A1

Production XAFS

BL01B1, BL14B2, BL32B2

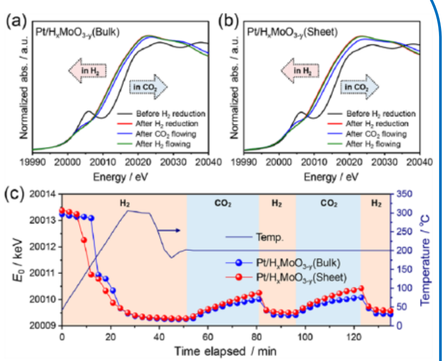
Keywords: Versatile XAFS, Automated XAFS, Local structure, Chemical state, Operando

- Features

X-ray absorption fine structure (XAFS) measures electronic states and the local atomic structure of matter. It is applicable to crystalline and amorphous materials, and has element selectivity that allows analyses of materials with many different elements. In-situ measurements of reactions is also possible. Automated systems for sequential measurements use robots for pellet fabrication. Our setup is designed for safe and easy handling of reaction gases and heating cells for operando measurements in catalytic chemistry.

- Examples

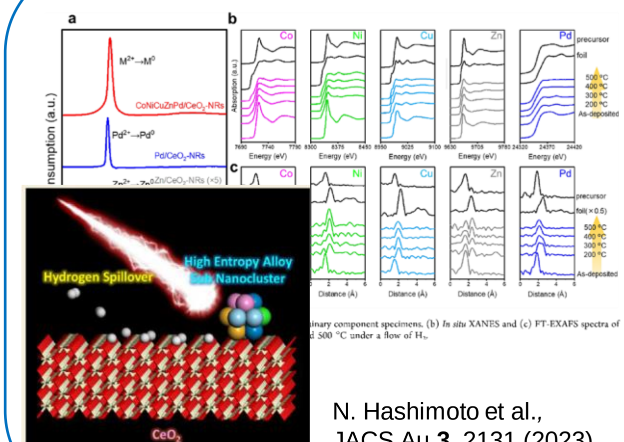
Reaction gas feeder and in-situ analysis of catalysts



BL01B1

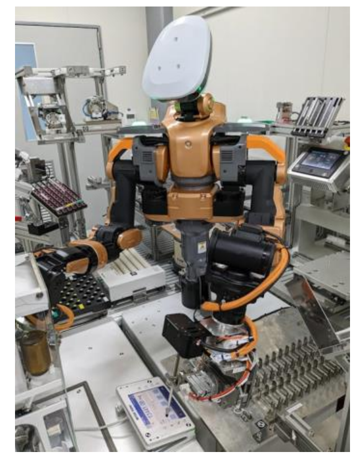
Y. Kuwahara et. al., J. Phys. Chem. C **127**, 4942 (2023).

Structural analysis of high-entropy alloy nanoparticles using element selectivity



N. Hashimoto et al., JACS Au **3**, 2131 (2023).

Pellet-fabrication robot for XAFS measurement



BL14B2

- Towards SPring-8-II

The photon density at the sample position will be increased by a factor of 10 by improving beamline optics. A high-count-rate fluorescence detector and a digital signal processor will be installed to realize XAFS measurements of low-concentration samples in a much shorter time in collaboration with data science researches.

A2 X-ray emission spectroscopy/HERFD-XAFS/ X-ray Raman scattering

BL39XU, BL36XU, BL12XU

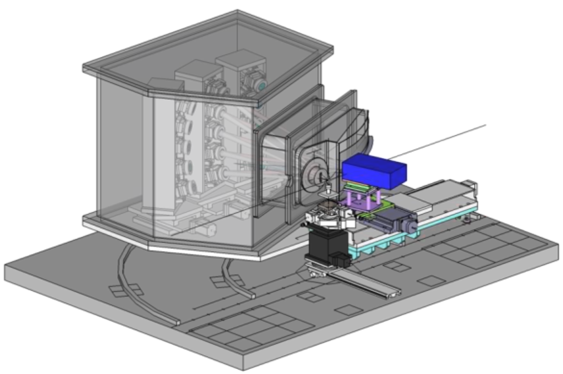
Keywords: High-sensitivity chemical state analysis, Trace element analysis, X-ray Raman scattering, RIXS

- Features

Our X-ray emission spectroscopy (XES) systems enables observation of fluorescent X-rays allowing practical high-energy resolution fluorescence detected (HERFD) XAFS, which can observe spectral fine structures and trace elements beyond the limits of conventional XAFS. This captures tiny changes in chemical states (local structures, bonding states, valence, etc.) of specific elements, used in a variety of fields, condensed matter physics, catalytic chemistry, and geo-environmental science.

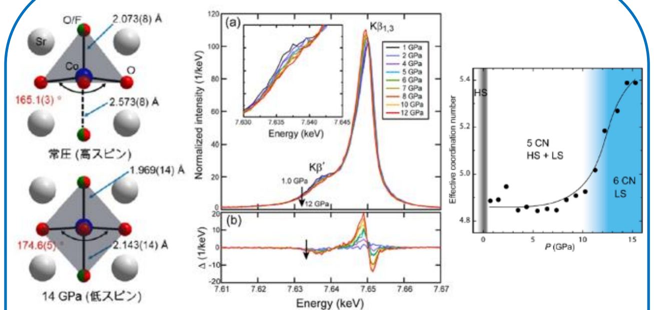
- Examples

High-sensitive X-ray emission spectrometer



✓ Up to 15 crystal can be mounted
✓ Analyzer crystals are placed in a vacuum chamberア

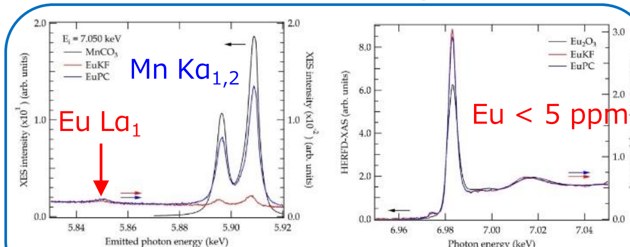
Spin transition under high pressure



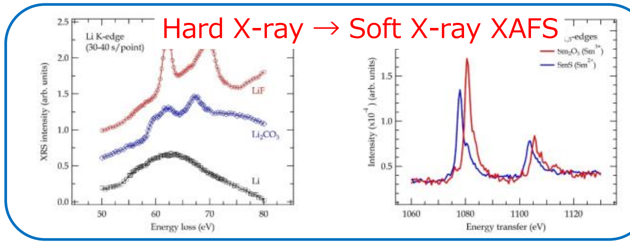
Observation of Co 3d state under high pressure
→ Verification of pressure-induced spin crossover

Tsujimoto, *et al.*, Sci. Rep. **6**, 36253 (2016).

Valence state analysis of dilute Eu by HERFD-XAFS



R. Konagaya, *et al.*, Chem. Lett. **50**, 1570 (2021).



X-ray Raman scattering

- Towards SPring-8-II

Improvement of spectral acquisition technology with high energy resolution and sensitivity will allow chemical state analysis of ultra-trace elements and observation of their electronic states. Our method will contribute to discovery of rare metal resources and development of next-generation functional materials using materials informatics through ultra-high throughput measurements with ultra-high sensitivity.

A3

XAFS-CT

BL36XU, BL37XU, BL39XU, BL29XU

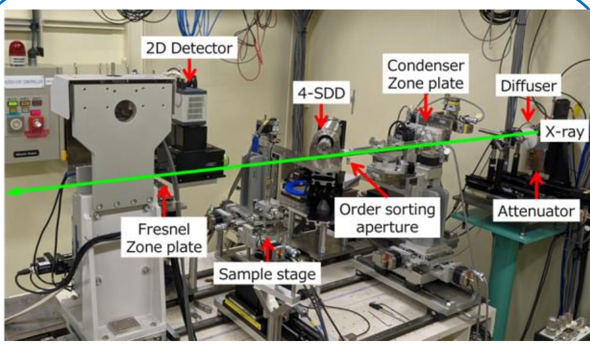
Keywords: Operando, Chemical state, Projection type, Imaging type, High-speed scanning type

• Features

The Ccombination of X-ray imaging with XAFS or X-ray fluorescence visualizes 2D/3D distributions of elements and their chemical states (local structures, bonding states), and is widely used in fuel cell catalytic research and environmental science. Projection and imaging measurements have been extensively used to shorten the measurement time. Recently, development of on-the fly scanning has enabled high-speed scanning commonly with fluorescence detectors with high-sensitivity and high count rate. Optical systems with variable magnification/reduction rates have been developed, leading to multi-scale imaging and observations of trace elements.

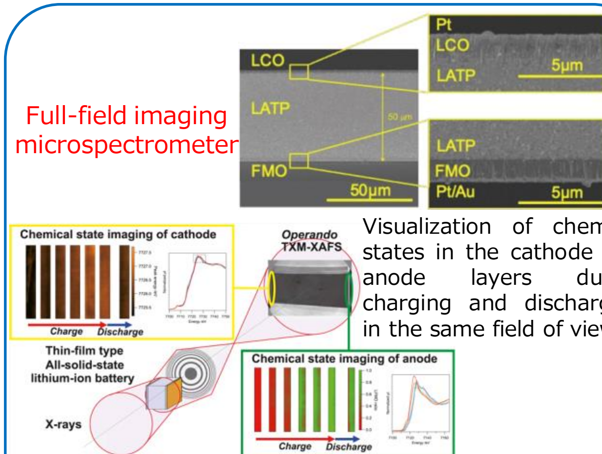
• Examples

Full-field microspectrometer



- ✓ Projection CT with high speed and wide field of view
- ✓ High-resolution imaging CT
  → High magnification with high efficiency

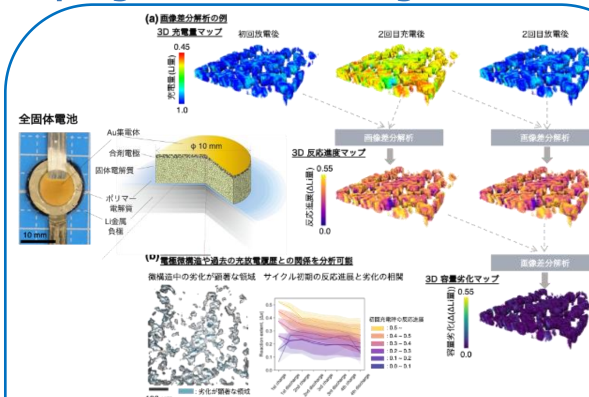
Visualization of electrode reactions in thin-film all solid state batteries



Visualization of chemical states in the cathode and anode layers during charging and discharging in the same field of view

N. Ishiguro, et al.,
ACS Appl. Energy Mater. **6**, 8306 (2023).

3D visualization of the degradation progression of storage batteries



Full-field projection microspectrometer

When/where/how degradation occurs in electrodes

Y. Kimura, *et al.*, Small Methods, (2023).
DOI : 10.1002/smtd.202300310

• Towards SPring-8-II

Improvement of XAFS imaging for higher spatial resolution, higher throughput, and multi-scale observation will realize highly efficient measurements of spatial distributions and time variation of elements and chemical states. This will contribute to development of higher functional materials by elucidation of mechanisms of chemical reactions. Collaboration with data-driven science will further develop 2D/3D spatial morphology and its kinetics.

BL37XU、BL39XU、BL29XU

A4 X-ray fluorescence, XMCD imaging

Keywords: Chemical state, Microstructure, Element distribution

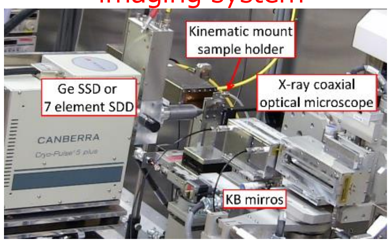
• Features

X-ray fluorescence imaging, in which X-rays are focused to a small size are scanned on a sample and fluorescent X-rays are generated, can target a variety of elements and visualize element distributions and chemical states with 100nm resolution. Imaging X-ray fluorescence microscopes using chromatic aberration-free mirror optics have been developed. X-ray magnetic circular dichroism (XMCD) imaging measurement methods are available, enabling scanning microscopy and CT-based 2D/3D magnetic domain structure observation.

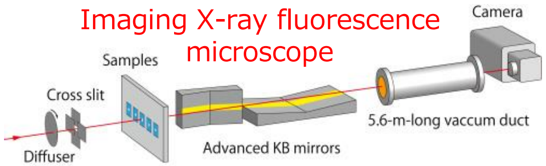
• Examples

X-ray fluorescence imaging system

Scanning X-ray fluorescence imaging system

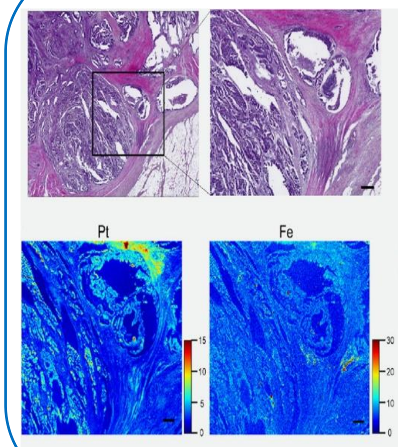


Imaging X-ray fluorescence microscope



S. Matsuyama *et al*., Opt. Express **27**, 18318 (2019).

Visualization of quantification of anticancer drug distribution in human tumor tissues

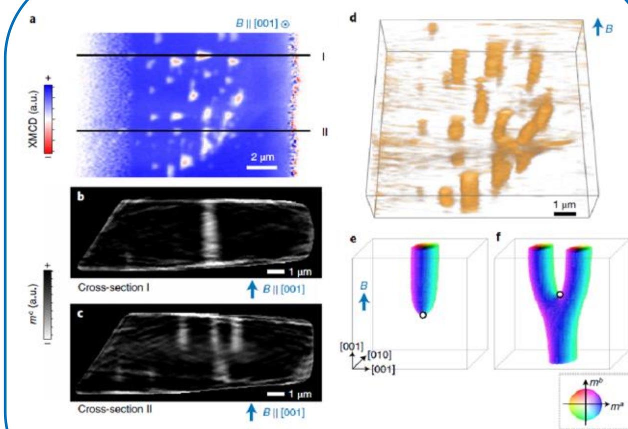


Typical histopathology of rectal cancer

Scanning XRF images
Left: Pt
Right: Fe

R. Koba *et. al.*, Int. J. Cancer. **146**, 2498 (2020).

3D visualization of Skirmion string by XMCD-CT



S. Seki et al., Nat. Mater. **21**, 181 (2022).

• Towards SPring-8-II

Innovations in focusing/imaging optics and detectors will realize high throughput imaging measurements, leading to correlation analysis of microstructures of materials. Operando/in-situ measurements of batteries, catalysts, magnetic materials, and biological samples will be available. Combining this with material informatics using databases and supercomputers will contribute to development of highly functional materials and even environmental problems.

A5

Fast XAFS

BL36XU

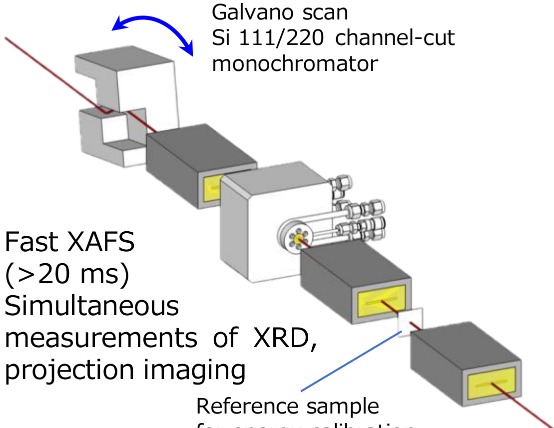
Keywords: QXAFS, Complex measurement, Chemical state, Operando

- Features

X-ray absorption fine structure (XAFS) needs scanning X-ray energy and takes a long time to complete element-selective measurements of chemical states and local structures. Our system has a compact spectrometer that enables millisecond-order fast XAFS (QXASF) used for tracking catalytic reactions of fuel cells. QXASF combined with X-ray diffraction (XRD) and projective imaging measures time dependence of 2D/3D distributions of chemical states.

