D1

Multi-purpose X-ray diffraction

BL13XU, BL19B2

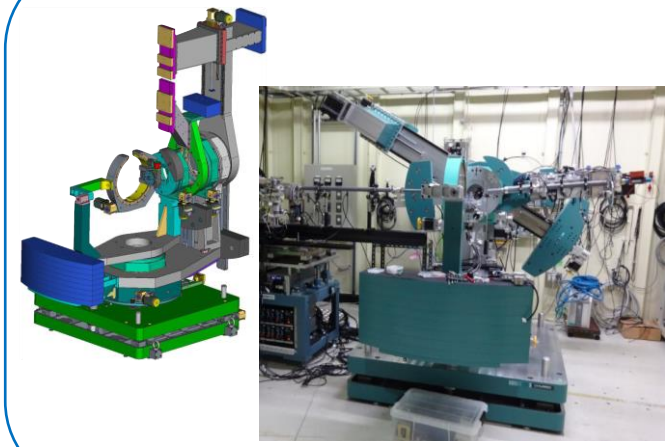
Keywords: Multi-axis diffractometer, in-situ, operand measurement, mapping, customized

• Features

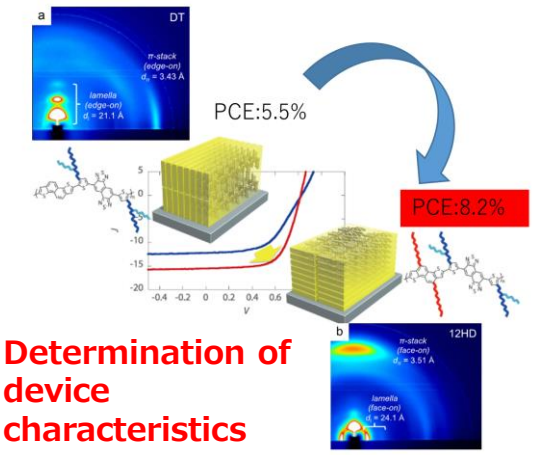
Our multi-purpose X-ray diffractometer has a large sample space, high load capacity, and a high degree of freedom in angle and position control. Various X-ray scattering and diffraction measurements are available with selectable detectors. Setups include parallel optics, 0D + slit/spectroscopic crystal, 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



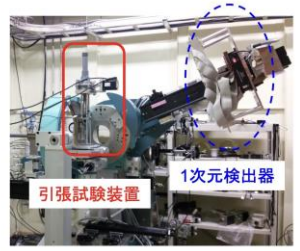
Evaluation of crystallinity and orientation of organic thin films



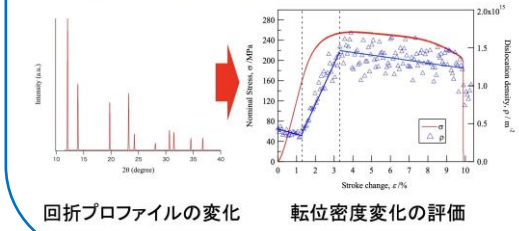
Determination of device characteristics

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



• 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

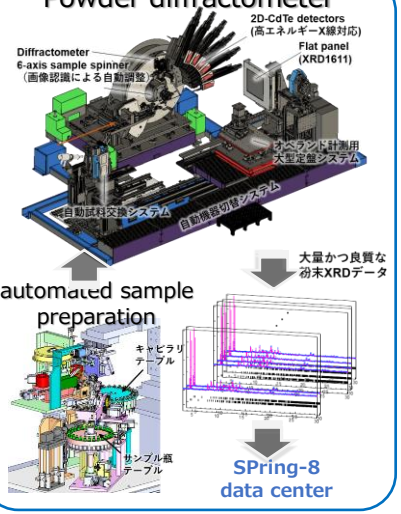
Keyword: Automated measurement, Operando, Crystal structure analysis

