High Pressure Research

An International Journal

ISSN: 0895-7959 (Print) 1477-2299 (Online) Journal homepage: https://www.tandfonline.com/loi/ghpr20

Opposed type double stage cell for Mbar pressure experiment with large sample volume

Yoshio Kono, Curtis Kenney-Benson & Guoyin Shen

To cite this article: Yoshio Kono, Curtis Kenney-Benson & Guoyin Shen (2020): Opposed type double stage cell for Mbar pressure experiment with large sample volume, High Pressure Research, DOI: 10.1080/08957959.2019.1710744

To link to this article: https://doi.org/10.1080/08957959.2019.1710744

Published online: 08 Jan 2020.

HIGH PRESSURE RESEARCH
https://doi.org/10.1080/08957959.2019.1710744

Taylor & Francis
Taylor & Francis Group

Opposed type double stage cell for Mbar pressure experiment with large sample volume

Yoshio Kono[a,b], Curtis Kenney-Benson[c] and Guoyin Shen[c]

[a]Geophysical Laboratory, Carnegie Institution of Washington, Argonne, IL, USA; [b]Geodynamics Research Center, Ehime University, Matsuyama, Japan; [c]High Pressure Collaborative Access Team, X-ray Science Division, Argonne National Laboratory, Argonne, IL, USA

ABSTRACT

A new opposed type double-stage large volume cell has been developed to compress large volume samples to more than 100 GPa (Mbar) pressure. A pair of second-stage diamond anvils is introduced into the first-stage Paris–Edinburgh press. The double-stage large volume cell allows the generation of ultrahigh pressures using a large culet diameter of the second-stage diamond anvils (diameters of 0.5–1.2 mm). Pressure generation up to 131 GPa has been achieved by using the culet diameter of 0.5 mm. Sample volume of the double-stage large volume cell can be more than ~100 times larger than that of conventional Mbar experiment using a diamond anvil cell. The double-stage large volume cell has a large opening in the horizontal plane for X-ray measurements, which is particularly suited for the multi-angle energy dispersive X-ray diffraction measurement, thus opening a new way of in situ structural determinations of amorphous materials at Mbar pressures.

ARTICLE HISTORY
Received 1 September 2019
Accepted 27 December 2019

KEYWORDS
High pressure; Mbar; large volume; glass structure

1. Introduction

Large-volume samples are desired for accurate measurements of structure and properties of materials under ultrahigh pressure conditions by using X-ray and neutron techniques such as X-ray diffraction, X-ray spectroscopy, X-ray imaging, and neutron diffraction and imaging. Large volume presses such as cubic-type apparatus and Kawai-type multi-anvil apparatus have been used as a conventional tool for high pressure experiments with large sample volumes [1,2]. However, high pressure generation capability in a large volume press is typically limited up to ~30 GPa [1]. There are some pioneering works of higher pressure generation up to 65 GPa by using a special tungsten carbide anvil [3] or up to 109 GPa by using sintered diamond anvils with an additional diamond powder in the large volume cell [4]. However, both techniques require special anvils of significantly high cost, and therefore these techniques are difficult to use as a common tool in large volume press experiments.

The diamond anvil cell (DAC) is another common tool for high pressure experiments, which provides relatively easy access to pressures higher than 100 GPa. However, there

CONTACT Yoshio Kono kono.yoshio.rj@ehime-u.ac.jp Geodynamics Research Center, Ehime University, 2-5 Bunkyo-cho, Matsuyama, Ehime 790-8577, Japan

is a drawback that the sample volume in DAC experiments is normally limited to be very small. Recently, Boehler et al. [5] developed a large volume DAC for neutron diffraction measurement, and they succeeded in generating pressure up to 94 GPa using large 1 mm culet diameter anvils, which allows the use of a sample volume about 100 times larger than that of a conventional DAC. However, generating pressures far above 100 GPa remains challenging with this single stage configuration.