- Examples

QXAFS measurement system



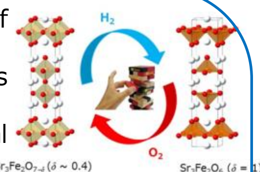
✓ Fast XAFS (>20 ms)
✓ Simultaneous measurements of XRD, projection imaging

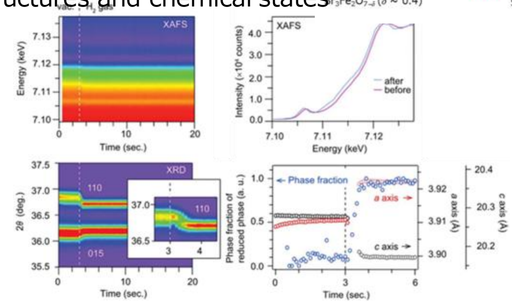
Observation of oxygen desorption reaction processes

Oxygen desorption reaction of $Sr_3Fe_2O_{7-\delta}$

Simultaneous measurements of XAFS/XRD

→ Correlation between crystal structures and chemical states



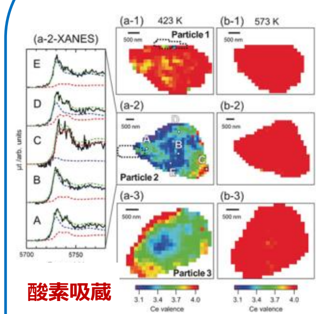


T. Yamamoto, *et al.*, Advanced Science **10**, 2301876 (2023).

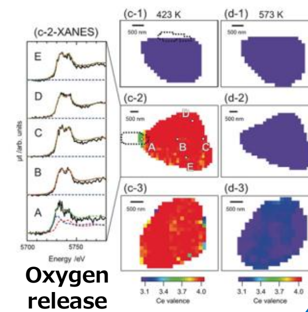
Spatial distribution of oxygen absorption and release reaction of catalyst particles

$Pt/Ce_2Zr_2O_x$

QXAFS + Projection imaging



酸素吸蔵



Oxygen release

H. Matsui, *et al.*, Angew. Chem. Int. Ed., **55**, 12022 (2016).

- Towards SPring-8-II

QXAFS measurements will be 100 times faster (order of microseconds) with higher precision spectral measurements. This will realize tracking of high-speed evolution of chemical reactions and local structures, acquiring multi-dimensional data, and establishing database for reaction mechanisms through collaboration with data science technology sucn as Bayesian statistics and AI, B1 **Production HAXPES**

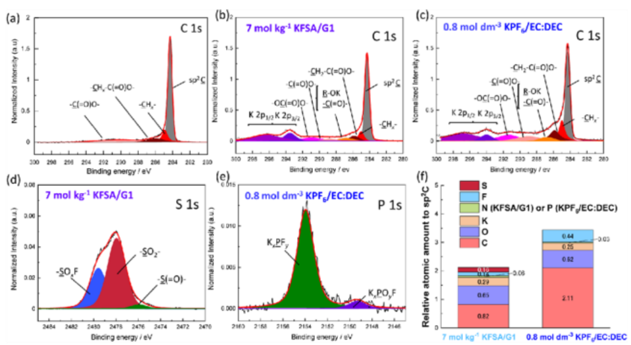
Keywords: Electronic state, Chemical state, Resonate HAXPES 3D space-resolved measurement, Automated HAXPES

- Features

Hard X-ray photoemission spectroscopy (HAXPES) non-destructively analyzes electronic states from the sample surface to the bulk (several tens of nm), and enables precise investigation of chemical bonding and electronic states in the bulk and buried interfaces. Our system has elements/orbital-selective methods called resonant HAXPES, which uses resonant excitation at the absorption edge, and 3D space-resolved analysis using focused X-ray beams and angular-resolved measurements.

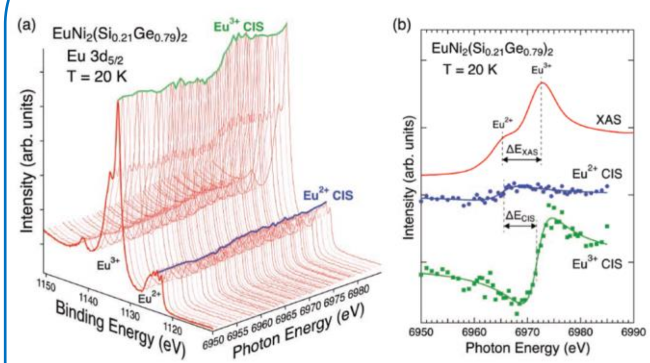
- Examples

Electrode surface-forming species for storage batteries



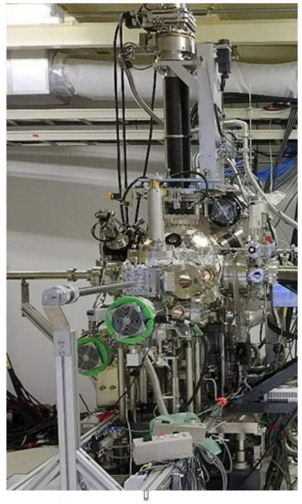
T. Hosaka et al., J. Mater. Chem. A, **8**, 23766 (2020).

Valence fluctuation phenomena in heavy electron systems



E. Ikenaga et al., Synchrotron Radiat. News **31**, 10 (2018).

High throughput HAXPES system (sample chamber)



- Towards SPring-8-II

Upgrading the focusing optics will enable electronic-state mapping of practical materials with high spatial resolution. Efficient analysis of multidimensional spectral data from continuous measurements will become possible, including 3D space-resolved measurements, energy scanning. Advanced analysis in a remote environment will be performed in real time.

B2

Atmosphere control HAXPES

BL09XU, BL46XU

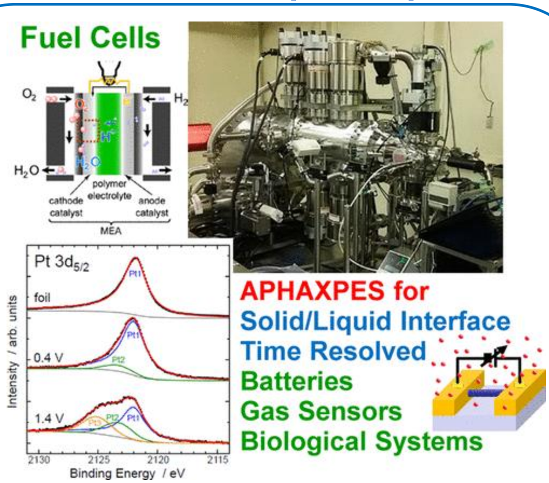
Keywords: Electronic state, Operando, Atmospheric pressure

- Features

Our hard X-ray photoemission spectroscopy (HAXPES) system has an atmosphere-controlled equipment for analyzing the electronic structures of samples under atmospheric pressure and specific atmospheres. This enables operando experiments under reactive conditions and electronic structure analysis of absorbed species.

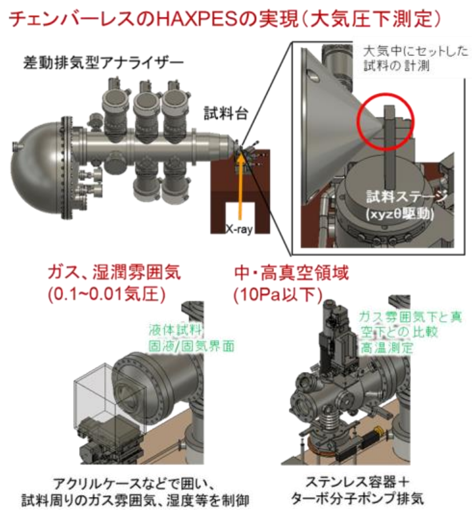
- Examples

Reaction mechanism of Pt catalysts for fuel cells atmospheric pressure

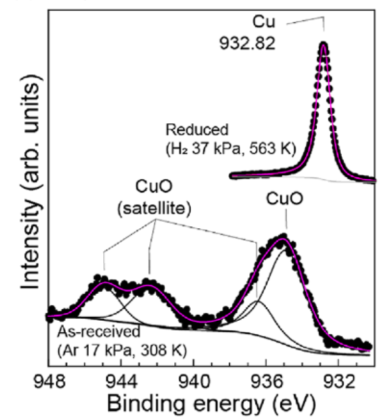


Y. Takagi et al., Acc. Chem. Res., **51**, 719 (2018).

Atmosphere controlled HAXPES system



Evaluation of catalysts under reaction gas atmosphere



T. Koitaya et al., J. Phys. Chem. C, **127**, 13044 (2023).

- Towards SPring-8-II

High-precision HASPES in-situ and under operando conditions will enable measurements of devices and under gaseous atmosphere.

C1

High resolution Compton scattering

BL08W

- Features

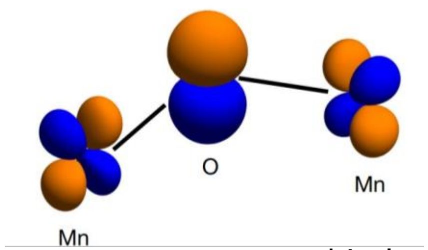
Keywords: Electron orbital states, Fermi surface

Electronic orbitals and Fermi surface are important for material properties. Lithium-ion batteries, for example, have the redox properties determined by the electronic orbitals that transfer electrons, while superconducting transitions, electric conduction and alloy regularization are affected by the shape of Fermi surfaces. High-resolution Compton scattering can visualize electron orbitals and Fermi surfaces, and extract related physical observables by measuring the electron density distribution in momentum space.

- Examples

Measurement of bulk states without surface effects

Mechanism of anion charge compensation (O^{2-}/O^{-}) of large-capacity cathode material $Li_xTi_{0.4}Mn_{0.4}O_2$



π-type Mn t_{2g} - O $2p$ orbitals

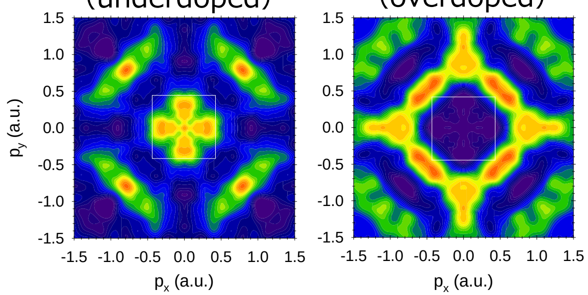
Nature **594**, 213(2021)

Measurement under electromagnetic fields and variable temperature

Visualization of hole states of high-Tc superconductor $La_{2-x}Sr_xCu_4O_4$

Hole creating superconductivity (underdoped)

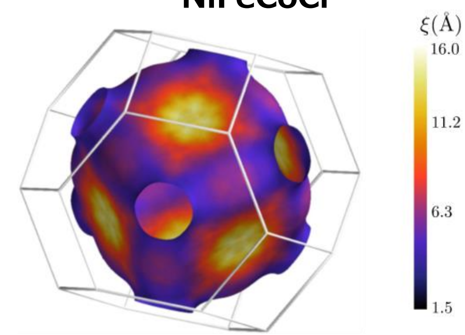
Hole breaking superconductivity (overdoped)



Science **332**, 698 (2011)

In situ measurement under ambient conditions or in solution

Fermi surface and electronic coherent length of random alloys NiFeCoCr



Phys. Rev. Lett. **124**, 046402 (2020)

- Towards SPring-8-II

High-resolution Compton scattering can measure bulk electronic states without interrupted by surface conditions. This method will promote fundamental understanding of the mechanism of high-capacity Li ion batteries, and contribute to the development of carbon-neutral materials.

Magnetic Compton Scattering

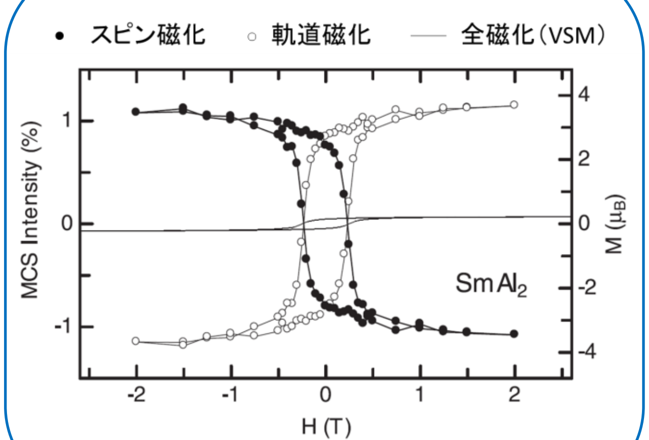
Keywords: Magnetic material, Magnetic property

• Features

Magnetic materials are important components in industry including development of electric vehicles. The magnetism has its origin in quantum phenomena, but it is difficult to evaluate on a microscale because most experimental methods can only extract total magnetization. To measure physical observables directly related to the electronic wavefunctions by spin states, magnetic Compton scattering is useful.

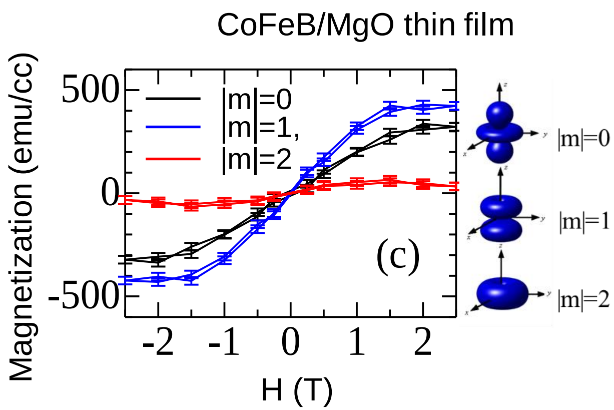
• Examples

Magnetization measurement with separated spin/orbital components



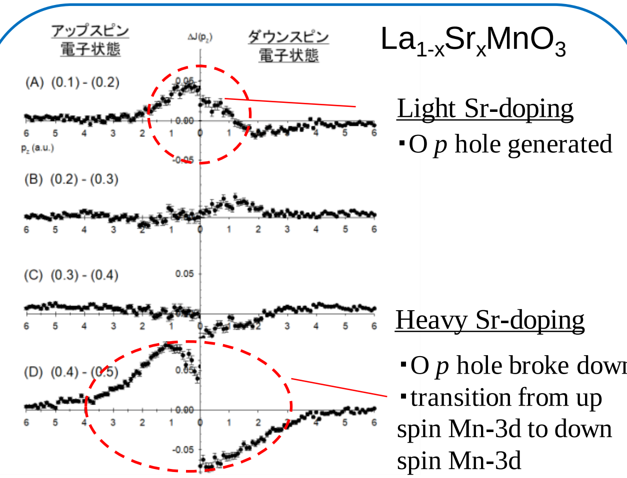
Appl. Phys. Lett. **102**, 082403 (2013)

Separated magnetization measurement by magnetic quantum number m



J. Phys.: Condens. Matter **28**, 436001 (2016)

Electronic states by up/down spin



Appl. Phys. Lett. **98**, 052107 (2011)

• Towards SPring-8-II

Magnetic Compton scattering uses high-energy X-rays having excellent penetration that enables evaluation of bulk properties free from surface states. The new source will realize high-speed mapping of magnetic properties inside magnetic materials/devices and at interfaces.

C2

BL35XU, BL19LXU

Nuclear Resonance Vibrational Spectroscopy - Active site of the enzyme -

- Features

Keyword: Element specific

Nuclear resonance vibrational spectroscopy is sensitive only to specific isotopes and provides information about their vibrational states. It is particularly useful for enzymes, because it can extract the vibrations of exactly the metals at the active center from a huge background. Combined with sophisticated simulations this provides valuable information about the structure of active centers of enzymes.