• Features

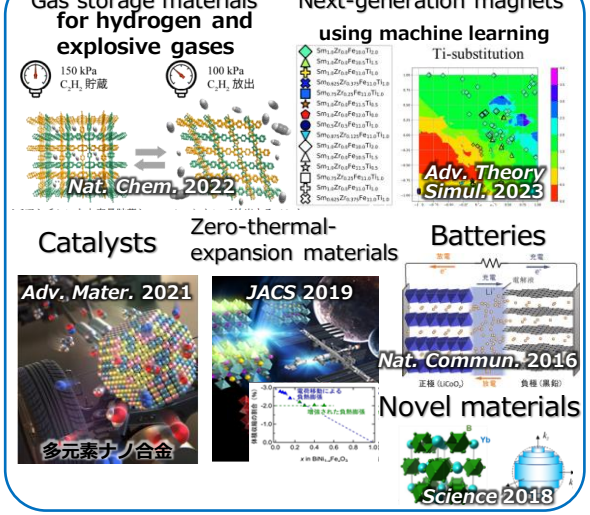
X-ray powder diffraction measures structures of crystals and their local properties. Our automated system, sample preparation to measurements, provides high-quality data over a large area of reciprocal space with high angular resolution. Short time measurements can be done even with small amounts, ~mg, 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

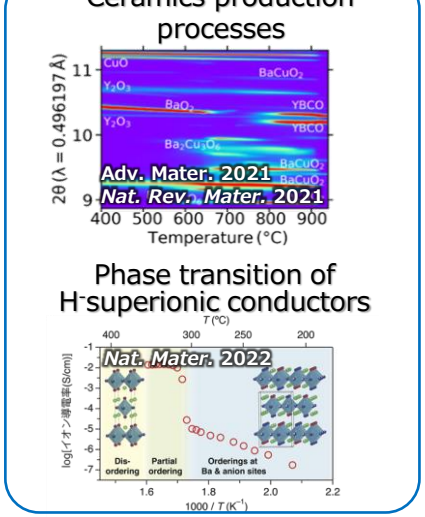
Automated powder diffraction system



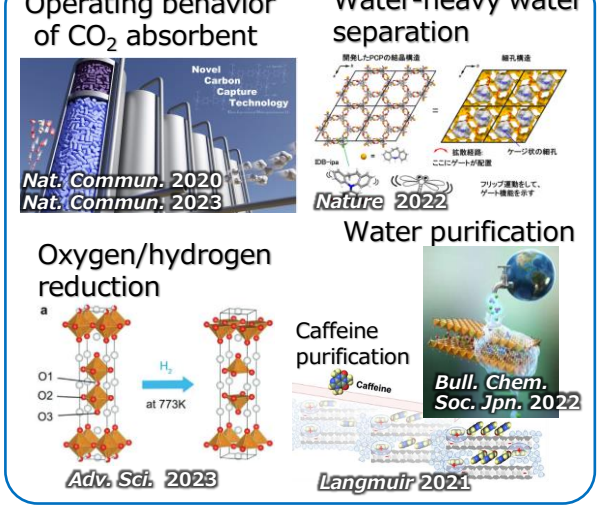
Creation and utilization of large volume and high quality data



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.

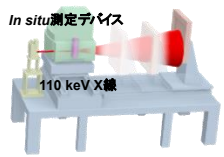
• Examples

PDF measurement systems

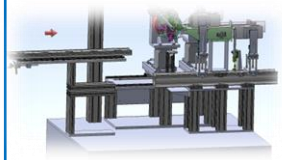
7 sequential detectors



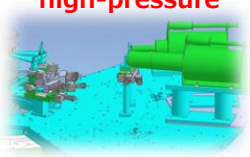
Time-resolved



High-throughput



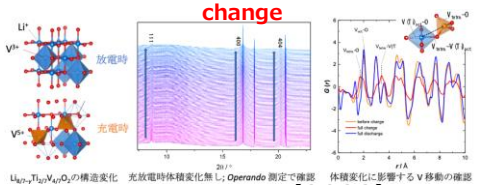
High-energy X-ray high-pressure



applicable to various fields of research

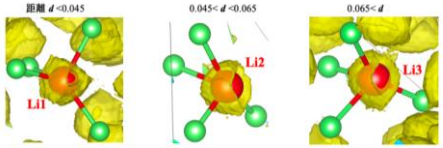
Next-generation rechargeable batteries

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



Nature Mater.(2023)

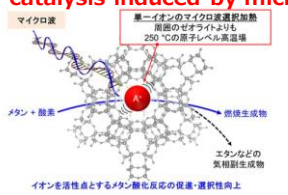
Structure of solid electrolyte in collaboration with Fugaku



