Development of high pressure apparatus for X-ray microtomography at SPring-8

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Abstract. A new high-pressure apparatus has been developed for studies of synchrotron radiation-based X-ray microtomography at SPring-8 of Japan. The high pressure tomography apparatus at SPring-8 is a compact hydraulic press with a 0.8 MN capacity and is equipped with an opposed anvil device. It has two wide windows for X-ray access with a 160-degree opening in the equatorial plane to the compression axis. Radiographs are acquired over 180 degree rotation for reconstruction of 3D image, in which some shadows occur, because the press frame blocks a 20-degree angular region. 3D tomography image computed from radiographs obtained using the high pressure tomography apparatus has a reasonably good quality enough to measure physical properties of materials.

1. Introduction

X-ray imaging has found a wide range of applications in large volume press for the study of physical properties of materials under high pressure and temperature. High-speed and high-resolution radiography (2D imaging), for example, allows us to measure the viscosity as low as $10^{-1}$ Pas of molten iron alloys at the pressure range to 16 GPa [e.g., 1-4]. Synchrotron radiation-based X-ray microtomography can extend X-ray imaging to 3D and will visualize the complete texture inside the pressure vessel. Wang et al. [5] developed the high-pressure tomography apparatus at GSECARS of APS to introduce this technique to high-pressure imaging. By using this apparatus, we observed interfacial tension of molten iron alloys under pressure by a sessile drop method [6, 7], that was the first measurement of surface properties of melts at high-pressures. Thus, 3D imaging is expected to give opportunities to measure physical properties of materials which have never been examined under pressure.

SPring-8 in Japan has four sophisticated beamlines which are oriented to X-ray imaging. Collaborating with the imaging group of SPring-8, we started high-pressure X-ray tomography. We have developed a new high-pressure tomography apparatus which is designed for use at the imaging beamlines of SPring-8. The commissioning test of this apparatus using synchrotron X-ray has been
carried out to evaluate its performance. In this paper, we describe details of our high-pressure tomography apparatus and report the preliminary results of X-ray microtomography under pressures at SPring-8. We believe that the development of high-pressure tomography in SPring-8 can open new frontiers for high-pressure sciences.

2. High-pressure tomography apparatus

3D image of X-ray microtomography can be computed from 2D X-ray radiographs. It is desirable for reconstruction of tomography image to acquire radiographs at small angular steps over 180 degree rotation with respect to the axis perpendicular to incident X-ray beam. At APS, this is achieved by use of the high-pressure tomography apparatus with rotational mechanism of which the anvils can be rotated under a uniaxial hydraulic load by thrust bearing system [5]. The high-pressure tomography apparatus of APS is driven by a large 2.5MN uniaxial press. It is, however, difficult for this size of press to install in a limited space of the imaging beamlines of SPring-8, so that we made a different choice than that of a rotational mechanism. We decided to use a compact press like a panoramic view type Paris-Edinburgh press [8] which has two wide windows for observation. In this case, it is impossible to obtain complete radiograph data set due to the shadow cast by the press frame which blocks a small angular range in the view. We evaluated the effect of shadow region on reconstructed tomography image by simulating various cases of the field of view. Spatial resolution of tomography image is reduced and image artifacts produced by shadow region are increased if the field of view is narrowed, whereas the maximum press capacity is limited if the window for X-ray is broadened. We took both parameters into consideration and optimized the window size.

The high-pressure apparatus for X-ray microtomography at SPring-8 is a compact hydraulic press with a 0.8MN capacity and a weight of about 30 kg, which can be set on a standard rotational stage for tomography measurement setup (Figure 1). The system compresses an opposed anvil device for which a cupped or a toroidal anvil cell [9] are equipped. The press body has a wide range of X-ray viewing angle of 160 degrees in equatorial plane perpendicular to compression axis (Figure 1A). In tomography measurement, during rotating the press around the compression axis, radiography images are acquired over 180 degree rotation with the shadow areas included. This apparatus is named TPH, Tomography Press 80 (Hachi-jyu in Japanese), after one of the authors.