- Gallery

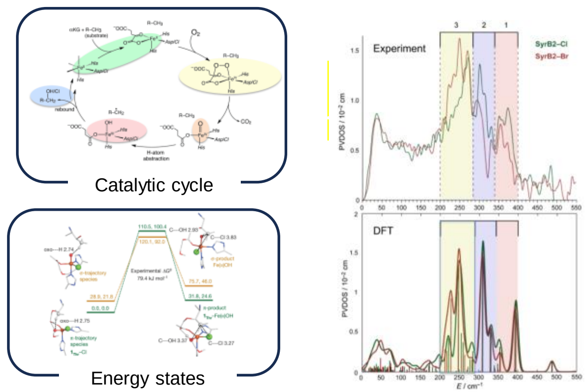
Average of 3 high impact (IF>12) publications every year

List of recent publications

[1] Nature Chem. Biol. (2023) https://doi.org/10.1038/s41589-022-01226-w.
[2] Inorg. Chem. 62 (2023)
[3] Faraday Discuss. 243 (2023).
[4] Biophys. J., 121 (2022) 346a.
[5] Proc. Natl. Acad. Sci. U.S.A., 118 (2021).
[6] J. Am. Chem. Soc., 143 (2021).
[7] Angew. Chemie Int. Ed., 60 (2021).
[8] J. Am. Chem. Soc., 143 (2021).
[9] Inorg. Chem., 60 (2021).
[10] Chem. Sci., 12 (2021).
[11] Biochemistry, 60 (2021).
[12] Angew. Chemie Int. Ed., 59 (2020).
[13] ACS Catal., 10 (2020).
[14] J. Am. Chem. Soc., 142 (2020).
[15] J. Am. Chem. Soc., 142 (2020).
[16] Chem. Sci., 11 (2020).

Active site in the enzymes

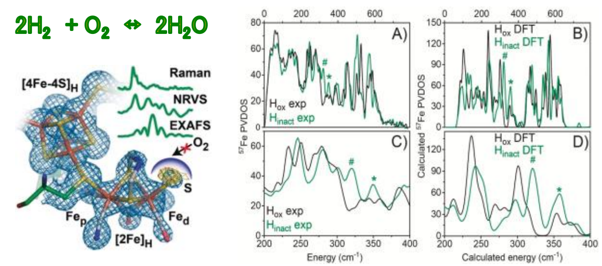
Elucidation of the Fe(IV)=O intermediate in the non-haem iron halogenase



Nature, 499 (2013) 320. doi:10.1038/nature12304

Enzyme for hydrogen: Hydrogenase

Crucial insight into O_2 stability mechanisms in the active site of the enzyme, hydrogenase



Angew. Chemie Int. Ed. **59**, 16786 (2020). https://doi.org/10.1002/anie.202005208

- Toward SPring-8-II

Smaller samples will be possible with a small beam size at SP8-II. We also hope to improve the resolution using the improved brilliance. Research of the artificial catalysts and material science, especially earth science, will be accelerated by these features.

C2

Energy domain Mössbauer Spectroscopy - Complex materials -

BL35XU, BL19LXU

• Features

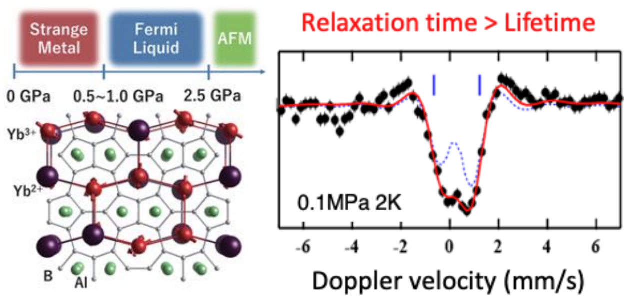
Keyword: Local electronic states

Energy domain Mössbauer spectroscopy at synchrotron radiation can investigate element-specific local electronic states with small beam size and without radioisotopes. This technique is especially useful for analyzing complex materials. Small beam size enable the study under the extreme conditions such as high pressure and/or high temperature easily. Basically this is hyperfine spectroscopy but with a much expanded range of resonances: e.g. Fe, Eu, Sm, Sn, Dy, K, Ge, Ni, Ir, Yb.

• Gallery

Charge fluctuations by ^{174}Yb EDMS

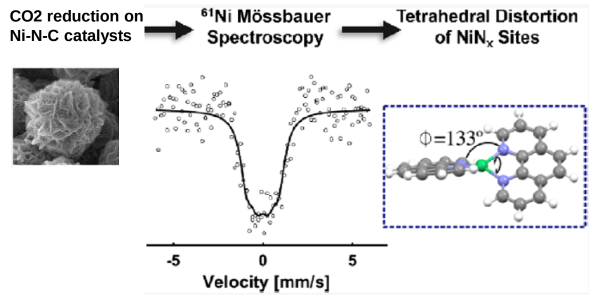
Observation of the charge fluctuations in a strange metal



Science **379**, 908 (2023).
DOI: 10.1126/science.abc478

Carbon Electrocatalysts by ^{61}Ni EDMS

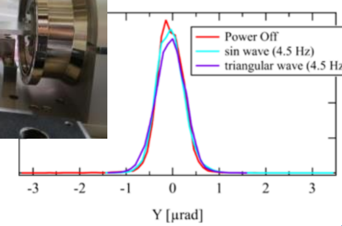
Investigation of the Structure of Atomically Dispersed NiNx Sites in Ni and N-Doped Carbon Electrocatalysts



J. Am. Chem. Soc. 144, 21741 (2022).
https://doi.org/10.1021/jacs.2c09825

Synchrotron Mössbuer Source

Synchrotron Mössbauer source of ^{57}Fe is under development with special device for public use



Work in prgress

• Toward SPring-8-II

Better spatial resolution can be obtained due to the higher brilliance. A synchrotron Mössbauer source of ^{57}Fe is under development for the public use at SPring-8-II. We hope to have a nm-scale beam size. Also, x-ray polarization analysis will become easier.

C2

Time Domain Interferometry - Slow Dynamics -

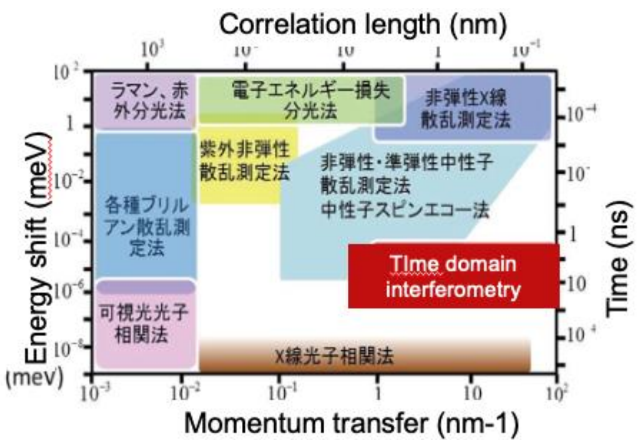
- Features

Keyword: Slow dynamics

Time Domain Interferometry (TDI) gives us the information of nanoscale dynamics from nsec to μsec. It can be applied to the soft materials such as ion liquid, ion conducting glass, rubber, liquid crystal and Membrane protein. This is basically the only alternative to Neutron Spin Echo (NSE) spectroscopy and is in fact better because it allows new access to materials with hydrogen and small samples.

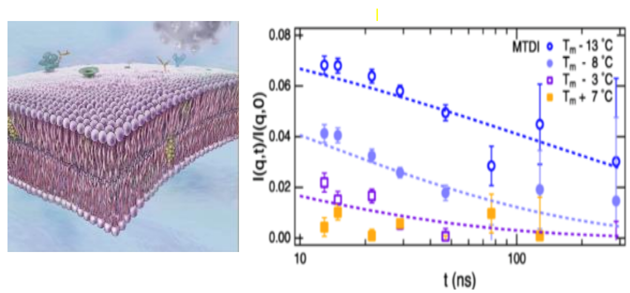
- Gallery

Energy and momentum region



Biomembranes

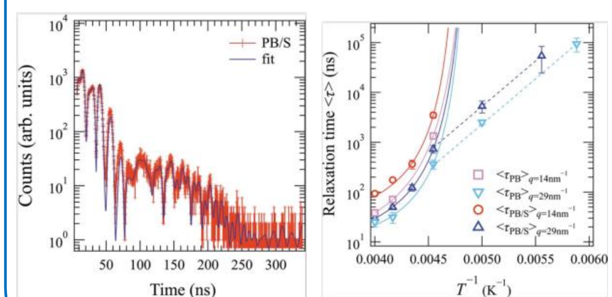
Better understanding properties of biomembranes essential for life activities



Phys. Rev. Lett. **127**, 078102 (2021).
https://doi.org/10.1103/PhysRevLett.127.078102

Tire Rubber

Microscopic observation of the effects of elongation on the polymer chain dynamics of crosslinked polybutadiene



J. Synchrotron Radiat. 29, 1180 (2022).
https://doi.org/10.1107/S1600577522007998

- Toward SPring-8-II

Better spatial resolution can be obtained due to the higher brilliance. The gamma ray quasi-elastic scattering in energy domain is also under development by SACLA/SPring-8 Basic Development Program.

C3

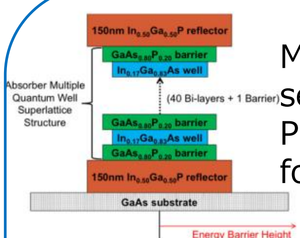
Thermal conduction and phonons BL35XU, BL43LXU

• Features

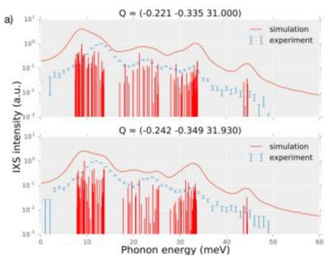
Thermal conduction in solids is carried out by electrons and phonons. Thermoelectric materials are required to exhibit high electrical conductivity but low thermal conductivity. Conversely, electrical insulators with high thermal conductivity are needed in, e.g., electronic circuits. In designing such materials, inelastic X-ray scattering is used to evaluate phonon properties. Such materials are sometimes synthesized in thin films, where IXS is one of the few probes that may be utilized.

• Examples

Phonons in multilayers



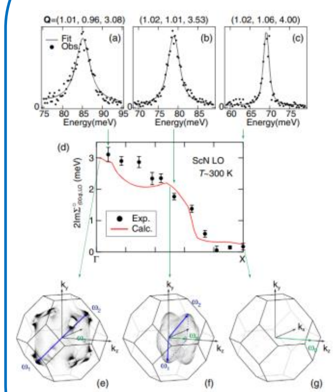
Multilayer thinfilm of semiconductors
Phonon engineering for thermo-electrics

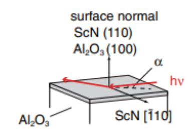


Consistency between calculations (red) and experiments (blue)

Appl. Phys. Lett. **110**, 043102 (2017)

Phonon Life time in ScN thin film

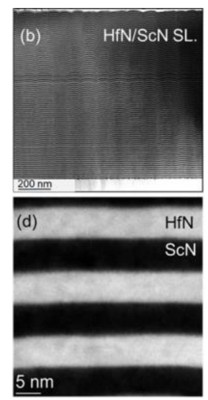




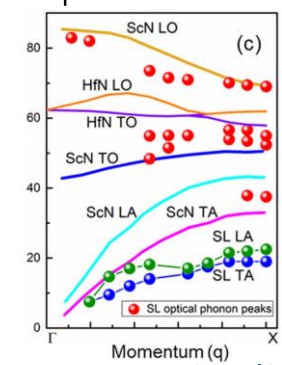
Measured phonon linewiths of ScN thin film can be explained 3-phonon model.

Phys. Rev. Lett. **120**, 235901 (2018)

Kohn anomaly in supper lattice



Kohn anomaly which is not observed in balk samples



Appl. Phys. Lett. **117**, 111901 (2020)

• Toward SPring-8-II

The high brilliance of SPring-8-II makes the measurements easier: the footprint the beam on the sample becomes smaller and the beam becomes more collimated. Consequently, it more sophisticated experiments may be realized, and, in particular, the region of total external reflection may be used to provide information with ~1nm sensitivity.

C3

Dynamics of Complex Materials

BL35XU, BL43LXU

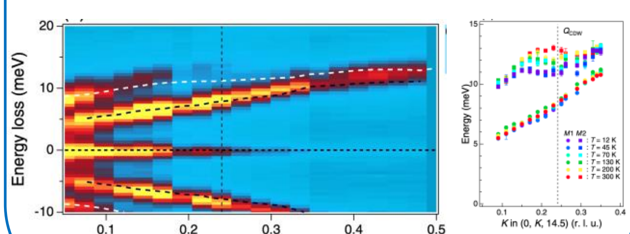
- Features

IXS serves a diverse community that investigates the interplay different degrees of freedom in complex materials: the properties of many interesting and technologically relevant materials arise from the interplay of lattice dynamics with electronic order, magnetic order, structure and thermal condutivity. The small x-ray small beam size allows investigations of phonons in samples that can not be measured by other methods.

- Examples

Common Softening in Cuprates

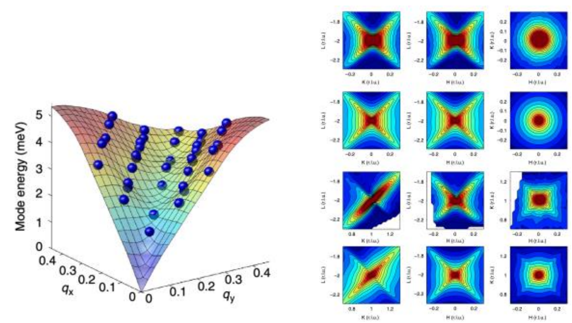
CDW instability, and related phonon softening is present in multiple families of cuprate superconductors



Phys. Rev. X **8**, 011008 (2018)

Microscopic Mechanism of Anti-Ferroelectricity

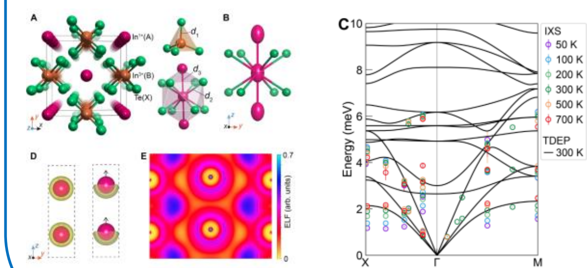
Phonon softening and diffuse scattering show antiferro-electricity come from a missed phase transtion.



Nature Comm. **4**, 2229 (2013)

Electrons Stopping Phonons

Combined structural/dynamics study shows lone pair electrons cause anharmonicity and reduced thermal conductivity



Angew. Chemie **62**, e202218458 (2023)

- Toward SPring-8-II

Smaller, faster, more extreme: The reduced beam size at SPring-8-II will make smaller samples easier to measure and permit access to different and potentially more extreme (higher temperatures, high pressures) conditions.

C3

Dynamics of Disorder

BL35XU, BL43LXU

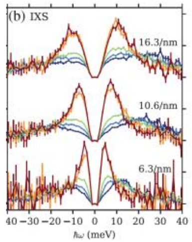
- Features

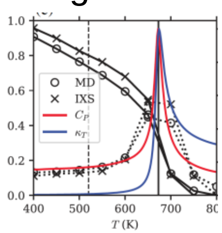
Disorder remains one of the frontiers of science. Materials, such as liquids and glasses, that are not periodic, are technologically important and poorly understood. At large length scales, they can be treated as continua, but as one approaches atomic length scales, the "mesoscale", such treatments fail. Inelastic X-Ray scattering with extremely high resolution ($\Delta E/E<10^{-7}$) provides a unique window on this region, unmatched by any other technique. It promotes new understanding of the mesoscale, including both identification of commonalities in behavior and discovery of new scienfic concepts.

- Examples

Gas-like liquids and liquid-like gases

Liquid near the critical point has regions that are dynamically gas-like



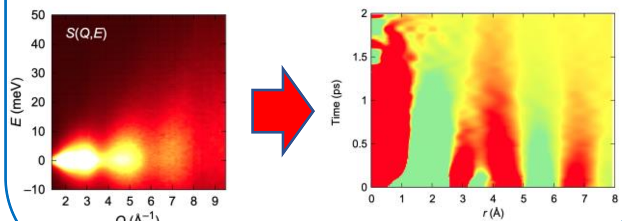


In water, this can be related to the hydrogen bonds

Phys. Rev. Lett. **125**, 256001 (2020)

Space-Time View of Liquids

A new approach show shows how looking at dynamics directly in real space allows on to gain inight – in this case the interaction of neighboring water molecules

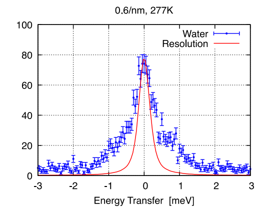


Science Adv. **3** e1603079 (2017)

Measuring where no one has measured befor

XIXS: Extreme Resolution IXS

0.38 meV FWHM World leading energy resolution for meso-scale liquid dynamics



Work in Progress

- Toward SPring-8-II

Extreme resolution will bridge the gap: Despite great effort, there is now no technique that bridges the gap between the length scale of ~60 nm (light scattering) and ~6 nm (IXS). The improved source characteristics at SPring-8-II will allow IXS to approach the light-scattering limit and provide access to dynamics that have been impossible to measure previously.