Energy Environ. Mater.(2023)

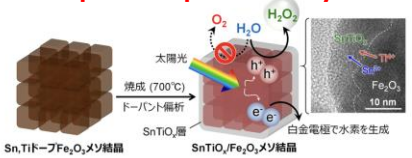
Environment catalysts

Visualizing the mechanism of selective catalysis induced by microwave



Sci. Adv. (2023)

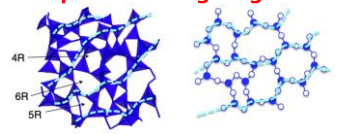
Structure of materials for inexpensive photocatalysts



Nature Commun. (2022)

High-temperature fusion

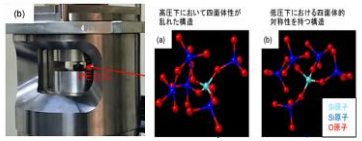
In-situ measurement of molten liquids forming to glass



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

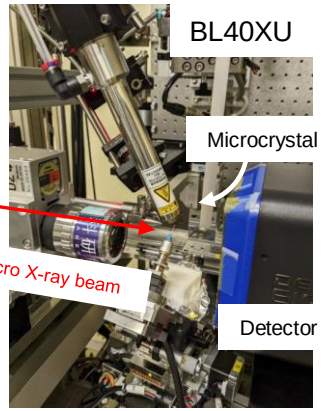
Keywords: Precise structure analysis, High throughput

• 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

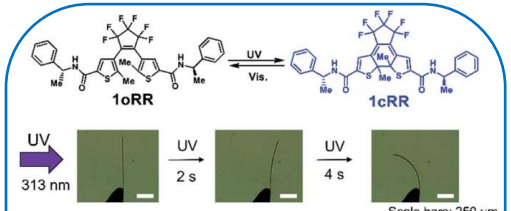
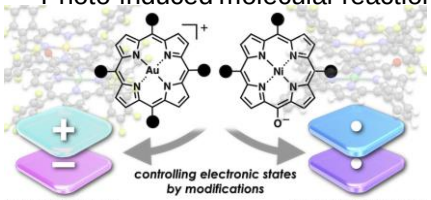
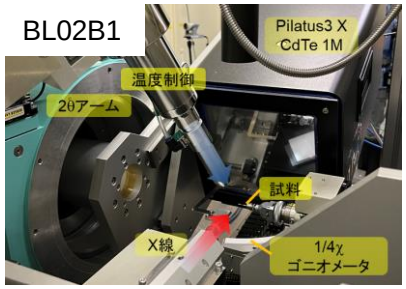


Photo-induced molecular reactions



Heterogeneous radical pair stacking structures

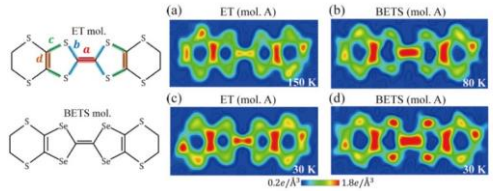
Precise structure analysis system



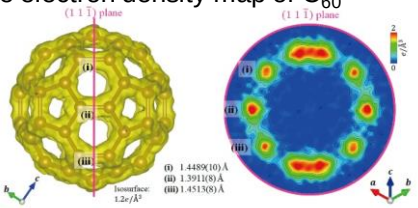
X-ray energy is 50 keV(λ=0.248 Å)

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

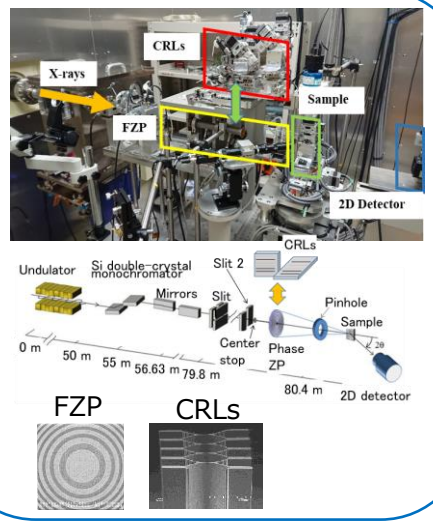
Keywords : Lattice deformation, mapping

• 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.