In this study, we introduce our recently developed opposed type double-stage large volume cell for large sample volume experiments at pressures higher than 100 GPa. The primary purpose of this new cell is for accurate measurement of pair distribution function of glasses and liquids at ultrahigh pressure conditions using multi-angle energy dispersive X-ray diffraction [6,7]. The double-stage large volume cell plays a critical role in the increase of X-ray signal from weakly scattering amorphous materials. In addition, large diffraction angle is essential for accurate determination of the structure factor with sufficiently large coverage of momentum transfer Q ($Q = 4\pi E \sin\theta/12.398$, where E is X-ray energy in keV and θ is the diffraction angle), and for high resolution in the reduced pair distribution function in real space. The new opposed type double-stage large volume cell is designed for compressing large volume samples to more than 100 GPa pressure with large solid angle access for X-ray measurements in the horizontal plan, which is beneficial not only for the structure measurement of amorphous materials but also for other applications such as X-ray tomography measurement.

2. Opposed type double-stage large volume cell

The double-stage large volume cell is developed in a 200 ton Paris–Edinburgh (PE) press [8]. A pair of second-stage diamond anvils is introduced into the first-stage PE cell (Figure 1). We used a cup-shaped tungsten carbide (WC) anvil with a 12 mm cup diameter with 3 mm flat bottom as the first stage anvil and (100)-oriented single crystal diamond with 0.5–1.2 mm culet as the second stage anvil. The first stage PE cell provides confining pressure on the second stage diamond anvils. Although there is no direct measurement of confining pressure in the PE cell in the double-stage configuration, we consider that the second stage diamond anvil is confined under pressure conditions similar to the normal PE cell experiments, which is ~3 GPa at 40 ton oil load and ~5 GPa at 80 ton oil load [7]. There have been similar attempts to generate high pressures by inserting diamond anvils inside multi-anvil large volume press [9–11]. However, these previous studies used cubic-type or Kawai-type multi-anvil presses, which severely limits solid angle access for X-ray diffraction. Combination of opposed anvils (both first- and second-stage) in our design provides a large opening in the horizontal plane for X-ray diffraction measurement of amorphous materials and/or other applications.

We used (100)-oriented single crystal diamond anvil with the Diacell design of 2.5 mm diameter (Almax easyLab) as the second stage anvil. The facet of the diamond anvil is mantled by the hexagonal boron nitride (hBN) pressure medium (Figure 1). The gasket is composed of a cubic boron nitride (cBN) + epoxy (10:1 in weight ratio) inner gasket with a metal outer gasket made of aluminum alloy (7075) or beryllium as X-ray transparent materials. Use of X-ray transparent gasket materials is required for X-ray measurement through the gasket, because it is not possible to path X-ray through the gasket gap by tilting the press, due to the limited gap of the first-stage WC anvil of ~0.9 mm at the

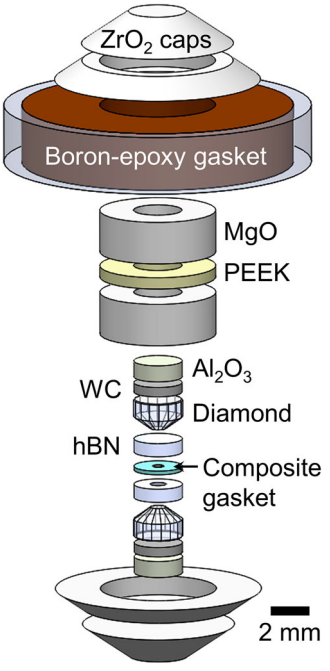

Figure 1. The extended view of the used parts in the cell assembly of the double-stage large volume cell. The second stage diamond anvils are inserted in the first stage Paris–Edinburgh cell. The composite gasket is composed of cBN+epoxy inner gasket with a metal outer gasket made of aluminum alloy (7075) or beryllium.

highest load of 100 ton, which provides only ±~4° opening in vertical direction. A piece of Au (cut from 0.05 mm diameter wire) is placed at the edge of the sample hole to determine pressure by the equation of state of Au [12]. The piece of Au was placed at the edge of the sample hole to avoid contamination of the X-ray diffraction signal from Au into the structure measurement of the glass sample. The pressure gradient between the center and edge of the sample hole in the 0.8 mm culet second-stage anvil design was up to 4 GPa at pressures up to 93.4 GPa [13].