Deep Earth Investigations

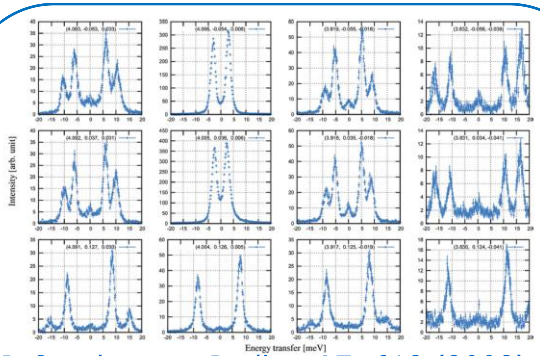
BL35XU, BL43LXU

- Features

The detailed chemical composition and temperature conditions of the Earth's interior are constrained by comparing the density and elastic wave velocity of candidate materials under high temperature and high pressure conditions in the laboratory with the observed values of the Earth. In order to do so, the technique to measure precise velocities under extreme conditions and pressure scales are required.

- Examples

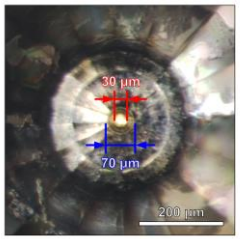
Absolute pressure scale with single crystals

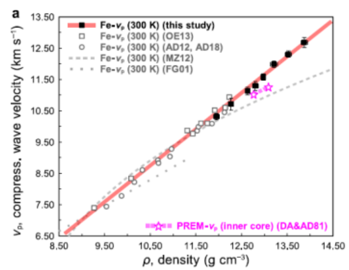


J. Synchrotron Radiat. **15**, 618 (2008)
Jpn. J. Appl. Phys. **56**, 095801 (2017)
Comptes Rendus Geosci. **351**, 236 (2019)
High Pressure Res. **40**, 465 (2020)
J. Appl. Phys. **132**, 055902 (2022)

sound velocity at pressure of earth's core

Iron at 300 GPa, the pressure at the inner core

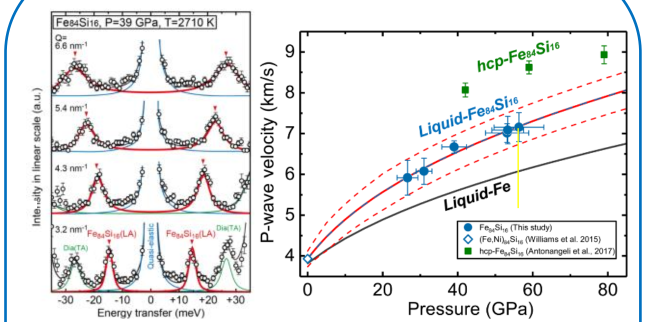




Density and longitudinal sound velocity of iron

Nature Comm. **13**, 7211 (2022)

Elastic wave velocity of Fe-alloy liquid



Silicon depleted outer core deduced from the sound velocity of Fe-Si alloy liquid

J. Geophys. Res. B **125**, 019399 (2020)

- Toward SPring-8-II

SPring-8-II will enable measurements of smaller samples and in more extreme conditions, allowing us to more close approach the conditions of Earth's core

C4

BL47XU

X-ray fluorescence holography

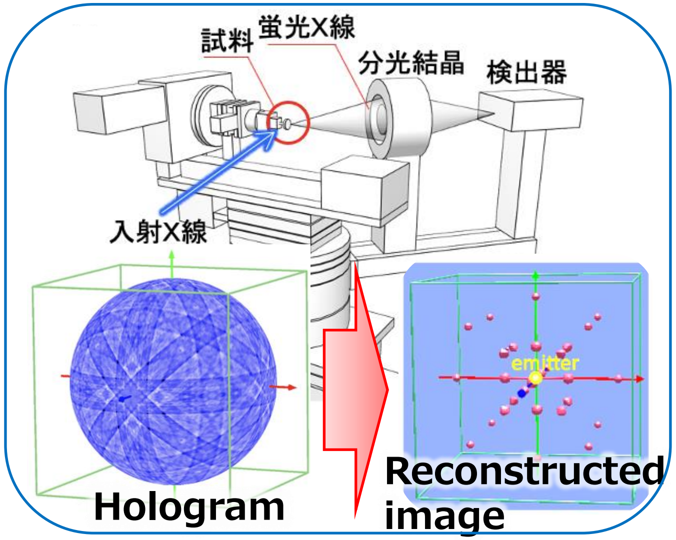
Keyword : 3D atomic image

• Features

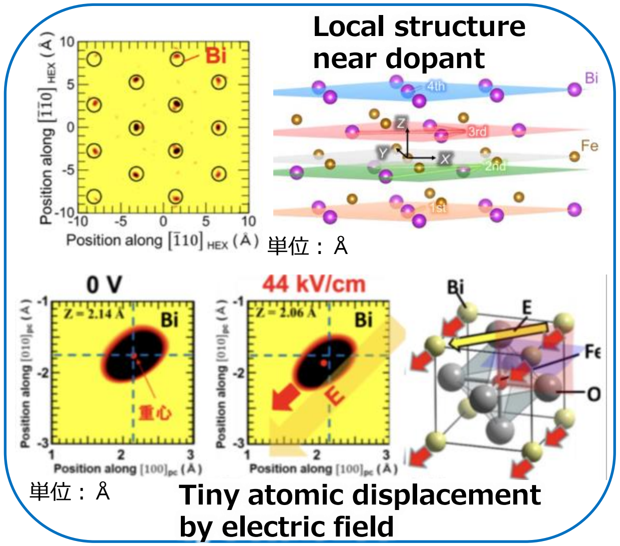
Holography has been widely used for 3D image reproduction in visible light, preventing counterfeiting of banknotes etc. Our hard X-rays enables element-selective 3D atomic images, which are used to analyze the local structure e.g. around specific additive elements in crystals of semiconductors, or around metallic elements in proteins. Our portable equipment having a large sample space is useful for detection and visualization of tiny deviations from the periodically ordered structures.

• Examples

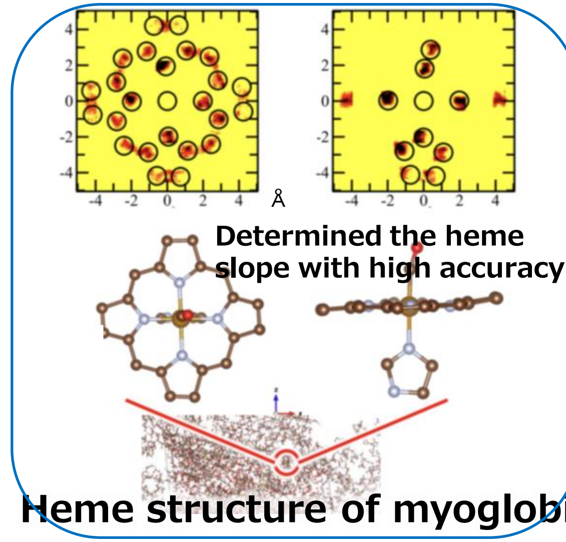
Setup for X-ray fluorescence holography



Visualization of atomic displacement around additive elements



Visualization of biosamples



• Towards SPring-8-II

Atomic scale visualization of intermediate phases between crystals and amorphous structures will contribute to creation of new functional materials. Our nanobeam will visualize local structures and structures within a single domain related to the functions of materials such as micron- and nano-sized crystal grains, porous zeolites, as well as composite defects and in crystal-like glasses.

C5

Compton scattering imaging

BL08W, BL05XU

Keywords: Operando observation, Digital twin

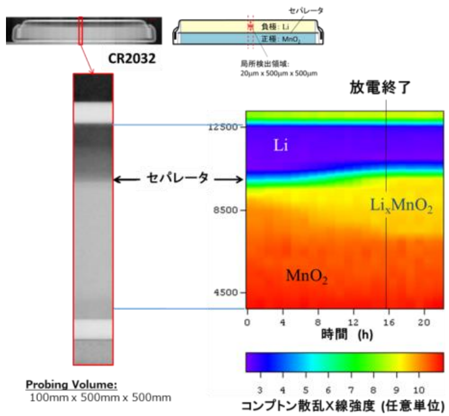
- Features

Observation of a cross-section of the object without rotation is useful in analyzing the inside of a palm-sized object. A storage battery is built into a system and is difficult to rotate and observe under operation. Compton scattering imaging enables non-destructive observation of the inside of a sample without rotation.

- Examples

Observation of a cross section of a battery without rotating it

Li transfer

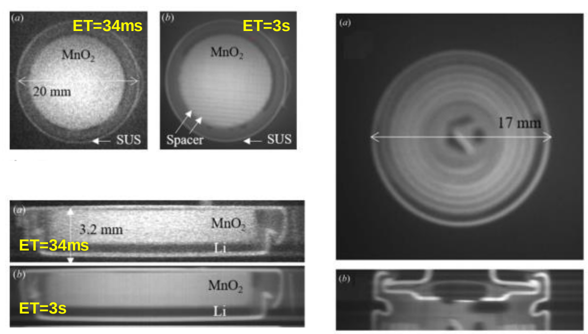


J. Synchrotron Rad. **22**, 161-164 (2015)

Sensitive to light elements

Internal structures of battery products

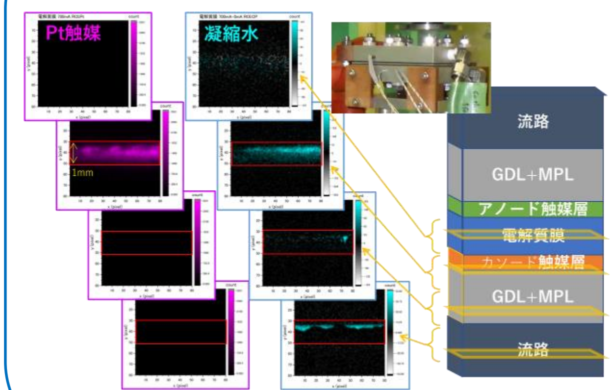
CR2032 CR123A



J. Synchrotron Rad. **28**, 1174-1177 (2021).

Simultaneous X-ray fluorescence analysis of heavy elements

Pt catalyst and water condensate distribution inside fuel cells



Appl. Sci. **13(19)**, 10753 (2023)

- Towards SPring-8-II

The new light source will enable high-speed observation that contributes to constructions of digital twin products and validation of phenomenon simulators.

D1 Multi-purpose X-ray diffraction

BL13XU, BL19B2

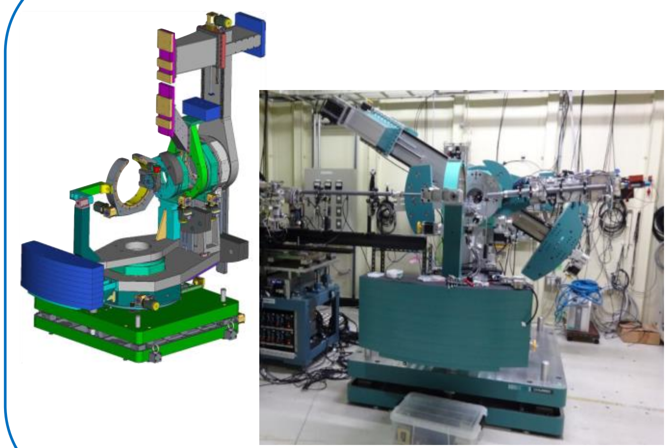
Keywords: Multi-axis diffractometer, in-situ, operand measurement, mapping, customized

- Features

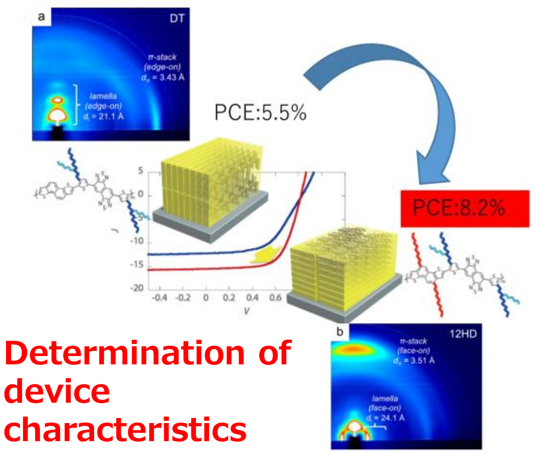
Our multi-purpose X-ray diffractometer has a large sample space, high load capacity, and a high degree of freedom in angle and position control. Various X-ray scattering and diffraction measurements are available with selectable detectors. Setups include parallel optics, 0D + slit/spectroscopic crystal, 1D, and 2D at X-ray energies of 5-72 keV. Various sample environment control devices can be mounted on the sample stage such as in-situ observation (heating/cooling, tensile, light irradiation, etc.), operando measurement (charging/discharging, voltage application, etc.), and mapping measurement of thin film, bulk, and practical materials.

- Examples

Multi-purpose 6-axis X-ray diffractometer



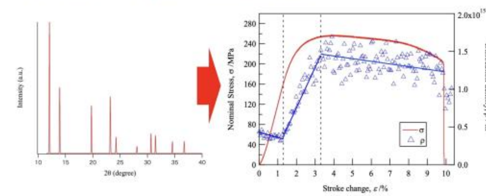
Evaluation of crystallinity and orientation of organic thin films



I. Osaka, *et al.*, J. Am. Chem. Soc. 2013, 135, 8834–8837

Evaluation of microstructural changes in metallic materials during deformation

Mechanism of deformation in structural materials



回折プロファイルの変化　転位密度変化の評価

- Towards SPring-8-II

High brilliance X-rays will accelerate the development of semiconductor devices, structural materials, and various other practical materials through faster and more efficient operando measurements as well as mapping measurements for analyzing mechanisms of manufacturing processes.

D2

X-ray **Powder Diffraction**

BL13XU, BL02B2, BL19B2

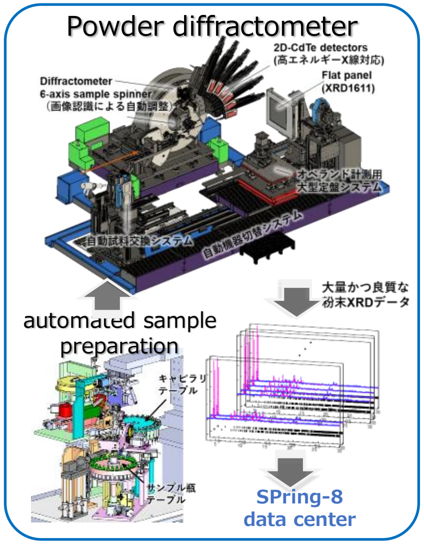
Keyword: Automated measurement, Operando, Crystal structure analysis

- ## Features

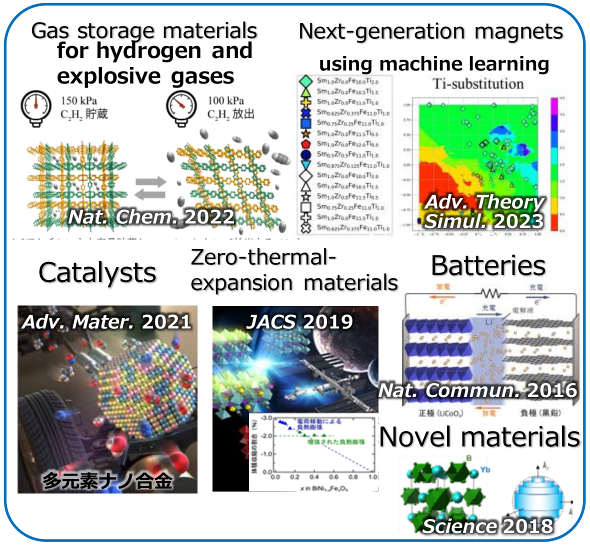
X-ray powder diffraction measures structures of crystals and their local properties. Our automated system, sample preparation to measurements, provides high-quality data over a large area of reciprocal space with high angular resolution. Short time measurements can be done even with small amounts, ~mg, of sample. Operando measurements are available for fast phenomena, which are transient or repetitive, with time resolution from seconds to milliseconds and in a variety of sample environments.

