

A trial of high pressure X-ray topography

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Due to technical difficulties, there has been a little progress on rheological study of high pressure mantle minerals in last decade. Development of in-situ observation techniques, which are applicable to high P-T condition, has been slow. We have recently introduced an in-situ macroscopic strain measurement method based on high pressure X-ray radiography (Kanzaki, 2001). What we need next is, a microscopic in-situ observation technique, which enables direct observation of defects in sample under pressure. As an effort to develop such technique, we have applied X-ray topography to high pressure sample. The X-ray topography has been used to observe defects in single crystals at ambient condition. Also in-situ studies at high temperature and ambient pressure, and at low temperature and modest pressure (few hundred bars) have been reported. However, there is almost no such study applied to the mantle P-T conditions. In this study, we applied this technique to a quartz crystal placed in a multi-anvil high pressure apparatus using synchrotron radiation.

We conducted high pressure topography experiments at BL04B1, SPring-8. For high pressure generation, SPEED1500 press with DIA-type guide block is used. We implemented Kawai-type double-stage anvil configuration as usual. We used an octahedral MgO pressure medium with 18 mm edge length. Eight WC cubes (11mm truncated edge length) are used as second-stage anvils. Pyrophyllite gaskets were used, except the area where X-ray passes. In these area, graphite is used instead to reduce X-ray absorption. Natural quartz crystal was used as sample, and was shaped as thin plates and rods. The c-axis of quartz was placed perpendicular to the X-ray beam, and is parallel to vertical direction. The topographic images are observed by a homemade CCD camera with a YAG(Ce) fluorescence plate. The camera was placed on horizontal goniometer stage used for powder diffraction experiments. This allows us to move the camera remotely using the goniometer control system, however, a distance between the camera and the sample becomes large (about 650 mm).

We first conducted X-ray topographic observation of quartz placed in the press at 1 bar. Most obvious problem of the technique when applied to multi-anvil apparatus, is shading of diffracted spots from the sample by first- and second-stage anvils. Thus, we could observe very limited spots. Adding to this, the sample can not be freely rotated. As a result, we have to move the camera to find the spots. We employed section topography technique, in which bulk sample instead of thin plate sample can be used. This allows us to use rod shaped sample for observation, and handling of such sample is much easy in the high pressure cell, compared to thin plate sample. However, due to small diffraction angle due to shading of first-stage anvils, the section topographic images are compressed horizontally, reducing resolution in this direction.

We conducted a high pressure experiment using a rod shaped quartz crystal (4 mm in diameter and 4 mm in height). The quartz is placed in a MgO pressure medium, and is surrounded by NaCl powder, except top and bottom ends. This configuration enables uniaxial stress loading to the sample along the rod's axis (c-axis). The press load was increased slowly. Above 15 tons, the spot gradually spread horizontally due to brittle deformation of the sample by the uniaxial stress. At 50 tons, the spot was difficult to detect by the CCD camera, however, we could see horizontally elongated spots using a Polaroid film. Recovered sample shows numerous cracks which mostly run to vertical direction.

Although the pressure was very low (few kbar), present study demonstrated that high pressure X-ray topography using the Kawai-type double-stage multi-anvil apparatus is feasible.

Refereces

M. Kanzaki (2001), Abstract of Japan Earth Planetary Science Joint Meeting, Tokyo.