Table 1 summarizes the configuration of the gasket with a different culet size of the second stage diamond anvil. The first development was carried out using a culet diameter of 0.8 mm [13]. The large culet size of 0.8 mm allowed us to use a large sample of 0.3 mm in diameter and 0.15 mm in height. The 0.8 mm culet anvil design has reached pressures up to 92 GPa [13] (Figure 2). It has been known that use of cBN+epoxy gasket significantly increases thickness of sample by several times thicker than that of conventional metal gasket [14], which plays important role to prevent contact of the edges of

Table 1. Configuration of the double-stage large volume cell and the generated highest pressure.

Culet size (mm)	Outer gasket material	Gasket thickness (mm)	Sample hole size (mm)	Highest pressure (GPa)
1.2	Al alloy	0.20	0.50	58
0.8	Al alloy	0.15	0.30	92
0.6	Al alloy	0.15	0.23	111
0.6	Beryllium	0.15	0.23	124
0.5	Beryllium	0.15	0.20	131

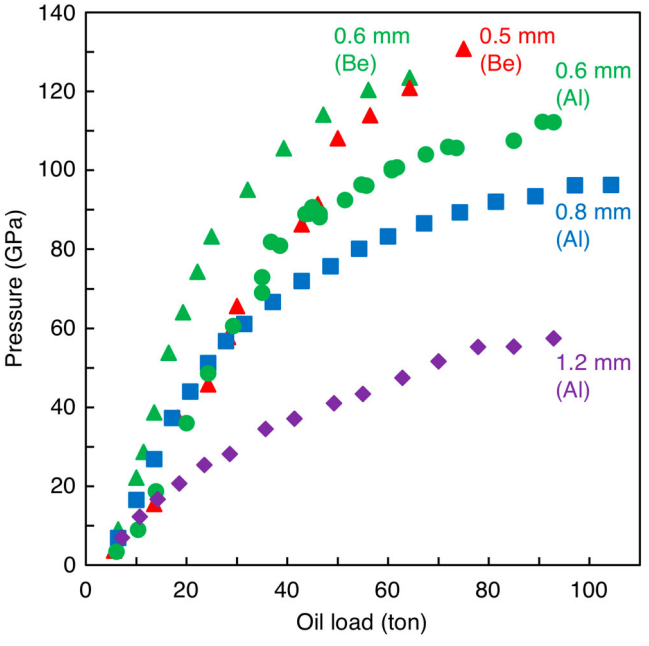

Figure 2. Pressure generation as a function of oil load of the first stage Paris–Edinburgh press. Culet diameters between 0.5 and 1.2 mm are used as the second-stage anvil with Al alloy (Al) or beryllium (Be) as outer gasket materials.

the diamond anvils and to keep thickness of sample even at ultrahigh pressure conditions. X-ray radiography measurement in the experiment shows that the sample size was ~0.24 mm in diameter and ~0.06 mm in height at the in situ ultrahigh pressure condition of 92 GPa [13].

Efforts have been made to generate pressures higher than 100 GPa. A culet diameter for the second stage anvil of 0.6 mm was adopted in Kono et al. [15]. For the 0.6 mm culet diameter anvil design, the diameter of the sample hole is reduced to 0.23 mm with a gasket thickness of 0.15 mm. Al alloy was used as outer gasket material in Kono et al. [15], the same as the 0.8 mm culet anvil design. The 0.6 mm culet anvil design enabled us to generate more than 100 GPa. Pressure generation up to 111 GPa was achieved in Kono et al. [15] (Figure 2). In addition, to generate higher pressures, we adopted beryllium as outer gasket material, instead of the Al alloy used in Kono et al. [13,15] (Table 1). Beryllium is harder material with markedly higher strength than Al alloy (7075). Use of beryllium as the outer gasket with the same gasket geometry as the Al alloy outer gasket enhanced pressure generation to 124 GPa (Figure 2).