- ## Examples

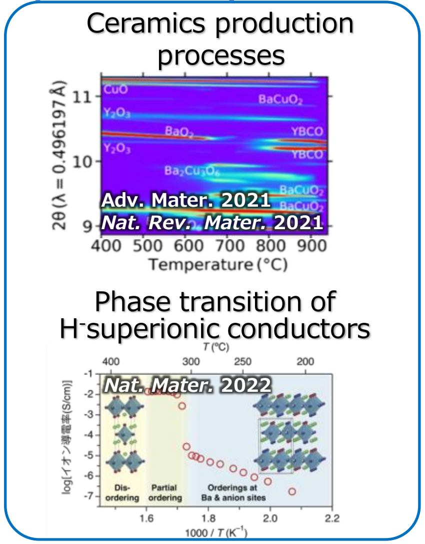
Automated powder diffraction system



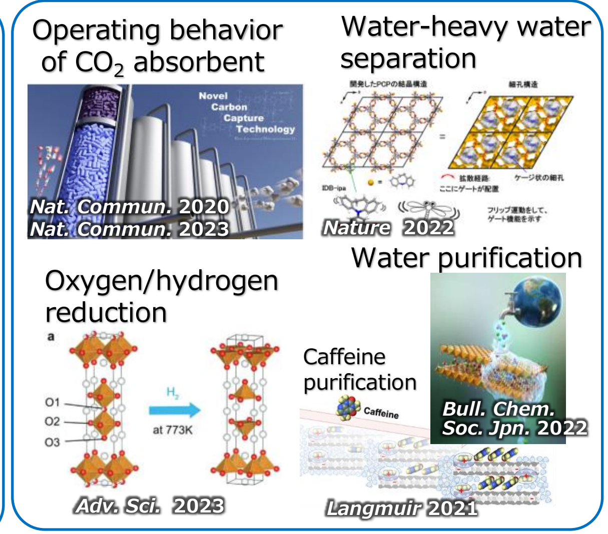
Creation and utilization of large volume and high quality data



Observation of material production processes



Operando observation of decarbonization and new energy materials



- ## Towards SPring-8-II

The high-brilliance beam will increase the number samples to ~1000 samples/day. The high-quality data automatically transferred to the data center, leading to next-generation DX with AI and materials informatics (MI). Operando analysis will enable high-speed observation of micro regions with sub-millisecond time resolution. This will advance the understanding of various reaction processes.

D3

High-throughput PDF, in-situ PDF

BL04B2, BL08W, BL05XU

Keywords: Local structure, High-speed measurement

• Features

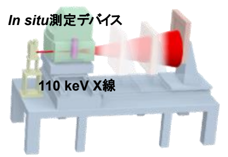
High-energy X-rays allow pair distribution function (PDF) analysis that gives quantitative data on local distortion in materials. It has been applied to many research fields from earth science to industrial uses such as rechargeable batteries and environmental catalysts. Our system offers high-throughput PDF measurements that are 10-50 times faster than the conventional systems. High-precision in-situ PDF measurements are also possible under extreme pressures such as those inside the earth.

• Examples

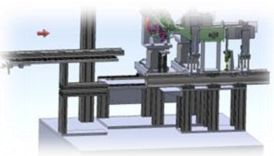
PDF measurement systems

7 sequential detectors

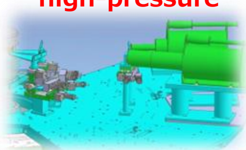
Time-resolved



High-throughput



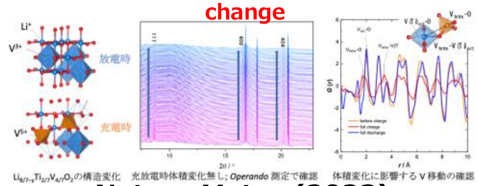
High-energy X-ray high-pressure



applicable to various fields of research

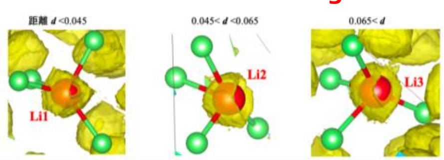
Next-generation rechargeable batteries

Structure of V-based cathode materials for rechargeable batteries without volume change



Nature Mater.(2023)

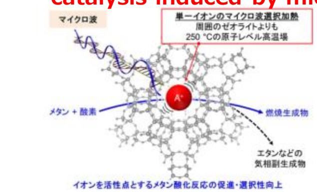
Structure of solid electrolyte in collaboration with Fugaku



Energy Environ. Mater.(2023)

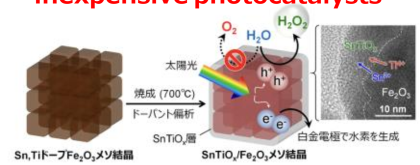
Environment catalysts

Visualizing the mechanism of selective catalysis induced by microwave



Sci. Adv. (2023)

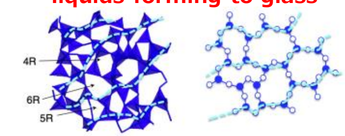
Structure of materials for inexpensive photocatalysts



Nature Commun. (2022)

High-temperature fusion

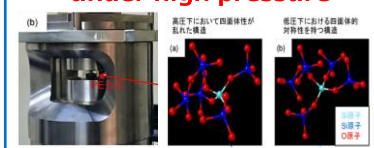
In-situ measurement of molten liquids forming to glass



Nature Commun. (2014)

Earth science

In-situ measurement of SiO_2 under high pressure



Nature Commun. (2022)

• Towards SPring-8-II

Innovation of PDF analysis, including in-situ measurements, will be realized by a pink beam having 100 times higher flux, which accelerate materials development. Collaboration with Fugaku supercomputer will establish ecosystems contributing to SDGs through further development of material informatics (MI).

D4

Fast/precise structure analysis on micro single crystals

BL05XU, BL02B1

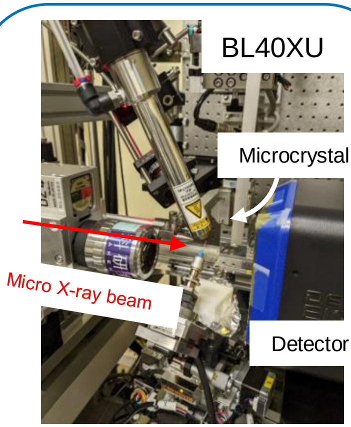
Keywords: Precise structure analysis, High throughput

• Features

Single crystal structure analysis determines molecular and atomic structures, and is widely used in research on organic/inorganic materials, hybrid materials, and in drug discovery. Our technique using micrometer size X-ray beams enables structure analysis of organic micro single crystals, precise measurements of electronic density at high spatial resolution, and in-situ single crystal structure analysis under external perturbations from light, heat, and pressure.

• Examples

Microcrystal structure analyzer



Collimated X-ray beam of micrometers

Molecular structures of functional materials

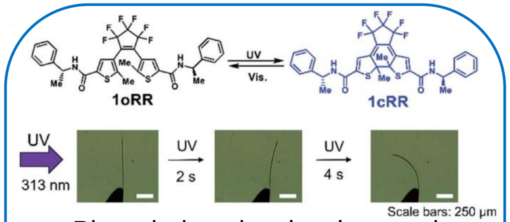
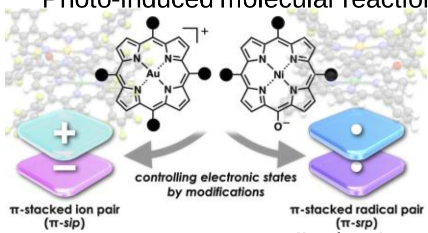
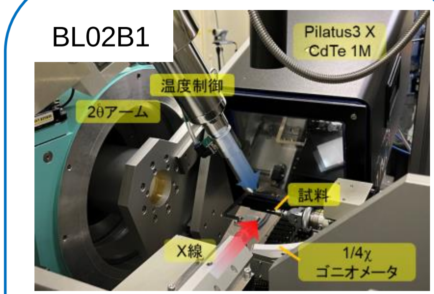


Photo-induced molecular reactions



Heterogeneous radical pair stacking structures

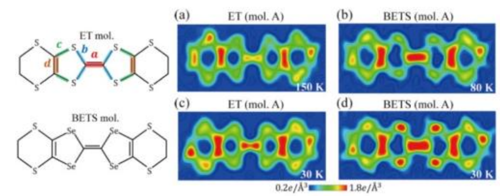
Precise structure analysis system



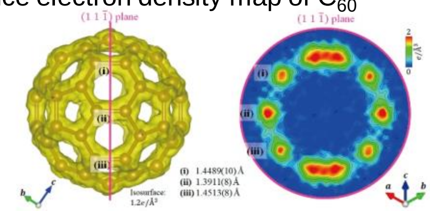
X-ray energy is 50 keV（λ=0.248 Å）

Electron density distribution at high-resolution

Precise analysis on density of Dirac electrons in organic molecules



Valence electron density map of C_{60}



• Towards SPring-8-II

High-brilliance X-rays and a new 2D detector CITIUS will enable higher precision measurements of smaller, submicron, microcrystals. AI-equipped automatic micro single crystal sampling systems and sample exchange robots will provide high throughput analysis of 150 micro single crystal per day, which is more than 10 times what is possible now. This will allow more efficient searches for target materials and drug discovery.

D5

Nanobeam X-ray diffraction/topography

・BL13XU

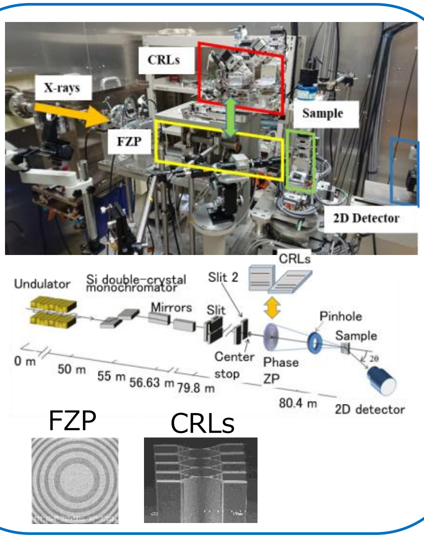
Keywords : Lattice deformation, mapping

• Features

Zone plates and refractive lenses allow an x-ray beam to be focused down to sub-micron spot sizes, enabling local analysis of crystal structures and lattice deformation. Our method has been used to analyze lattice distortion caused by microstructure formation and strain distribution around lattice defects in semiconductors and related device materials. Recently the method has been extended to structural materials and polymers.

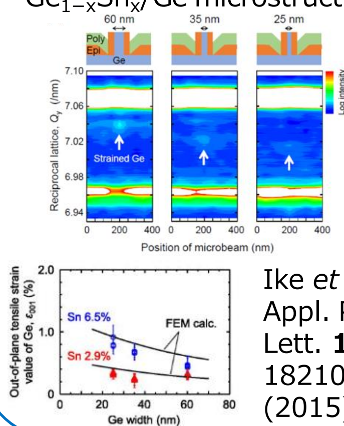
• Examples

Experimental setup



Evaluation of local strain inside semiconductor microstructures

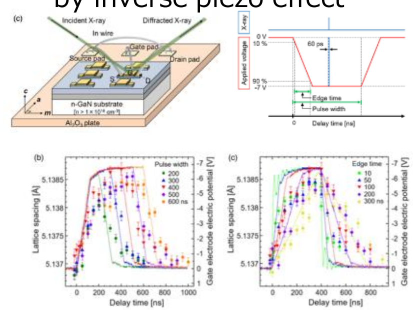
Evaluation of local strain inside embedded $Ge_{1-x}Sn_x$/Ge microstructures



Ike *et al.*, Appl. Phys. Lett. **106**, 182104 (2015).

Operando measurement by time-resolved nano XRD

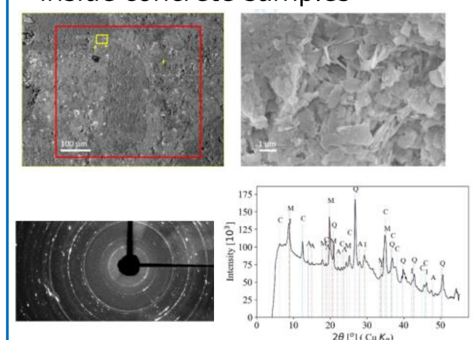
Analysis of lattice deformation in AlGaN/GaN by inverse piezo effect



H. Shiomi et al., Appl. Phys. Express **14**, 095502 (2021).

Local crystal analysis inside materials

Distribution of crystalline phases inside concrete samples



A. Aili et al., J. Am. Ceram. Soc. **105**, 6924 (2022).

• Towards SPring-8-II

The improved X-ray nanobeam intensity, 100 times higher than now, will enable mapping of a larger number of samples including polycrystals and microcrystals in a short period of time. The nanobeam will also be further miniaturized to achieve a resolution of several tens of nanometers. X-ray topography, as a complimentary method, will also be further improved for digital topography with a large-angle camera having a higher resolution.

XRD imaging/3DXRD

BL47XU, BL05XU

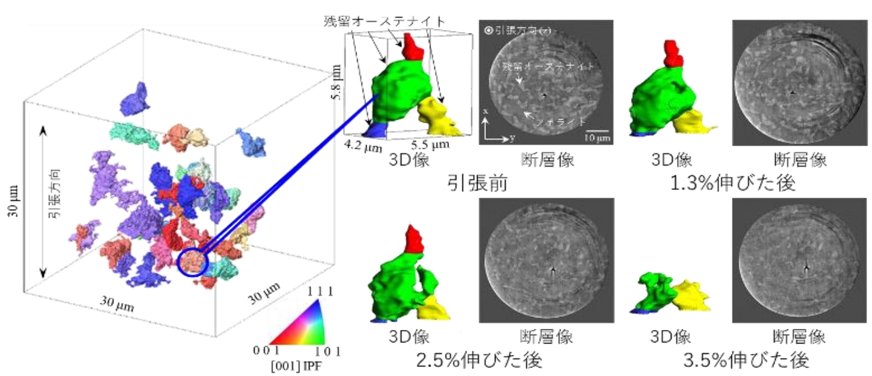
Key words: Grain, Orientation mapping, Non-destructive, Operando

- Features

X-ray diffraction of microbeams may be used to identify the shape and orientation of individual grains of metallic materials such as steel, aluminum alloys, and solder. Through statistical processing of intergranular orientation differences, grain shapes etc., this method has revealed the nano-scale changes of matter disturbed mechanically by external forces, temperature variations, and other factors. Utilizing high-energy X-rays with high transmission capability in the 100 keV range enables non-destructive analysis of the internal structure of practical material samples with ~ cm thicknesses.

- Features

Phase Transformation in Steel Materials

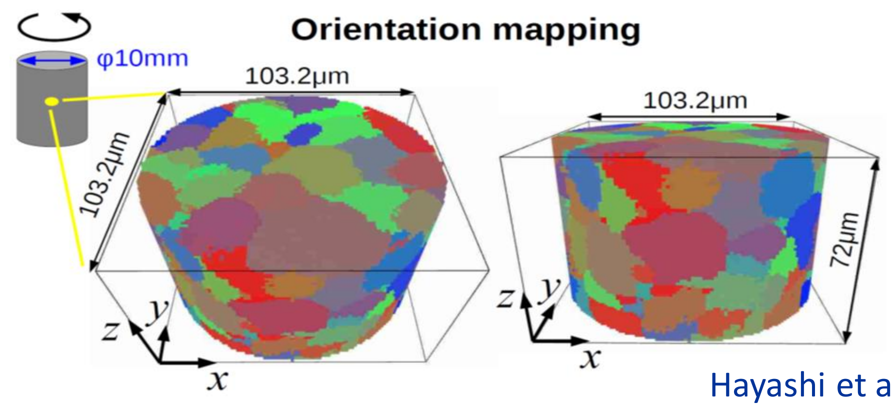


残留オーステナイトの3D像　　個々の残留オーステナイトの相変態挙動

Non-destructive 3D nano-observation of Austenite particle phase transformation in TRIP steel

Toda et al., Acta Mat. (2022).

