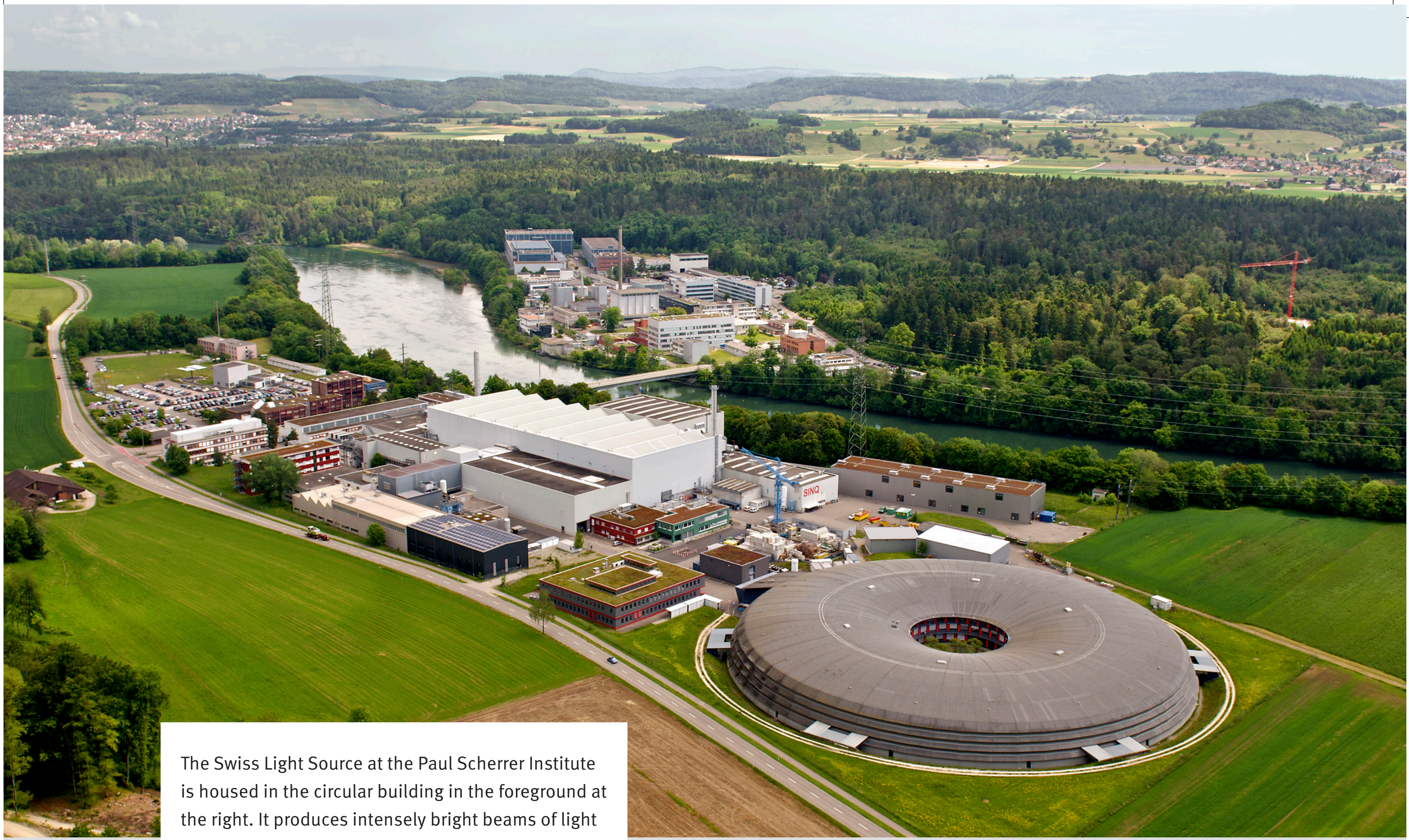
How does a battery electrode change when operating?
Visualization and quantification of electrochemical and mechanical degradation in Li-ion batteries
M Ebner et al, Science 342 (2013) 716;
doi: 10.1126/science.1241882

How do chlorides corrode stainless steel?
In situ synchrotron x-ray micro-tomography study of pitting corrosion in stainless steel
S Majid Ghahari et al, Corrosion Science 53 (2011) 2684;
doi: 10.1016/j.corsci.2011.05.040

How fast do dendrites form in an aluminium-copper alloy?
Development of a laser-based heating system for in situ synchrotron-based x-ray tomographic microscopy
J Fife et al, J. Synchrotron Rad. 19 (2012) 352;
doi: 10.1107/S0909049512003287
Financing of this work through the Swiss Competence Centre of Materials Science and Technology (CCMX) and its industry partners Asulab, Constellium, Kugler Bimetal, Novelis, Rolex and Varinor.

Is the surface layer of a blended polymer film well-connected to the bulk?
Simultaneous surface and bulk imaging of polymer blends with x-ray spectromicroscopy
B Watts & C R McNeill, Macromol. Rapid Commun. 31 (2010) 1706;
doi: 10.1002/marc.201000269

Do two forms of carprofen have the same structure?
Thermal, spectroscopic, and ab initio structural characterization of carprofen polymorphs
G Bruni et al, J. Pharma. Sci. 100 (2011) 2321;
doi: 10.1002/jps.22470



The Swiss Light Source at the Paul Scherrer Institute is housed in the circular building in the foreground at the right. It produces intensely bright beams of light from the infrared through to hard x-rays.

PSI in brief

The Paul Scherrer Institute PSI is a research centre for natural and engineering sciences, conducting cutting-edge research in the fields of matter and materials, energy and environment and human health. By performing fundamental and applied research, we work on sustainable solutions for major challenges facing society, science and economy. PSI develops, constructs and operates complex large research facilities. Every year more than 2200 guest scientists from Switzerland and around the world come to us. Just like PSI's own researchers, they use our unique facilities to carry out experiments that are not possible anywhere else. PSI is committed to the training of future generations. Therefore about one quarter of our staff are post-docs, post-graduates or apprentices. Altogether PSI employs 1900 people, thus being the largest research institute in Switzerland.

SLS Techno Trans AG

SLS Techno Trans AG is funded by PSI to make the research opportunities at the Swiss Light Source more visible to industry. It coordinates interactions between industry and PSI scientists specialised in the relevant fields and experimental techniques.

Contact

Stefan Müller
Tel. +41 56 310 54 27
stefan.mueller@psi.ch

SLS Techno Trans AG
Swiss Light Source
Paul Scherrer Institut
5232 Villigen PSI, Switzerland
www.psi.ch/sls-techno-trans-ag/

Paul Scherrer Institut :: 5232 Villigen PSI :: Switzerland :: Tel. +41 56 310 21 11 :: Fax +41 56 310 21 99 :: www.psi.ch