Development of a High-Pressure Tomography Apparatus for Computed Microtomography under High Pressure and Temperature

Y. Wang, [1] T. Uchida, [1] M.L. Rivers, [1] F. Westferro, [2] J. Gebhardt, [2] S.R. Sutton, [1]

[1]GSECARS, The University of Chicago, Chicago, IL, U.S.A.;
[2]ChemCARS, The University of Chicago, Chicago, IL, U.S.A.

Introduction

Complex, heterogeneous structures occur naturally at various length scales under a vast range of conditions. The response of these heterogeneous structures to pressure, temperature, and stress is of interest to physics, earth sciences, materials science, as well as engineering. Microtomography is a powerful tool for nondestructive investigations of heterogeneous structures. However, to our best knowledge, we are not aware of any experimental instrumentation in existence that allows microtomography to be conducted under high pressure. Here, we report our recent development in the design, contruction and commision of an apparatus for conducting computed microtomography under high pressure and temperature.

System Description

Design Requirements

The following requirements are considered for the design of high-pressure tomography apparatus: (1) it must be able to generate enough pressure - our goal is 10 GPa, (2) the sample under high pressure must be able to be accurately rotated so that tomography images can be collected, (3) the sample chamber must be transparent to the X-rays to allow absorption contrast to be recorded, and (4) the sample should be large enough in order to obtain information on microstructure and its evolution in a meaningful way. In addition, we require that the apparatus must be small enough to fit into the existing 250 ton hydraulic press at beamline 13-BM-D of the Advanced Photon Source [1]. These requirements are addressed by the following compnents.
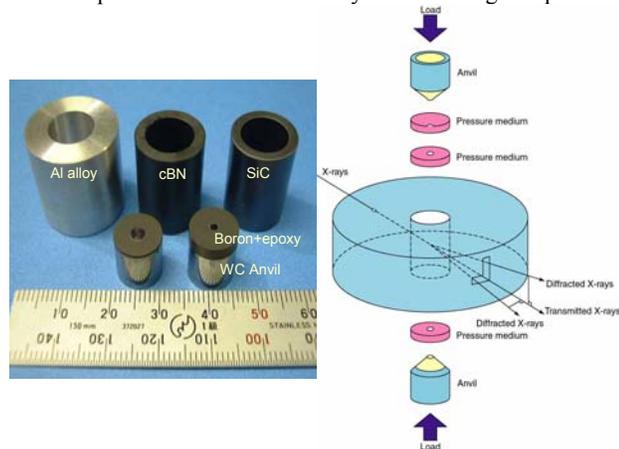
Fig. 1. Convetional Drickamer anvil cell (right) with modified containment rings (left).

The X-Ray Transparent Drickamer Anvil Cell

The Drickamer cell is a well-known and widely used device for high-pressure and temperature experimentation (Fig. 1). It is an opposed anvil device, similar to the Bridgman anvil apparatus and the diamond anvil cell (DAC). However, the anvils are compressed inside a containment ring, which restricts the extrusion of the pressure medium, and thus is capable of maintaining a finite sample length, attaining a bulk sample under pressure.

The traditional Drickamer cell uses a thick containment ring, made of either hardened steel or tungsten carbide (Fig. 1). This is not feasible for microtomography experiments. Our solution is to replace the containment ring with a thin and light Al alloy ring, which allows modest pressure generation, while allowing X-ray imaging at the same time. Al alloy is readily available, inexpensive, and homogeneous in physical properties. Depending on the truncation size and tapering angles of the anvils, about 8 GPa has been generated with a load of 25 tons.