![Figure 1](image1.png)

Figure 1. (A) High pressure apparatus for X-ray microtomography (TPH). (B) TPH is set on the experimental stage at the imaging beamline (BL20B2) of SPring-8.
3. X-ray tomography measurements at high pressures

Tomography experiments were conducted at experimental hutch 1 of BL20B2 of SPring-8 [10] which is dedicated to X-ray imaging and has equipment for microtomography. Incident beam was a monochromatic X-ray with a size of 75 mm (horizontal) and 4 mm (vertical), with a transmitted X-ray detected using a CCD camera with P43 scintillator. Energy of X-ray was tuned for desired value between 30 and 51 keV.

The TPH was installed on a conventional stage with the X-Y-Z and the rotation axes to acquire radiography images within the sample space. Exposure time was 300ms. It took about 15 minutes to collect a data set for computing tomography image with 0.2 degree step over 180 degree rotation, which is one order of magnitude shorter than that at APS. It is advantageous to complete measurements in such a short time for high temperature, because, during heating, anvil gap is normally reduced to change the field of view in terms of time. In our case, the spatial resolution was about 12 μm (5.6mm resolution with 2X2 binning). Higher resolution is achievable with slightly longer exposure time.

![Figure 2. Tomographic slice image of olivine and nickel powder mixture. (a) Reconstructed image with a complete data set. (b) Image reconstructed by limited data which are acquired by using TPH.](image-url)

In order to evaluate the effect of the shadow region cast by the press frame, we compared two tomography images of nickel grains with diameters of 50-150 μm embedded in powdered (Mg0.9Fe0.1)2SiO4 olivine matrix. Volume fraction of nickel is 0.1. The sample was enclosed in a boron nitride capsule, which was inserted in a boron-epoxy resin pressure medium. One tomography image was constructed using a dataset collected outside the press, with radiographic images measured over a complete 180 degree rotation, without any shadowing (Fig. 2a), and the other is computed from radiographs measured by using high-pressure apparatus TPH at atmospheric pressure (Fig. 2b). While the conventional tomography yields clear slice image, the high pressure tomography image shows the effect of shadow area. There are many crossing bright streaks elongated from nickel grains, which are computed pseudo image originated from shadow region in radiographs. Although tomography image obtained by TPH includes false streaks, nickel grains can be clearly distinguished from olivine matrix and resemble those of the conventional image in their shape, size and distribution in olivine matrix. The two images in Figure 2 do not show exactly the same cross section of the sample, and their vertical positions are slightly different. Taking this into consideration, we conclude that the high pressure tomography using TPH can give computed 3D image with reasonably good quality.

Tomography measurements using TPH were also carried out at high pressures up to 4GPa with a toroidal anvil cell. Computed tomography images under pressure had essentially the same quality as
that at atmospheric pressure, but the field of view apparently decreased along the compression axis with increasing pressure. When we used the toroidal anvil with a centre hole of 8 mm diameter, the gap between top and bottom anvils decreased from 2.1 mm at 0.1 MPa to 1.0 mm at 4 GPa. To keep the anvil gap wider, the use of a toroidal anvil cell is preferable to a simple cupped or a Drickamer anvil cells, although maximum attainable pressures are limited at the same press load. Regardless of the cell types to be used, anvils made of X ray transparent materials (such as sintered cubic boron nitride or diamond with SiC binder) will help increase field of view in the vertical direction under pressure. TPH combined with those anvils are expected to expand the pressure range of high pressure X-ray microtomography.

Although we experienced the high-temperature tomography under pressure at APS [6, 7], tomography measurement at high-temperature using TPH is getting ready at SPring-8. During heating, anvil temperature increases to several hundred K, reducing the strength of the anvils. To achieve temperatures as high as 1500 K without anvil failures, a water cooling system for anvils is attached to TPH. Short duration of tomography measurement at SPring-8 will make it easy to conduct high pressure and temperature tomography using TPH.

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