Non-destructive Orientation Mapping Inside Iron Columns



Hayashi et al..

Non-destructive 3D mapping of grain orientations in a Φ10 mm iron rod using 100 keV X-rays

- Towards SPring-8-II

The increased intensity of the focused beam of high-energy X-rays by a factor of 100 will reduce measurement from the present ~1/2 day to just a few minutes. This will enable routine measurements of many practical materials, contributing significantly to the optimization of production processes.

Large-capacity high-pressure press

BL04B1, BL05XU

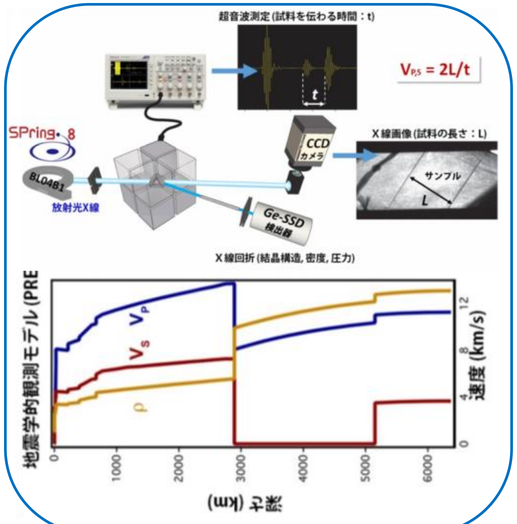
Keywords: Earth planet dynamics, Extreme condition

• Features

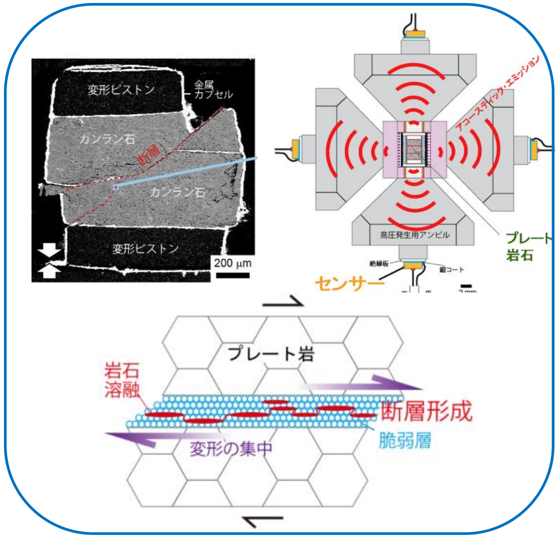
Measurements of crystal structures and physical properties under high pressure has become an important method in industrial research, earth and planetary science, and for developing novel materials such as high-Tc superconductors. Our large-capacity press system can perform precise X-ray diffraction measurements of deformation and fracture dynamics under high hydrostatic pressure and uniform heating environments. This method can reveal information such as micro-macro phenomena of samples under highly controlled deviatoric stress.

• Examples

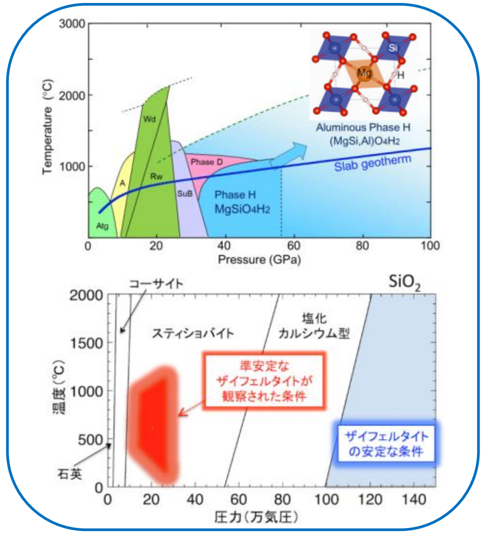
Earth and planetary deep structures



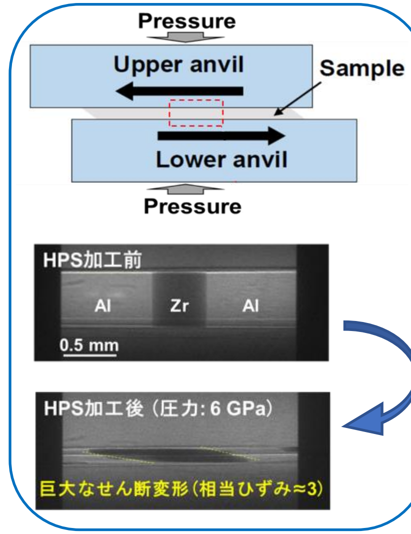
Mechanism of earthquakes



Exploring new minerals and metastable phases



High-pressure giant strain processing



• Towards SPring-8-II

The new source will enable measurements on millisecond timescales useful for understanding phenomena such as astronomical collisions as well as more mundane but extremely important tasks like observing material processing and quality control. Application to investigating earthquakes and volcanoes will give important social contributions.　It will also enable 2D/3D mapping of chemical compositions and crystal structures of naturally heterogeneous samples.

E1

Production SAXS

BL19B2, BL40XU, BL40B2, BL05XU

Keywords : SAXS, USAXS, GI-SAXS, Automated measurement, Operando measurement

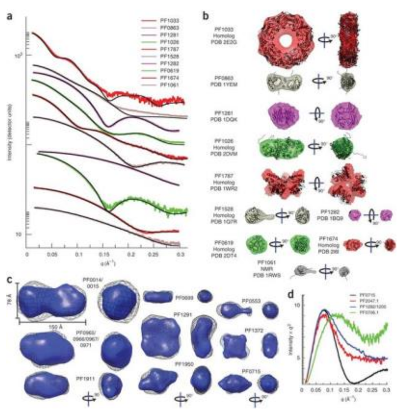
• Features

Small angle X-ray scattering (SAXS) has been used for structural evaluation of mesoscopic objects (order of 1nm to 100nm) such as microparticles, micelles and lamellae, inside materials. Our technique enables operando measurements that track structural changes in situ during reaction processes. Ultra-small angle X-ray scattering (USAXS) makes it possible to evaluate large and higher-order structures of 100nm such as aggregates. Automated measurement systems used for a large of samples and GI-SAXS that measures X-rays totally reflected near the sample surface are also available.

• Example

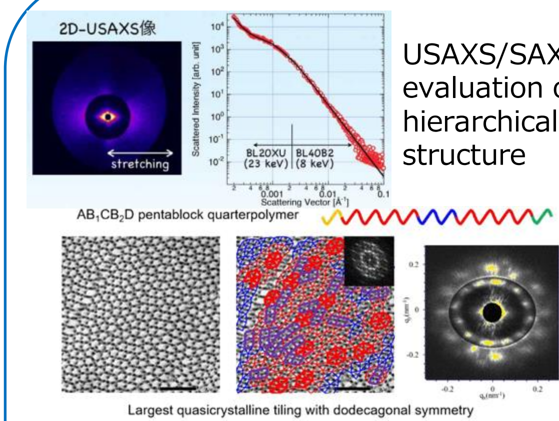
SAXS

Extract sample size, structure and surface information



G.L. Hura et al., Nat. Meth. 6, 606 (2009)

USAXS

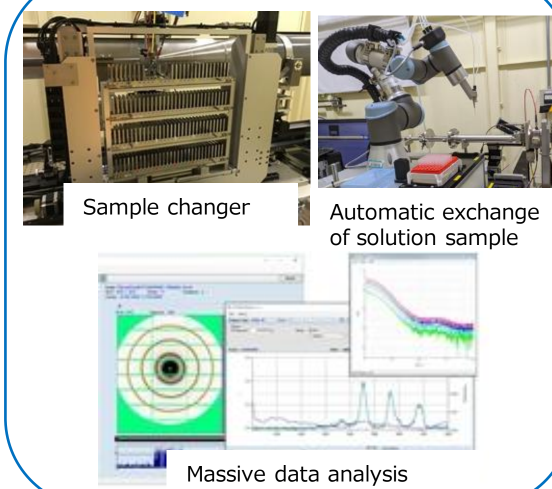


USAXS/SAXS evaluation of hierarchical structure

Evaluation of long-period structure of quasicrystals

M. Suzuki et al., ACS Nano 16, 6111 (2022)

Automated SAXS measurement system



• Towards SPring-8-II

High-brilliance will reveal the formation mechanism of higher order structures that are essential in the functions of polymer materials through higher speed operando measurements of larger volume samples. Promoting materials informatics using machine learning and compiling a database of a large numbers of SAXS images will bring innovation to the utilization of X-ray scattering data.

Imaging SAXS

Keywords: Local structure analysis, Scattering CT, Simultaneous scattering imaging

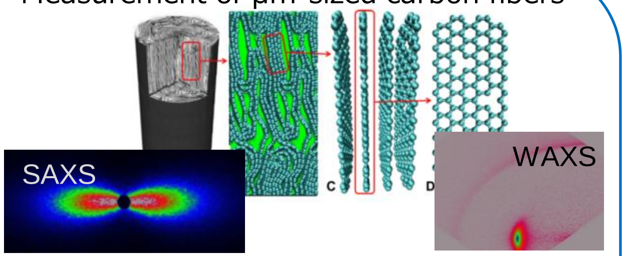
- Features

Small angle X-ray scattering (SAXS) measures structural information of nano- and micro-scale structures inside samples. Combined with other techniques such as CT, SAXS can extract local and/or 3D structural distribution by using microbeams.

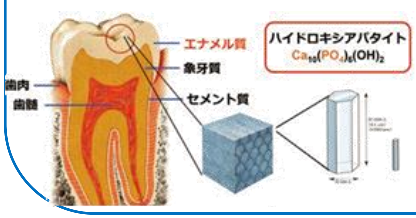
- Example

microbeam SAXS

Measurement of μm-sized carbon fibers



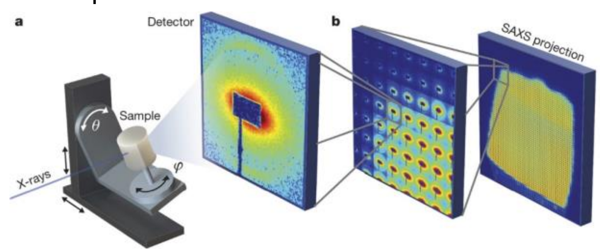
C. Zhu et.al. Carbon 235 (2012)



Microbeam analysis on tooth remineralization

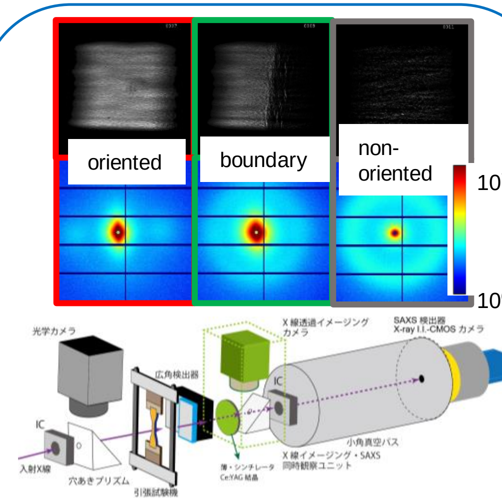
SAXS-CT

Combination of scattering measurements with CT enables evaluation of distribution of parameters (size, period, shape) inside samples.



Schaff et al. Nature 527, 353 (2015)

Simultaneous scattering and transmission imaging



- Towards SPring-8-II

Improved brightness and high-speed detector CTIUS will reduce a measurement time of SAXS-CT from 10 hours to 10 minutes. A new system will reveal the distribution of nanoscale structures in real space as well as averaged structural information. A large number of scattering and diffraction data of materials with different manufacturing methods and compositions with matching analysis would be useful in material development and forensic science.

E3

Fast SAXS/XPCS

BL40XU, BL29XU

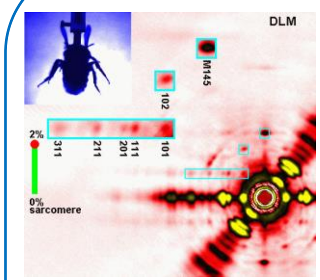
Keywords: Nonuniformity, Dynamics analysis, in-situ、in-vivo

• Features

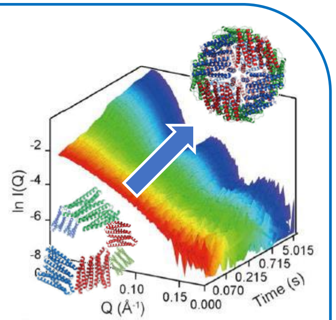
Small angle X-ray scattering (SAXS) analyzes nano- and/or micron-scale structures inside samples. It enables fast tracking of structural time evolution such as reaction processes during solution mixing, sample deformation processes during tension and compression, and structural changes by temperature. Hard X-rays with high transmission allow flexible environment around samples, as is relevant for in-vivo and in-situ measurements. Coherent X-rays enable X-ray photon correlation spectroscopy (XPCS) that measures sample hardening and softening processes due to changes in the internal structures.

• Example

Time-resolved SAXS



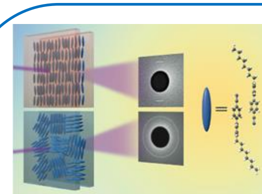
Science, 341, 1243 (2013)



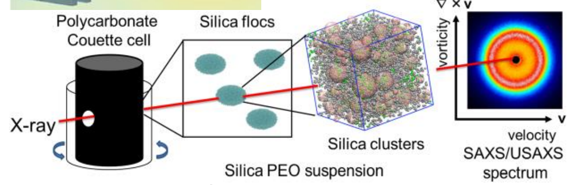
Biochem. 55, 287 (2015)

Tracking structural changes and synthesis processes

Rheology Scattering X-ray



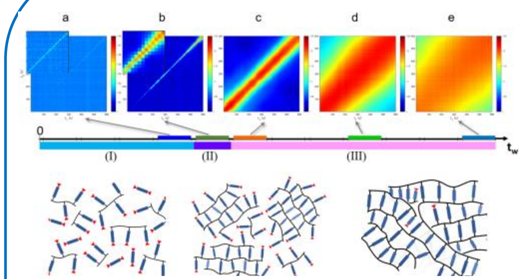
Orientation of liquid crystal molecules by shear force
Phys. Chem. Chem. Phys.,23:131 (2021)



Colloids & Surfaces A, 658, 130727 (2023)

Tracing structural changes of a sample exhibiting viscoelasticity deformed by external forces.

XPCS



Sci. Rep. 11, 9767 (2021).

Curing processes off epoxy resin. The mesh structure changes with the curing temperature.

• Towards SPring-8-II

Submillisecond resolution will be available for continuous measurements, and nanosecond resolution for pup-probe methods, covering time scales from 10^{-9} to 10^{2} seconds. High coherent flux will enable speckle image analysis for local structures, and its temporal variation will reveal the dynamics of density fluctuations and phase change processes.