The Load-Bearing Rotation Mechanism

The overall configuration of the Drickamer-anvil high-pressure apparatus is illustrated in Fig. 2. The sample (center of the apparatus, not shown) is compressed by a pair of Drickamer anvils (A), supported by a steel column (B), through a WC spacer (C). The column is supported by a thick steel plate (D), to spread the load to two concentric low-profile thrust bearings (E1 and E2), on each half of the apparatus. The load support column (B) and the Drickamer anvils (A) are attached to the passive rotating member (Flexspline; F) of the HarmonicDrive[TM] unit, driven by the active member (Wave Generator; G) of the unit, through the coupler plate (H). The Wave generator is attached to a large precision gear (I), driven by a smaller gear (J), through a right-angle gear box (K) and a stepper motor (L).
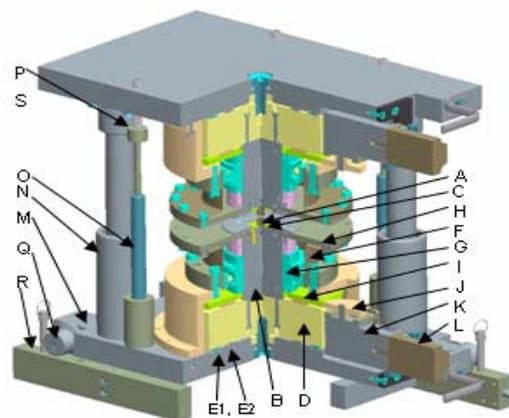
Fig. 2. Rotation mechanism design. Maximum load is tested to be 25 tons.

The load bearing frame was designed based on the development of the Drickamer anvil device. In order to generate up to 10 GPa pressure, we require the thrust bearings to be able to support ~50 ton load. This is achieved by introducing two concentric thrust bearings at the end of each Drickamer anvil. The thrust bearing we adopted are INA[TM] low-profile needle rollers, described by [2]. The difference between the current design of load support and that of [2] is that we utilize

two thrust bearings concentrically to further reduce rotational friction. In addition, thrust bearings are installed at the ends of both Drickamer anvils, so that the entire cell can be rotated. In the case of rotational deformation apparatus, only one anvil needs to be rotated relative to the other. The axial dynamic and static load specifications are 148 and 1020 kN, respectively, for the large thrust bearing AXK160200, and 91 and 560 kN, respectively, for the smaller AXK100135. With the specified grease for lubricant, the effective frictional coefficient is 0.05, and the calculated torque for rotation is 745 N-M at a motor speed of 2000 rpm.

The HarmonicDrive^{TM} is has a combination of advantages that make it unique for our application. It has the highest torque-to-weight ratio, essentially no backlash, and high position accuracy. We chose model SHF-58-160-2A-GR, with a gear ratio of 160:1.

The 8" diameter gear (I; 303 stainless steel) attached to the Wave Generator (G) has 260 teeth machined on the outer rim. This gear is driven by a pre-loaded gear (J) with 80 teeth. The right-angle gear box (K) has a gear ratio of 10:1. Overall, a total gear reduction of 5200:1 is achieved, allowing low applied torque to rotate the apparatus under hydraulic load. Angular resolution is 0.0167° per stepper motor revolution, with 400 steps per revolution.

The apparatus is built in a die set, with the thrust bearings loaded in the hardened steel die plate (M). The top and bottom halves of the apparatus are aligned by four ball-bearing die posts (N) and kept separated at a desired distance by four gas springs (O). When hydraulic load is applied, the gas springs will comply to allow the two halves to be compressed. The bottom die plate has wheels (Q) attached, so that the entire apparatus can be rolled in and out of the hydraulic press over the installed rails (R). Screws (S) through the tapped holes in the top die plate allows the top half to be raised and lowered during operation of loading and removing the Drickamer anvils with the sample assembly. The complete setup is shown in Fig. 3.