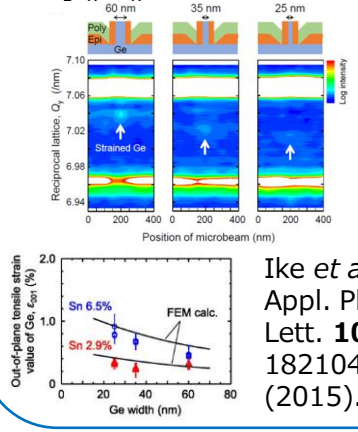
• Examples

Experimental setup



Evaluation of local strain inside semiconductor microstructures

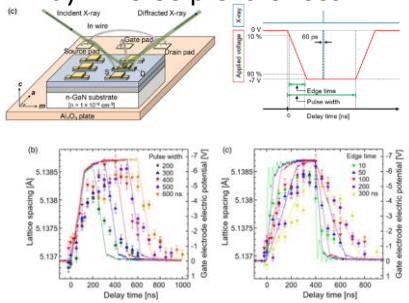
Evaluation of local strain inside embedded $Ge_{1-x}Sn_x$/Ge microstructures



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

Operando measurement by time-resolved nano XRD

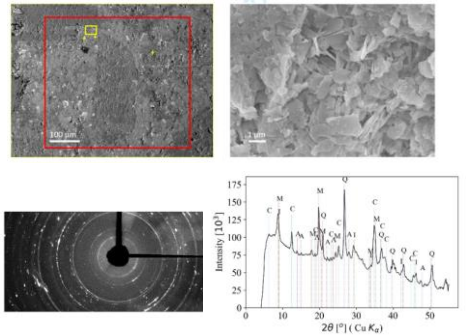
Analysis of lattice deformation in AlGaN/GaN by inverse piezo effect



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.

D6

XRD imaging/3DXRD

BL47XU, BL05XU

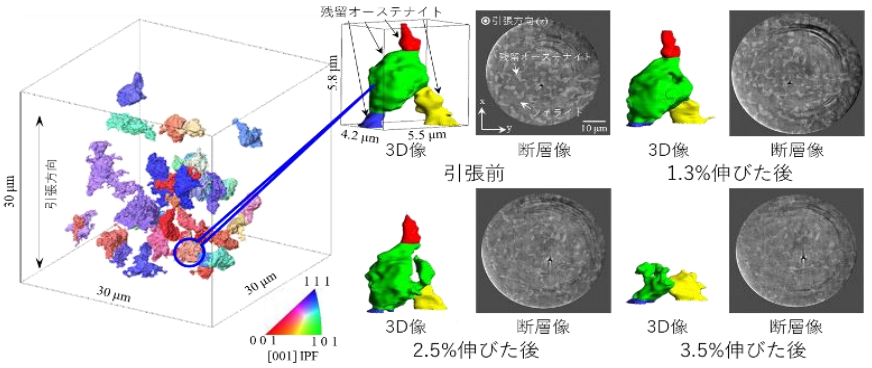
Key words: Grain, Orientation mapping, Non-destructive, Operando

- 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

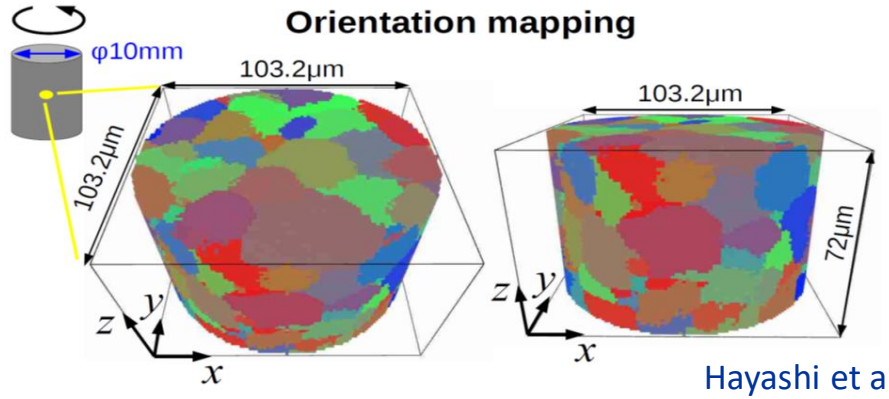


残留オーステナイトの3D像　　個々の残留オーステナイトの相変態挙動

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.