Soft X-ray XAFS

BL27SU, BL17SU

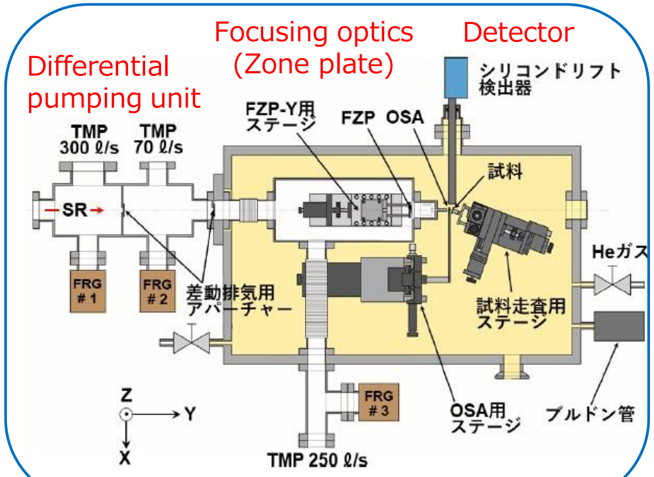
Keywords: Soft X-ray MCD, Atmospheric pressure measurement

• Features

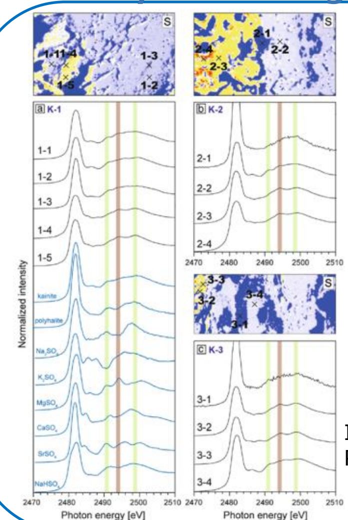
Soft X-ray XAFS enables observation of electronic states of light elements in organic materials and metals in functional materials, and covers various research fields from condensed matter physics to environmental science. XAFS imaging is also available to investigate the disribution of elements and their chemical states in high vacuum and in helium environments as well as ultra-high vacuum by using differential pumping. A fast switching technology for circular polarizations and an electromagnet of up to 2T enable highly efficient measurements of SX-MCD, allowing investigation of magnetic properties of samples with elemental sensitivity.

• Examples

Soft X-ray XAFS imaging system



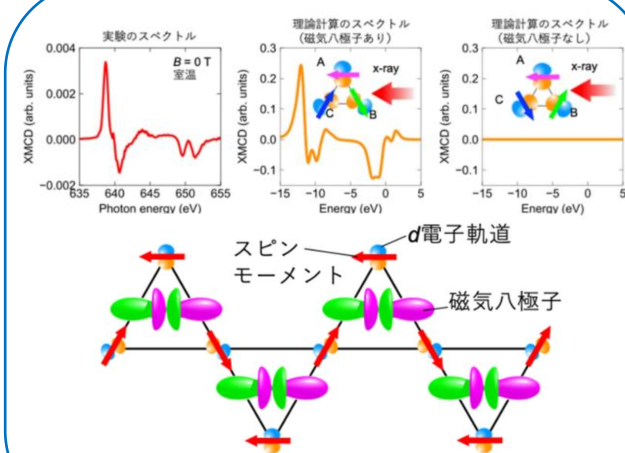
Trace elements behavior by XAFS imaging



Scanning microspectroscopy
✓ Analysis of composition of minerals
✓ Extraction of trace elements
→ 鉱物を構成する元素組成と硫黄との相関を可視化

Isaji *et al.*, Prog Earth Planet Sci. **6**, 60 (2019).

Detection of magnetic octupoles by SX-MCD



Kimata *et al.*, Nat. Commun. **12**, 5582 (2021).

• Towards SPring-8-II

XAFS imaging with high spatial resolution and high throughput will realize highly efficient measurements of spatial and temporal variations of elements and chemical states, and also contribute to revealing chemical reaction mechanisms. Collaboration with data-driven science will give new insight to 2D/3D spatial morphology and its kinetics.

F2

Soft X-ray photoemission spectroscopy, ARPES

BL25SU

Keywords: Microbeam, Space resolved measurements

• Features

Angle-resolved photoemission spectroscopy visualizes the motion of electrons as momentum-energy distributions and the chemical/electronic states of functional materials. Our setup has micro-focused intense soft X-rays that enable observation of electronic states in 3D momentum space with high energy and angular resolution. A wide range of sample morphologies, including bulk crystals, thin films, and flakes, may be measured. Real-space-resolved measurements of electronic states using micro-focused soft X-rays are also possible, as is useful for device performance analysis.

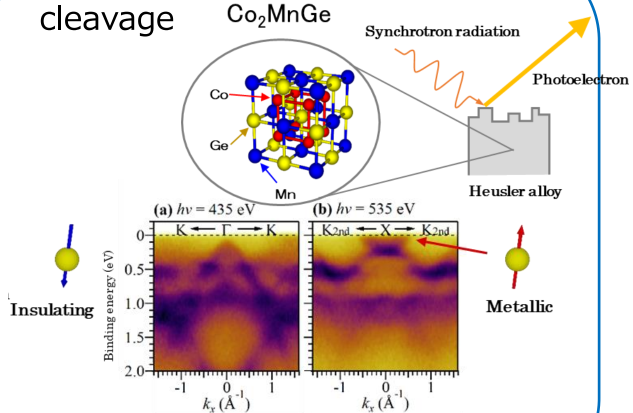
• Examples

Micro-focused soft X-ray ARPES system

Spot size: 10x10 μm^2
Energy resolution: E/ΔE < 20000
Temperature: 7 K ~ 300 K

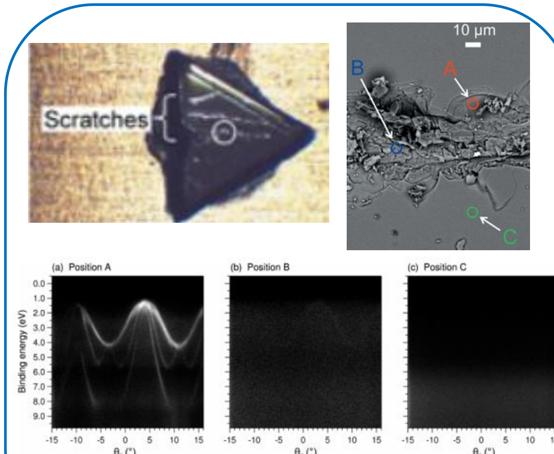
Direct observation of half-metallic band

Heusler alloys with difficult cleavage



Kono et al., Phys. Rev. Lett., **125**, 216403 (2020).

Real-space resolved ARPES measurement of silicon single crystals



Muro, *et al.*, J. Synchrotron Rad. **28**, 1631 (2021).

• Towards SPring-8-II

Soft X-rays of > 100eV will be enhanced for establishment of a platform for 3D momentum-resolved dynamics of electronic states. A denoising system using deep learning will be installed for realizing ultra-high energy resolution with high throughput.

F3 Soft X-ray imaging (Photoelectron imaging type, scanning type)

BL17SU、25SU
Keywords: Spectroscopic imaging, PEEM, SPELEEM, Nanobeam

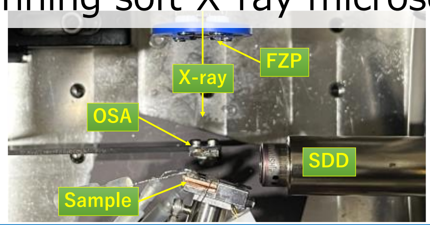
- Features

Soft X-ray imaging visualizes light elements and their chemical state distributions, and is applicable to magnets, semiconducting devices, dielectrics, strongly-correlated materials, biological samples, and organic materials as is relevant for many research fields including material science, earth and planetary science, and forensic science. Scanning the focused beam and mapping the intensity of fluorescent X-rays and excitation current reveal rich information including elemental distribution, valence states, and magnetic/charge orders at resolution of 10-10nm. Photoemission electro microscopy (PEEM) is also available that has high throughput data.

- Examples

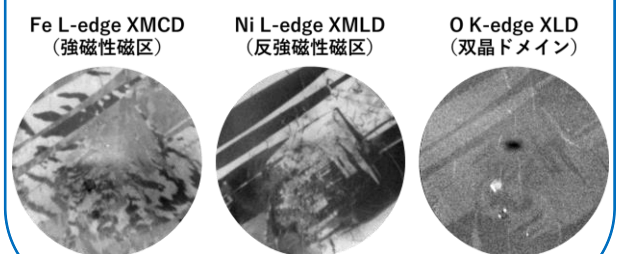
Measurement system

Scanning soft X-ray microscope



PEEM

Magnetic/charge domain structure of NiO(001)/Fe(1 nm) heterojunction



Scanning soft X-ray microscope

Chemical state analysis in microscopic region using X-rays focused several 100m

Thin, chip, and thick specimens are available if their surface is smooth.

Possible analysis:
・Low-vacuum
・He atmosphere
・Operando
・Insulator

- Towards SPring-8-II

Possible detection of tiny elemental/magnetic signals and speeding up image acquisition will improve effective spatial resolution. High flux beam with high throughput will elucidate the mechanism of emergent functions and facilitate data mining by machine learning.

G1

Prodution CT·Automated CT

BL20B2, BL28B2, BL05XU

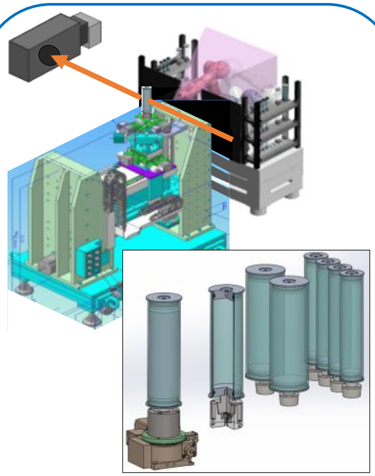
Key words: mirco-CT

- Features

Non-destructive imaging by X-ray CT is widely used for medical, industrial, and security inspection. SPring-8 enables high-spatial resolution CT images for thick samples by using intense, high-energy X-rays. Sophisticated X-ray cameras allow the FOV to several-cm with a pixel size below several microns. Large and/or precious samples, even objects of cultural heritage, can be analyzed non-destructively, by sending them to SPring-8. Our automated CT instrument provides high-resolution 3D reconstruction images. As it is non-destructive this can be useful as pre-analysis before conducting applying methods.

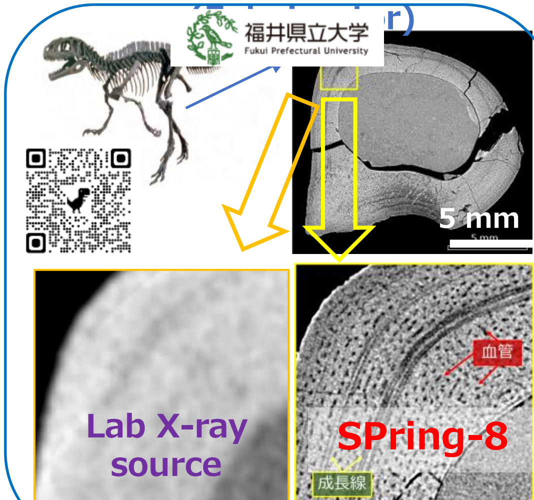
- Examples

Instrument



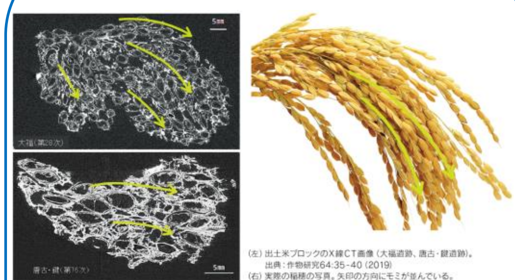
Dedicated capsule

Fossil of a dinosaur



Imai et al., JSR (2023).

Cultural heritage

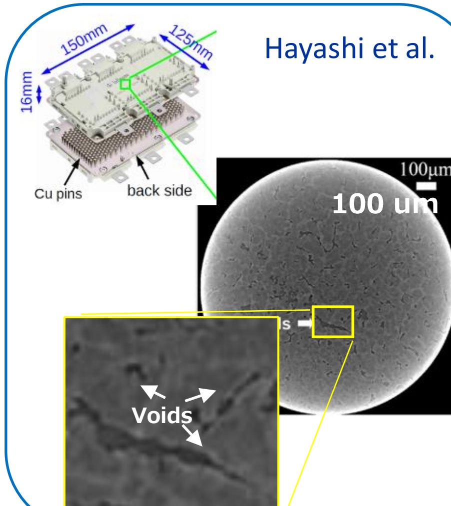


Understanding the Rice Cultivation Practices in the Yayoi Period from CT Scans of Excavated Rice Blocks

Inamura et al., 作物研究 (2016).

Soldering in power modules

Hayashi et al.



Towards SPring-8-II

The utilization of X-rays with high penetration capabilities will be enhanced. The captured data will be automatically transferred to the SPring-8 Data Center. This will facilitate advanced analyses such as 3D segmentation and functional analysis, as well as Region of Interest (ROI) analysis for extracting specific areas of interest. Collaboration with th e 'Fugaku' supercomputer will contribute to various areas such as big data analysis of infrastructure materials, failure analysis of components and products, and the digitization and cataloging of fossils and cultural artifacts.

G2

Nano CT/Ptychography

BL20XU, BL47XU, BL29XU

Key words: Multiscale CT, Operando

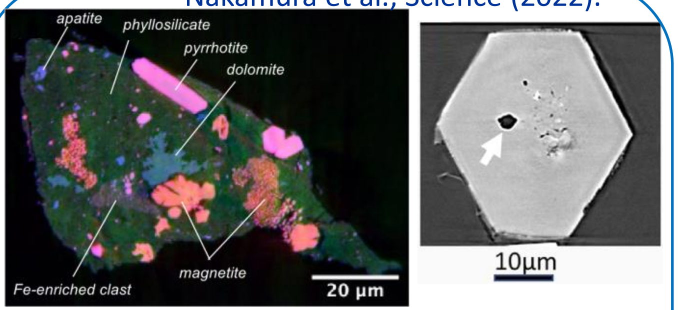
• Features

X-ray CT enables the visualization of the 3D internal structure of objects. Our technique combines Nano-CT and Micro-CT to visualize the interior of samples at a resolution of 100 nm for millimeter-sized specimens. This technique, known as multi-scale CT, is particularly effective for high-energy X-rays above 15 keV. Recent advancements in ptychography, utilizing coherence, have led to measurements with high resolution below 10 nanometers. Applications are broad, including asteroid samples and neural cells through the depiction of intricate three-dimensional structures without disrupting the areas of interest.

• Examples

Particles from Asteroid Ryugu

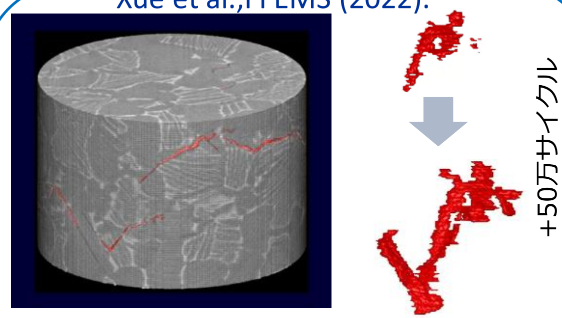
Nakamura et al., Science (2022).



Various minerals and organic matter are present in a fine-grained matrix composed of layered silicates. Voids within pyrrhotite and calcium carbonate particles contain fluids such as H2O-CO2

Direct Observation of Crack Propagation in Titanium Alloy

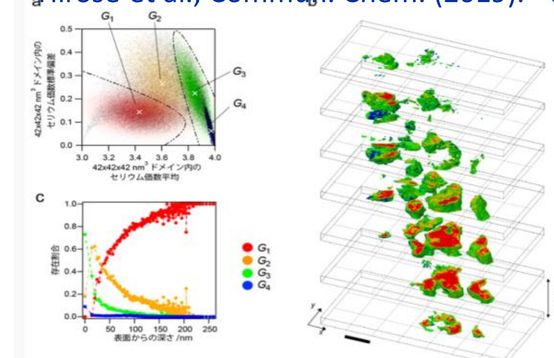
Xue et al.,FFEMS (2022).



Direct observation of ultra-high cycle fatigue in Ti-6Al-4V. The initiation point of fracture was identified, and the progression of the fracture was captured.

Nanoscale Visualization of Catalytic Functions

Hirose et al., Commun. Chem. (2019).



Ptychography XAFS-CT enabled to visualize the 3D distribution of oxidation states in catalytic particles at the nanoscale

• Towards SPring-8-II

Higher brightness resulting in higher spatial resolution and phase sensitivity in nano-CT fills the resolution gap with transmission electron microscope CT (TEM-CT), and enables 3D observations at various scales. The research areas would expand into biomimetics, unraveling and utilizing the 3D microstructures and functions in living organisms. Fully coherent X-rays and the CITIUS detector will realize ptychography measurements with sub-nanometer resolution on a routine basis, enabling nano-level measurements of crucial materials such as semiconductors.