Test Results

Pressure Generation in Modified Drickamer Cell

A series of tests was conducted on pressure generation in the Drickamer cell, using various anvil geometries and containment ring materials. The diameter of the WC anvils varied from 10 to 20 mm, the diameter of the anvil tips from 2 to 4 mm, and the anvil taper angles from 10 to 30°. Three materials were tested for the containment ring: SiC, cubic boron nitride (cBN), and Al alloy. The ring thickness varied from 5 to 10 mm. The optimum performance was found for a combination of 10 mm anvil diameter, 10° taper angle, with 5 mm wall thickness Al alloy rings. Fig. 4 shows test results on pressure generation for this configuration with 3 mm anvil truncation. The upper panel shows that on increasing hydraulic load, the cell pressure (measured using NaCl diffraction lines based on the Decker equation of state [3]) increases almost linearly, reaching ~7 GPa at 30 tons. The lower panel plots the vertical hydraulic ram stroke as a function of ram load. The 1.5 mm ram stroke from ~1 ton to 30 ton includes shortening in the sample assembly as well as elastic deformation of the entire Drickamer anvil column. Imaging indicates that majority of the shortening occurred inside the sample assembly, so that the initially 2.5 mm long sample chamber became 1 mm at the peak load.   In addition to the performance in pressure generation, Al alloy rings were found to be best suited for imaging quality, because it is the least absorbing material among the three, with the most homogeneous properties throughout.
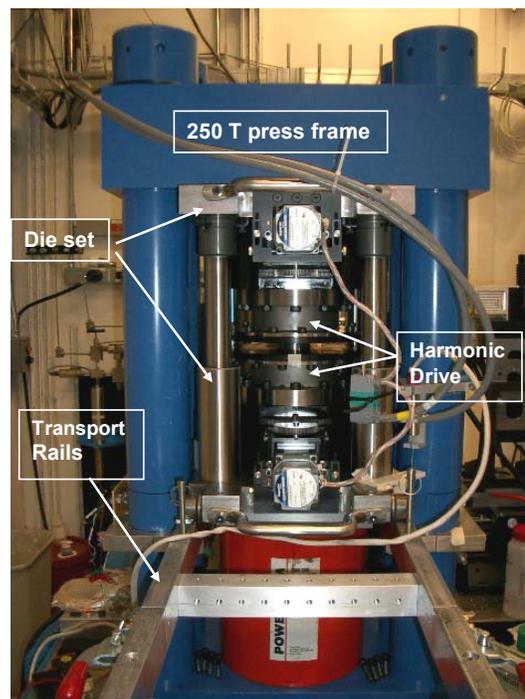
Fig. 3. Actual setup of the high pressure tomography apparatus inside a 250 ton hydraulic press frame.
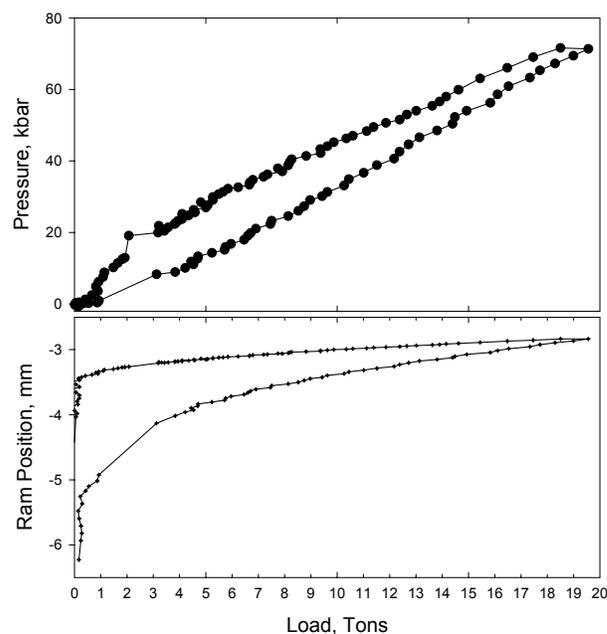
Fig. 4. Upper panel: pressure versus ram load for a 10 mm diameter Drickamer anvil cell. Lower panel: ram stroke versus load.

High-Pressure Tomography Imaging Test Results

Powered $Fe_{0.9}S_{0.1}$ was loaded into a 2 mm diameter sample chamber, with a 1 mm diameter sapphire sphere embedded in it. Both the sample chamber (3 mm in height) and gaskets were made of a mixture of amorphous boron and epoxy resin. The Fe-S sample was 2 mm in height, and a layer of hexagonal BN powder was added to the top of the sample to fill the sample chamber. The assembly was compressed to 12 tons, with tomographic images collected at 3 ton increment. At each

pressure, the high pressure tomography apparatus was rotated at an increment of 0.5°, from 0 to 179.5°.

