Development of high pressure and high temperature *in situ* X-ray diffraction system using Drickamer-type apparatus

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A new high pressure and temperature *in situ* X-ray diffraction system has been developed using Drickamer-type apparatus combined with synchrotron radiation. There are two different types of high pressure apparatuses in SPring-8. One is a large volume apparatus such as SPEED1500 and SMAP, and the other is a diamond-anvil type apparatus. Large volume apparatus has the advantage to achieve stable and uniform heating of the sample under high pressure, although the pressure range is limited to about 30 GPa. On the other hand, diamond anvil can compress samples to above 100 GPa, although it is, so far, difficult to perform high temperature experiment. The new system developed in the present study has the advantage to cover higher pressure range compared to the large volume apparatus, and to cover higher temperature range compared to diamond anvil apparatus. Moreover, it has the capability to study the mechanical property of materials under uniaxial compression.

Drickamer-type apparatus is an opposed-anvil type apparatus and the applied pressure has large uniaxial stress component. Because of the geometry, the stress filed has a cylindrical symmetry and the method of analysis is etablished(1) if the strains in two different directions are measured. In the present system, d-spacings of the sample in two different orientations, one is in the direction parallel to the uniaxial stress component and the other is in the direction perpendicular to it, can be measured using two detectors(Fig. 1). Energy dispersive powder X-ray diffraction method was employed. The sample is compressed using 75 ton hydraulic press. White X-ray from the ring is collimated to a thin beam, about 50 μm x 50 μm, and is irradiated to the sample from the direction perpendicular to the compression axis of the uniaxial press. Diffracted X-rays from the sample are measured, after passing through two slits which work to eliminate diffractions other than the sample, using two solid state detectors. Two diffractometers can be adjusted independently so that the signals from the sample can be observed with high S/N ratio.

In the present machine time, a new system designed for SPring-8 was installed for the first time. It was necessary to make many small changes, which were difficult to check in advance, and the method of adjustment was established. Unfortunately, the high background noise in the experimental hatch caused serious problem and we could not get a diffraction from the sample until the end of the current machine time. However, we could understand the problems and we have made plans how to improve the system. Preliminary results indicates that the present system is quite promising and we have started preparing for the next machine time.

Ref. (1) T. Uchida, N. Funamori, and T. Yagi, J. Appl. Phys. 80, 739 (1996).

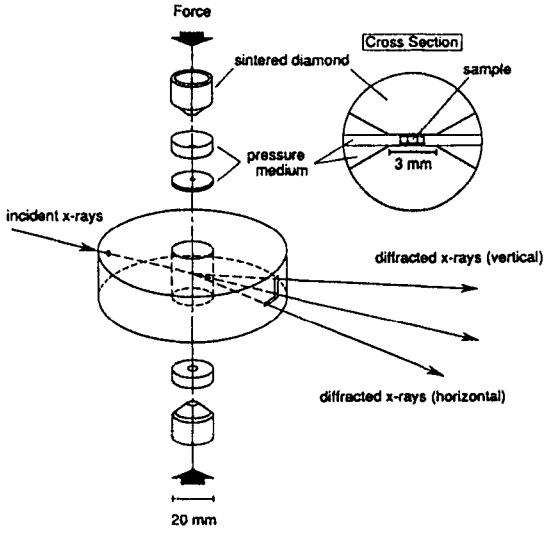

Fig. 1 A geometry of the Drickamer-type apparatus and X-ray system.