We also have carried out experiments by using the second stage anvils with a culet size of 0.5 mm. The highest pressure generation of 131 GPa was achieved by a beryllium gasket with a sample hole size of 0.2 mm and a thickness of 0.15 mm [16] (Figure 2). The pressure generation of 131 GPa is an important landmark particularly in the study of the Earth's deep interior because it is comparable to the pressure at the bottom of the Earth's mantle (135 GPa). On the other hand, it is important to note that the initial pressure generation efficiency of the 0.5 mm culet experiment is lower than that of the 0.6 mm culet experiment (Figure 2). There is no difference in the cell design between the 0.6 and

0.5 mm culet experiments except for the different sample diameters (Table 1). We guess that use of smaller culet size of 0.5 mm with the same initial sample thickness as the 0.6 mm culet experiment might cause more flow of the sample and gasket materials at the beginning of compression, and therefore initial pressure generation efficiency might be lower in the 0.5 mm culet experiment than that of the 0.6 mm culet experiment. So far, we have used 0.5 mm culet anvil only in the experiment by Ohira et al. [16]. We expect that more experiments with variable gasket geometry will allow for higher pressures to be reached by using the 0.5 mm culet second-stage anvil cell.

In contrast to the challenges for >100 GPa ultrahigh pressure generation, we have designed a double-stage large volume cell using a very large culet diameter of 1.2 mm as the second-stage diamond anvil. The 1.2 mm culet anvil allows for use of a significant sample volume in the 0.5 mm diameter sample hole and 0.2 mm thick gasket (Table 1). This development opened a new way to investigate pair distribution functions of low-Z amorphous materials such as glassy carbon [17] (cf. Section 4). We have succeeded in generating pressures up to 58 GPa by using the 1.2 mm culet anvil design (Figure 2).

3. Comparisons of large sample volume Mbar experiments with conventional DAC experiment

Our developed double-stage large volume cell enables the use of larger culet size of the second stage diamond anvil. As a result, the device can be used to compress significantly larger sample volume under Mbar pressure conditions, compared to the traditional DAC device. Dunstan and Spain [18] proposed an inverse square relationship between the culet diameter and the maximum achievable pressure ($P_{max} = \frac{12.5}{d^2}$, where d is culet diameter (mm)) in conventional DAC experiments at relatively low pressure conditions to ~30 GPa (Figure 3). Dunstan and Spain [18] noted that the maximum pressure used in experiments should be somewhat lower than this relationship, such as $0.8P_{max}$. O'Bannon et al. [19] showed another relation between the culet diameter and the maximum pressure generation in DAC experiments by fitting to the compiled data set at the pressure conditions to 1000 GPa ($P_{max} = 1727(1000d)^{-0.54}$). The P_{max}-culet diameter relationship of O'Bannon et al. [19] well reproduces the compiled data set above around 80 GPa.

The P_{max}-culet diameter relationship of Dunstan and Spain [18] shows P_{max} of 9 GPa for the culet diameter of 1.2 mm, of 20 GPa for 0.8 mm culet diameter, and of 35 GPa for 0.6 mm culet diameter (Figure 3). In contrast, the highest pressures generated by using the culet diameter of 1.2, 0.8, and 0.6 mm in our double-stage large volume cell are 58, 92, and 124 GPa, respectively, which are significantly higher than those of conventional DAC experiments (Figure 3). In addition, the P_{max}-culet diameter relationship of O'Bannon et al. [19] shows that 0.2 mm is the typical culet diameter required to generate 100 GPa in conventional DAC experiments. On the other hand, our double-stage large volume cell succeeded in generating near 100 GPa pressure (92 GPa) by a 0.8 mm culet second stage anvil, and to much higher pressures of 124 GPa by a 0.6 mm culet second stage anvil. Use of such large culet diameters in the double-stage large volume cell enabled us to compress ~100 times larger sample volume in Mbar pressure experiments.