Thetomography setup and procedure are similar to that given in [4]. The Si (111) monochromator was tuned to 45 keV, with collimated beam size of 2x3 mm. Flat field images were collected before and after the data collection by driving the apparatus several millimeters horizontally, perpendicular to the incident beam. The distance was determined based on the size of the sample as well as the thickness of the Al containment ring, so that the flat field image is not contaminated by the sample and, at the same time, stays completely within the "shadow" of containment ring. At each pressure, two sets of radiographs were taken. Each radiograph was collected for 10 s for 2x2 binned dataset and 40 s for unbinned dataset.
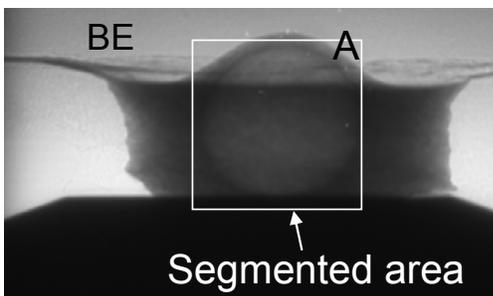
Fig. 5A. Tomograph based on data collected at 3 GPa. Dark trapezoidal shaow below the sample is WC anvil.
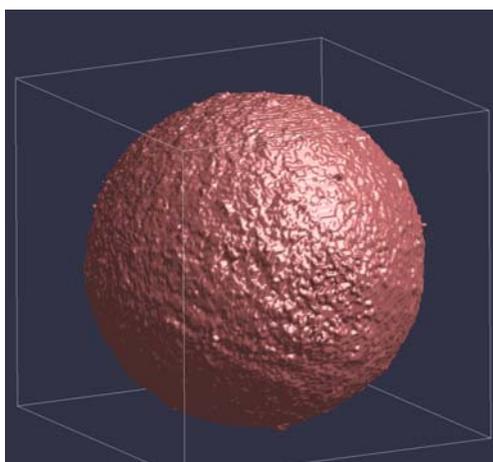
Fig. 5B. Extracted sapphire volume using Blob3D.

Figs. 5A and B show an example of the test result at 3 GPa. The sapphire sphere and the Fe-S sample were imaged clearly in Fig. 5A. By analyzing this tomograph using the software Blob3D, with a predefined intensity threshold, the Fe-S sample and the sphere were digitally separated and Fig. 5B shows the sapphire sphere image, which was then used to measure the sphere volume, with a typical error of about 0.5%. The results demonstrate that tomography imaging can be conducted using this apparatus. Furthermore, a systematic change in the sapphire sphere volume as a function of applied load (i.e., pressure) was observed.

Conclusions

We have developed the high-pressure tomography apparatus and successfully tested the feasibility of tomography microscope under high pressures. Work is under way to fully characterize the system in terms of spatial resolution, imaging quality, and high temperature capabilities. This technique has the following important potential applications:

1. Imaging microstructure evolution under high pressure and temperature.

2. Inclusions studies under pressure and temperature. By mimicking pressure and temperature conditions in the earth's deep interior, it is possible to simulate inclusion formation history in the laboratory using this apparatus, by collecting a sequence of tomographs

3. Glass and liquid properties. Our test experiment, although performed using a solid sample, demonstrates the feasibility of measuring sample volume by tomographic imaging. This technique can be useful in glass and liquid volume measurement, providing information on equations of state of non-crystalline materials. The 3-D information on glasses and liquids is also useful in the determination of density. The panoramic X-ray accessibility of the Drickamer cell also allows the determination of radial distribution function for glasses and liquids.

4. Deformation related microstructure evolution. By rotating the top and bottom Drickamer anvils in opposite directions, the apparatus can be used as the rotational deformation apparatus [2]. After deformation, both anvils can be rotated in synch to collect tomographs of the deformed the sample.

5. Neutron tomography. It is possible to apply similar concept for a much larger apparatus for high-pressure neutron tomography studies.

Acknowledgments

References

[1] Y. Wang, M. Rivers, T. Uchida, P. Murray, G. Shen, S. Sutton, J. Chen, Y. Xu, D. Weidner, *Proceedings of the International Conference on High Pressure Science and Technology (AIRAPT-17),* Science and Technology of High Pressure*, vol. 2, pp. 1047-1052 (2000).
[2] D. Yamazaki, S. Karato, *Rev. Sci. Instrum.*, **72**, 4207-4217 (2001).
[3] D.L. Decker, *J. Appl. Phys*. **42**, 3239-3244, 1971.
[4] M.L. Rivers, Y. Wang, T. Uchida, in: *Developments in X-Ray Tomography IV*, ed. U. Bonse, Proc. of SPIE Vol. 5535, pp. 783-791, 2004.