G3 High-speed imaging & CT

BL20B2, BL47XU, BL05XU

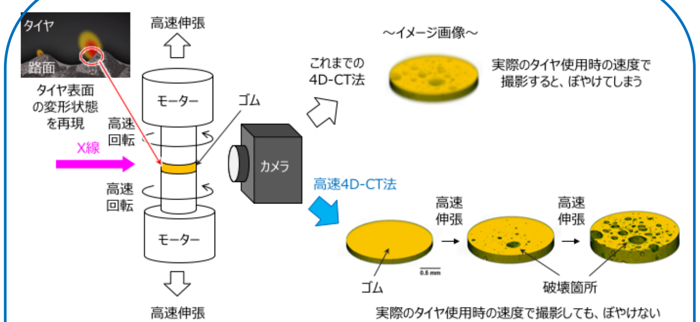
Key words: Operando CT、High-speed movie

- Features

High-speed cameras for the visible light cannot access the interior of objects. At SPring-8, intense X-rays and high-speed X-ray cameras enable high-speed video recording and operando CT measurements of transient or repetitive fast phenomena with a time resolution ranging from milliseconds to microseconds. This capability enables the detailed capture of rapid deformations inside objects, the mechanisms of processing, and the movement of biological entities. Our technique is applicable to a wide range of fields such as material development, engineering and medicine.

- Examples

Deterioration due to High-Speed Deformation in Rubber

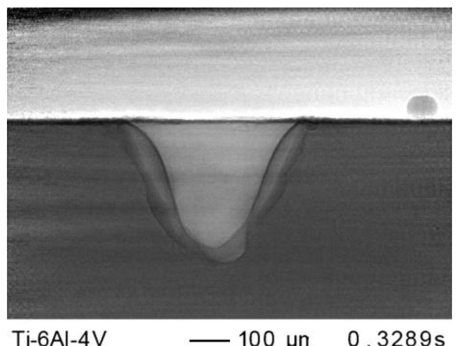


住友ゴム

東北大学

Mashita et al., JSR (2021).

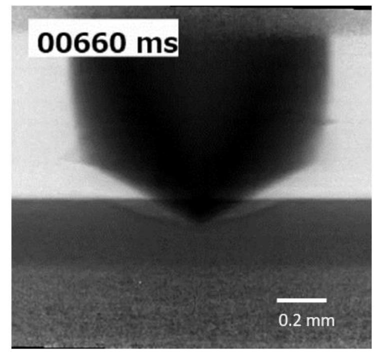
Observation of the Metal Melting and Solidification Process



Observation of metal melting and solidification phenomena induced by laser heating at speeds exceeding 10 kHz.

九州大学 KYUSHU UNIVERSITY

Understanding the Principles of Precision Machining Processes



Mimura et al.

東京大学 THE UNIVERSITY OF TOKYO

Elucidating the principles of various precision machining processes through ultra-high-speed, non-destructive measurements at the order of 10,000 fps

- Towards SPring-8-II

Improved brightness, temporal and spatial resolution will enable microsecond timescale analysis for sub-micron regions. This will contribute to ultra-precise and high-speed advancement of manufacturing by revealing the true principles behind conventional phenomena such as material processing, fracture, and dielectric breakdown.

Protein Crystallography (Single Crystal Diffractometer)

BL26B1, BL32XU, BL41XU, BL45XU

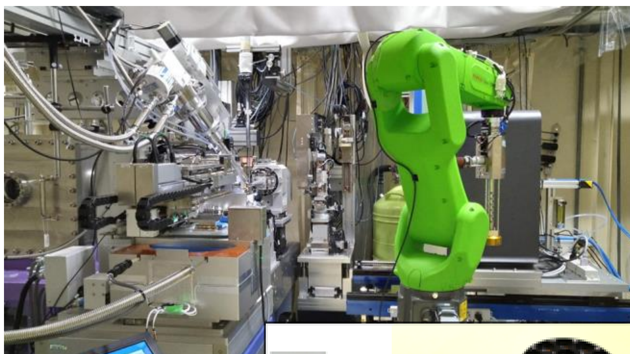
Keyword: Automated measurement

- Features

X-ray protein crystallography is a fundamental technique in structural biology and drug discovery. High-brilliance X-rays, sample exchange robots, and high-speed detectors with high sensitivity make it possible to obtain high-resolution data in a short period. Frozen crystal samples housed in world-standard sample pins and cassettes can be sent to us for automated or remote measurement. Ultra-high resolution measurements using short X-rays (~30 keV) are also available.

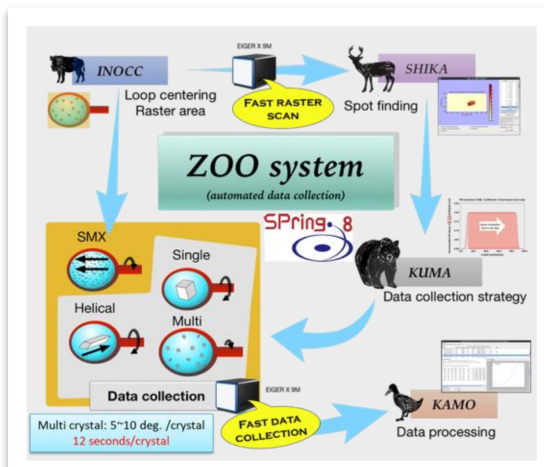
- Examples

Automated diffractometer

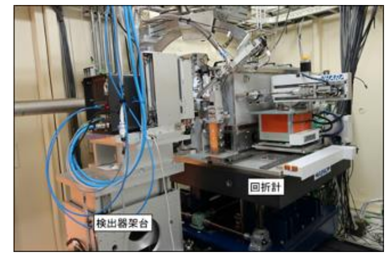


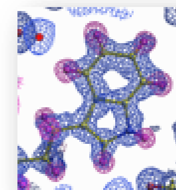
Sample loading

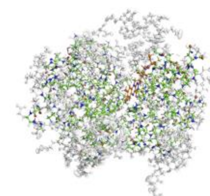
ZOO system for automated measurement



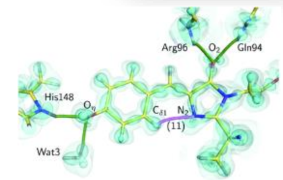
High resolution measurement by short wavelength diffraction







Takaba et al., *Sci Rep* **7**, 43162 (2017)



Takaba et al., *IUCrJ* **6**, 386-400 (2019)

- Towards SPring-8-II

Serial measurements with high-brilliance microbeam will expand the number of samples and promote higher precision in automated appraoches, including faster screening required for drug discovery and synthetic biology. Improvement of our techniques will contribute to high-precision analysis using a diffractometer for biopolymers in combination with other quantum beam probes.

Correlative structural analysis

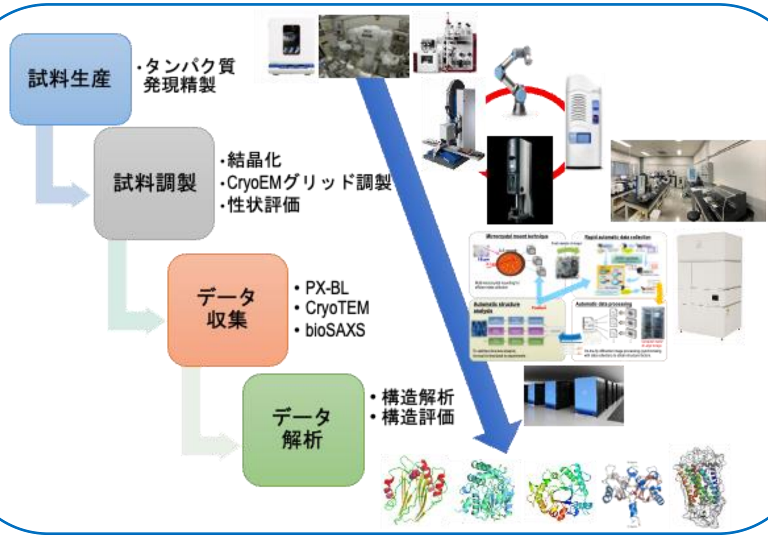
Keywords: Single particle analysis, character evaluation, Cryo-TEM

- Features

Structural analysis of biopolymers has become increasingly diversified. Correlative structural analysis combined with multiple measurement methods can reveal more detailed molecular behaviors. CryoTEM and BioSAXS techniques enable character evaluation of samples that are difficult to crystallize. Our experimental environment includes everything from sample production to data analysis, and will support efficient sample preparation and diffraction measurement with crystallization plates.

- Examples

Structural Analysis Pipeline



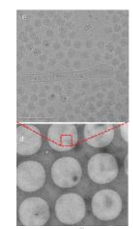
Crystal preparation environment and evaluation measurements

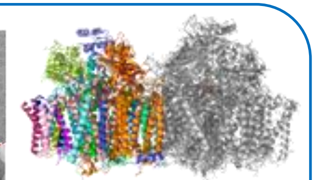
Crystal sample preparation facility

Plate diffractometer

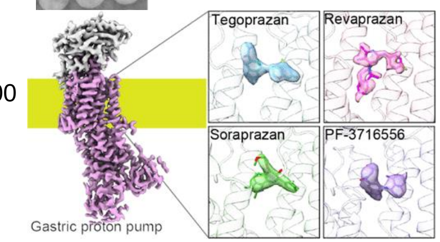
CryoTEM

EM01CT
JEOL
CRYO ARM 300





Membrane protein complex analyzed by CryoTEM



Structure of gastric proton channel and inhibitor complex analyzed by CryoTEM

- Towards SPring-8-II

To promote multifaceted structural studies of biopolymers, user support will be provided by using CryoTEM and bioSAXS in combination with dynamical crystal structure analysis. Development of an integrated environment from the preparation of target samples optimized for each method will accommodate the various measurement methods that become available via the increased beamline performance.

H3

BioSAXS

BL40B2, BL38B1

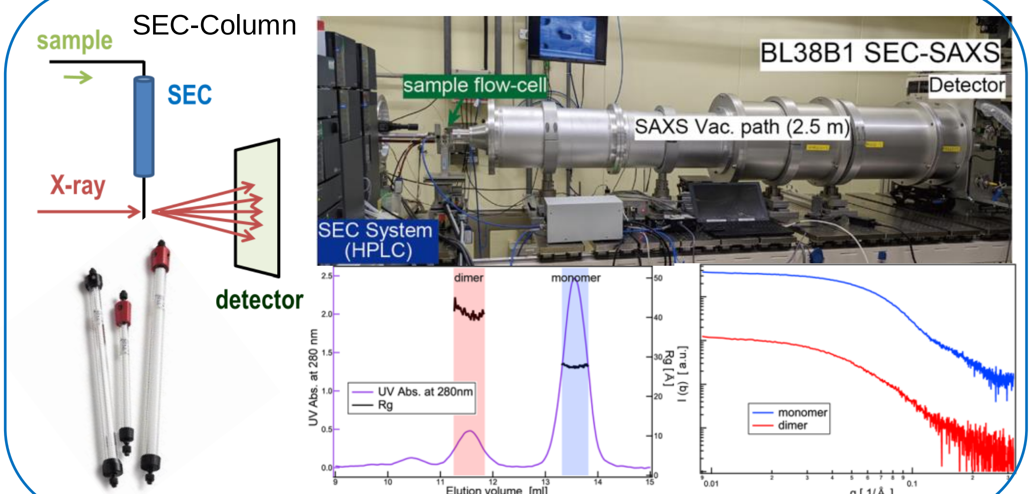
Keywords: SEC-SAXS, Dynamical analysis

- Features

BioSAXS is a method to analyze the solution structure of biopolymers such as proteins. High intensity X-rays enable short-time measurements of structures under dilute conditions. Our high-precision detectors for narrow X-ray beams have achieved high resolution in small angles scattering and are used in many research fields from academia to industry. The SEC-SAXS method is also available, in which measurement cells are connected to gel filtration chromatography for protein separation. This technique gives monodisperse solutions and their components may be analyzed even from solutions mixtures of various molecular sizes.

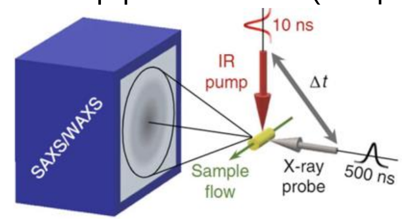
- Examples

Size-exclusion chromatography coupled SAXS system

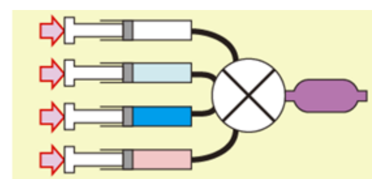


Dynamical structure analysis

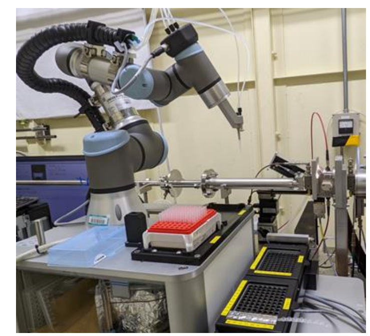
Pump-probe method (ns - μs)



Stopped-flow method (ms)



Automated measurement and analysis



Automatic measurements by solution exchange robot

- Towards SPring-8-II

Higher-quality, higher-brilliance X-ray beams will enable comprehensive measurements under various conditions (concentration, temperature, pH, ligand concentration). They will also realize titration-type SAXS, high time-resolution, and scattering measurements over a wide angular range. Combinations of these methods with other experimental methods will further improve functional analysis such as dynamical properties of samples.

H4

Dynamical Crystal Structure Analysis at Room Temperature

BL41XU, BL26B1

Keywords: Time-resolved measurement, structural polymorphism Serial crystal structure analysis

• Features

Protein crystallography has revealed an importance of dynamical properties at room temperature, especially by using XFEL. Our experimental systems offer serial measurements for dynamical crystal structure analysis that enables detection of diffraction data in various delay time from possible states of reacting processes induced by light irradiation or substrate addition. Diffraction measurements with crystallization plates are also available.

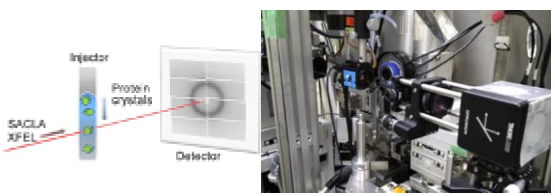
• Examples

Dynamical structure analysis using serial crystallography

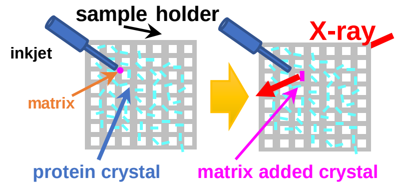
Structural analysis targeting time resolution of msec-sec

- Time-resolved measurement system using laser excitation incorporating the injector system developed at SACLA
- Development of serial synchrotron crystallography (SSX) techniques

Dynamical structure analyzing system



Fixed serial target method



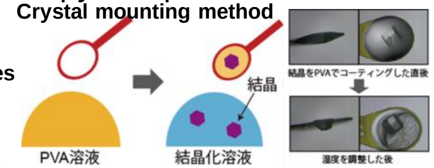
Measurement at room temperature – HAG method

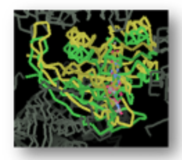
Water-soluble polymer coating and temperature/humidity control

- temperature(4-70℃), humidity(50-100%RH), pH change available
- Application to microspectroscopy and photoexcitation

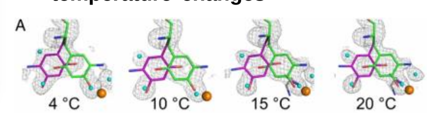
Crystal mounting method

Structural changes due to humidity changes





Structural changes due to temperature changes



• Towards SPring-8-II

Our pink beam technology provides more than two orders of magnitude increase in intensity. A microbeam will enable even smaller crystals and time-resolved measurements on the order of microseconds. It will also enable dynamic structural genomics and lead to applications such as structure-based drug discovery, including transition structure information.