D7

Large-capacity high-pressure press

BL04B1, BL05XU

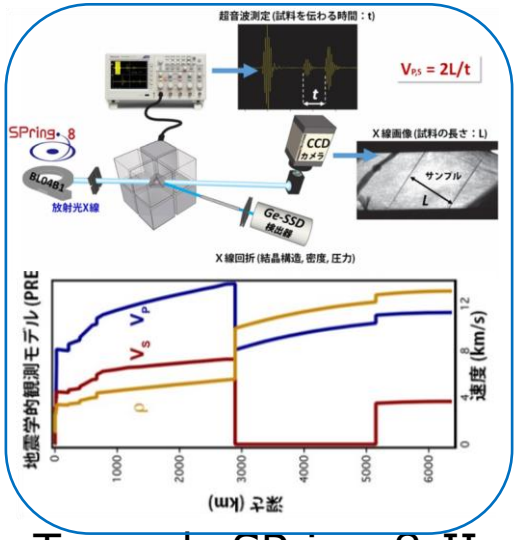
Keywords: Earth planet dynamics, Extreme condition

• Features

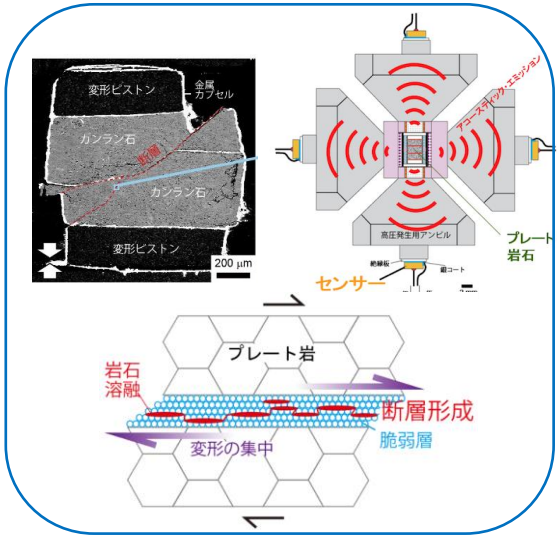
Measurements of crystal structures and physical properties under high pressure has become an important method in industrial research, earth and planetary science, and for developing novel materials such as high-Tc superconductors. Our large-capacity press system can perform precise X-ray diffraction 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

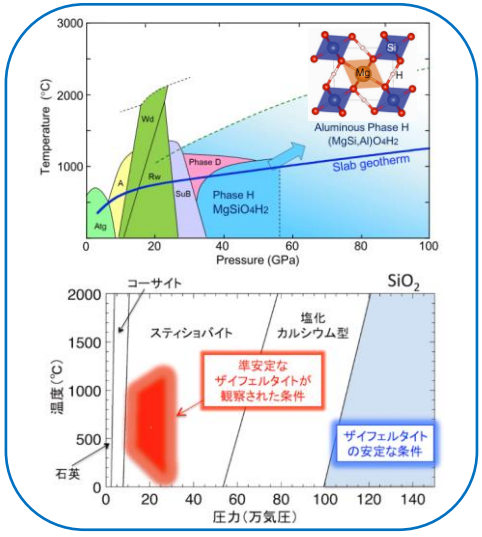
Earth and planetary deep structures



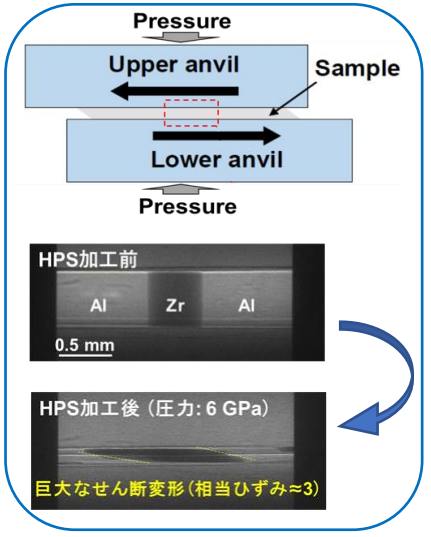
Mechanism of earthquakes



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.