Other efforts have been made to compress a large sample volume to Mbar pressure conditions. It has been reported that the use of nano-polycrystalline diamond (NPD) as

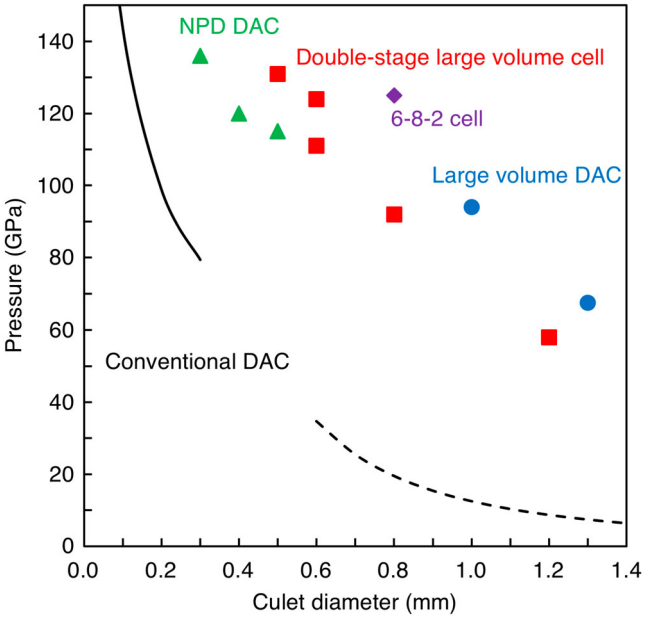

Figure 3. The maximum achieved pressure as a function of culet diameter of the diamond anvils. Black dashed [18] and solid [19] lines are pressure generation by conventional diamond anvil cell (DAC). Markers represent the maximum generated pressures by large sample volume experiments (large volume DAC: [5]; 6-8-2 cell: [11]; double-stage large volume cell: this study; NPD (nano-polycrstalline diamond) DAC: [20]).

anvil material enhances the achievable pressure in conventional DAC experiments for large culet anvils of more than 0.3 mm [20] because NPD has high toughness and isotropic mechanical properties. Nakamoto et al. [20] showed a pressure generation of 115 GPa using 0.5 mm culet NPD anvils (Figure 3), which is markedly higher than that of 68 GPa by using single crystal diamond anvils. The data clearly shows the enhancement of pressure generation by using tough NPD anvils in DAC. On the other hand, our double-stage large volume cell showed pressure generation up to 131 GPa by using 0.5 mm culet second-stage anvils, which is higher than that of the NPD DAC experiment (Figure 3). In addition, the double-stage large volume cell currently uses a single crystal diamond as the second stage anvil. Further high pressure generation may be possible by using the NPD as the second stage anvil of the double-stage large volume cell.

In addition, there are two other types of previous studies for large volume sample compression to Mbar pressure. One is the large volume DAC developed by Boehler et al. [5]. Boehler et al. [5] developed a new design of strongly supported conical diamond anvils for neutron diffraction, and they succeeded in generating 94 GPa using 1.0 mm culet diamond anvils, which is a larger anvil size than the second-stage anvil of 0.8 mm in our double-stage large volume cell used to generate similar pressure (Figure 3). Another type of large sample volume Mbar experiment is 6-8-2 type multi-anvil apparatus [11]. Kunimoto and Irifune [11] designed the triple-stage multi-anvil apparatus by inserting an additional set of the third-stage diamond anvil in Kawai-type (6-8 type) multi-anvil apparatus. They have reported a pressure generation of 125 GPa by using 0.8 mm culet diamond anvil as the third stage anvil, which is markedly higher than that of our double-stage large volume cell

with the same culet diameter of 0.8 mm (Figure 3). The 6-8-2 multi-anvil apparatus design provides confining pressure of ~20 GPa on the third stage anvil [10], which is much higher than that of the double-stage large volume cell (up to ~7 GPa). The high confining pressure on the third stage anvil by the multi-anvil press in the 6-8-2 cell may play an important role in high pressure generation capability. On the other hand, note that there is a drawback of the use of the Kawai-type multi-anvil apparatus for high confining pressure. The opening access is severely limited in the Kawai-type multi-anvil apparatus for X-ray diffraction with a typical 2θ angle of only 6°, which limits the applicability of the 6-8-2 cell in X-ray experiments. The opposed type double-stage large volume cell uses opposed anvils for both first and second stages, and it provides a large opening (140 degrees) in the horizontal plane for X-ray measurements such as X-ray diffraction measurement at high Q for structure measurement of amorphous materials, and X-ray tomography measurement.

