BL10XU: High Pressure Research

The beamline BL10XU is fully dedicated for X-ray diffraction measurements at high pressure and low/high temperature using a diamond anvil cell (DAC) (Figure 1) [1]. The high-resolution monochromatic angle-dispersive X-ray diffraction patterns allow us to the accurate determination of equations of state, precise determination of phase relation, structure refinement by Rietveld analysis, and charge density distribution analysis in crystals submitted to extreme pressures. Pressure can induce dramatic changes in physical and chemical properties of materials through volume compaction or decrease in inter-atomic distance, providing significantly difference from common properties known at ambient pressure. To have a better understanding of high-pressure study using a combination of synchrotron radiation and a DAC technique through this BL practice course, in situ high-pressure and high-temperature X-ray diffraction experiments will be carried out, including a calibration of energy, sample loading into a DAC, X-ray diffraction measurements, and data analysis.

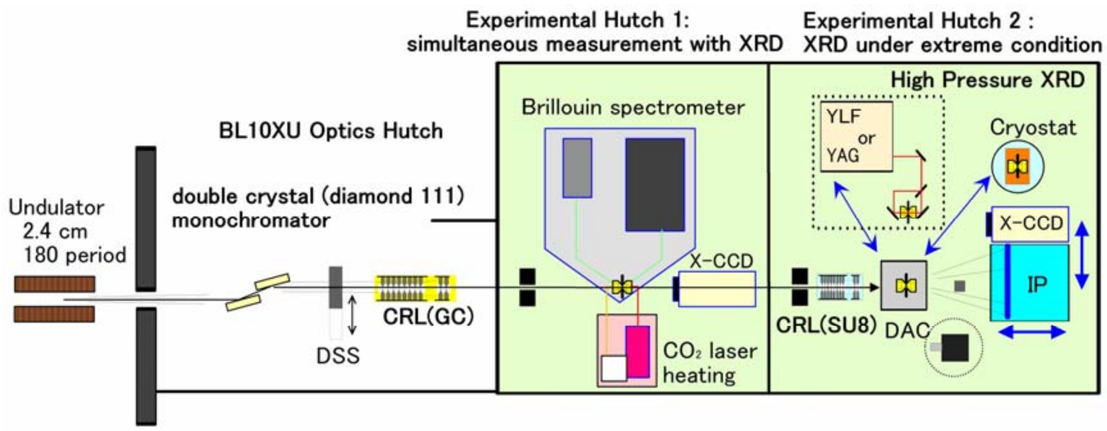

Figure1. Schematic layout of beamline BL10XU [1].

References:
[1] Ohishi *et al*. (2008) *High Press. Res.* **28**, *163–173*.

– GLOSSARY –

Diamond anvil cell

Diamond anvil cell is one of high-pressure generation techniques by static compression. The sample is placed in a pressure chamber created between the flat parallel faces (culets) of two opposed diamond anvils and the hole penetrating a hardened metal foil (= gasket) (Figure 2). Pressure is applied by forcing the diamonds together. Diamond is the premier anvil material because of hardest substance and transparency to electromagnetic radiation over a wide spectral range from the infrared to hard X-rays.

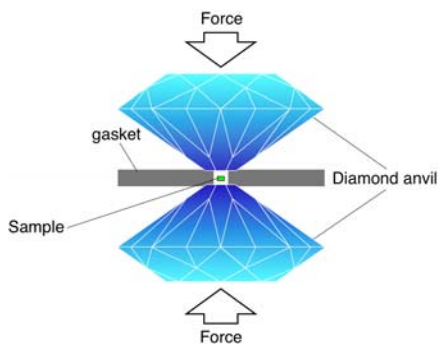

Figure 2. Diamond-anvil cell technique.

– Practice –

The mineral olivine is one of the common and most abundant minerals on Earth. Transparent olivine is sometimes used as a gemstone called peridot. This is a magnesium silicate with a composition of $(Mg,Fe)_2SiO_4$, a continuous solid solution between forsterite Mg_2SiO_4 and fayalite Fe_2SiO_4. Olivines have high-pressure polymorphs, which is important to understanding the nature of the Earth's upper mantle.

In the practice, synthesize and identify high-pressure phases on olivine $(Mg,Fe)_2SiO_4$, one of the primary minerals in upper mantle, using a laser-heated diamond anvil cell and in-situ synchrotron radiation X-ray diffraction.

1) Energy calibration

Angle dispersive X-ray diffraction (XRD) requires a monochromatic X-ray beam. The accurate determination of the wavelength for incident X-ray beam and of the distance between sample and detector of imaging plate (IP) allow to determine precise crystal structures and the unit-cell parameters of materials. In this practice, we use photon energy tuned to 30 keV (0.4133 Å) for the XRD measurement. The calibration of the X-ray wavelength and the distance is based on the standard material of cerium oxide (celia, CeO_2).

1. Obtain XRD images of CeO_2 at two different IP distances.
2. Convert X-ray images to conventional one-dimensional X-ray pattern.
3. Calculate the wavelength and the distance between sample and detector accurately.

2) Sample loading into a DAC

We use a symmetric cell suitable for double-sided laser heating. In DACs, a sample is placed in a chamber hole drilled in a rhenium foil between the culets of two opposed diamond anvils.

3) High-pressure generation and pressure measurement

After sample loading into a gasket, pressure is generated with forcing the two opposing anvils. For a symmetric cell, controlled tightening of force generation through screws forces together the two plates that support the two anvils.

Pressure in the DAC is determined by ruby pressure scale, established by measuring simultaneously the shift of the ruby R1 luminescence line and the specific volume of metals. On monitoring the shift of R1 line, increase to desired pressure above 25 GPa.

4) Laser-heating for high-temperature generation and in-situ XRD data collection

We carry out double-sided laser heating with Nd:YAG laser (1064 nm) to synthesize high-pressure phases on the $(Mg,Fe)_2SiO_4$ system. To study stable phases at high pressure and high temperature, collect in-situ XRD images.

5) Data analysis and phase identification

The basic principle of the X-ray diffraction is the Bragg's law: $\lambda = 2d\sin\theta$, where λ is the wavelength of the X-ray, d the lattice spacing, θ the angle of the incident beam and the diffracting lattice plane. The diffraction peaks are observed when the *d*-spacings satisfied the Bragg's law.

1. Convert the X-ray image to conventional one-dimensional X-ray pattern.
2. Calculate *d*-spacing for each peak from 2θ value using the Bragg's law.
3. Index peaks and identify high-pressure phases.