4. Applications in the structural measurement of glasses under ultrahigh pressure conditions

One of the foremost applications of the double-stage large volume cell is the structural measurement of amorphous materials under ultrahigh pressure conditions. The large opening in the horizontal plane for X-ray diffraction measurement is well suited for the structure factor measurement in a wide coverage of Q. Combination of the double-stage large volume cell and multi-angle energy dispersive X-ray diffraction have enabled in situ structure measurements of glasses at ultrahigh pressure conditions [13,15–17]. The multi-angle energy dispersive X-ray diffraction measurement was conducted at Beamline 16-BM-B, HPCAT of the Advanced Photon Source [7]. Figure 4 shows examples of the structure factor (S(Q)) measurements for GeO_2 glass at 92 GPa [13], $MgSiO_3$ glass at 88 GPa [15], Al_2O_3–SiO_2 glass at 131 GPa [16], and glassy carbon at 49 GPa [17]. Combination of the double-stage large volume cell with the multi-angle energy dispersive X-ray diffraction provides good quality structure factor data with the Q range larger than 13 $Å^{-1}$.

Kono et al. [13] first succeeded in measuring the structure of GeO_2 glass to 92 GPa (Figure 4). The glass structure measurement was conducted by using unfocused white X-rays with an acquisition time of ~4.5 h for 1 structure factor measurement. However, using the unfocused white X-rays, the structure measurement of silicate glass in the double-stage large volume cell was a challenge, due to the weak X-ray scattering from light elements in silicate glass compared to GeO_2 glass. In order to overcome this issue, Kono et al. [15] installed a 200-mm-long Pt-coated K–B mirror with an incident angle of 1.25 mrad to focus white X-rays in the horizontal direction. The K–B mirror in the horizontal direction increased X-ray signal by about 2.5 times, and Kono et al. [15] succeeded in measuring the structure factor of $MgSiO_3$ glass at high pressures, with the quality similar to that of GeO_2 glass using similar acquisition time of ~3 h (Figure 4). In addition, the K–B mirrors were installed in both horizontal and vertical directions to further increase the X-ray flux, and Ohira et al. [16] have succeeded in measuring structure factors of Al_2O_3–SiO_2 glass up to 131 GPa by using the smaller 0.5 mm culet second stage anvil (Figure 4). Furthermore, Shibazaki et al. [17] succeeded in measuring structure factors of glassy carbon up to 49 GPa (Figure 4). Since glassy carbon is an amorphous material composed of a light element composition, the structure measurement required a large volume sample with focused white X-rays. Use of 1.2 mm culet anvil in the double-stage large

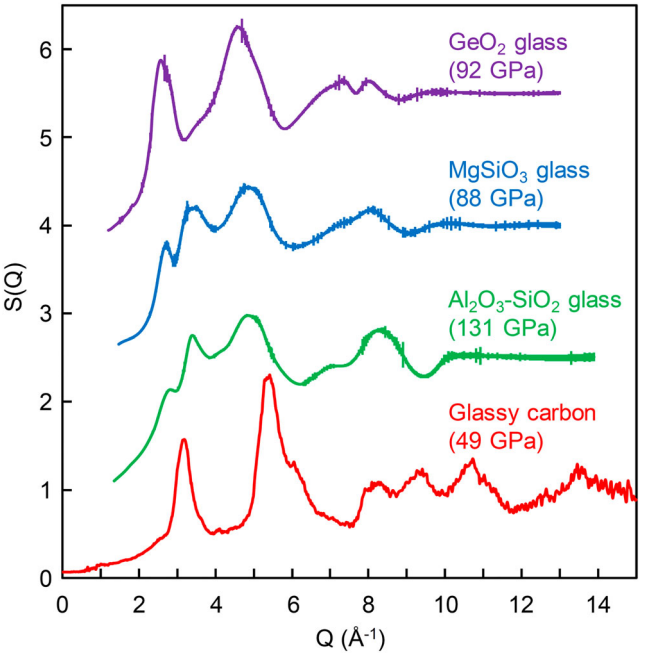

Figure 4. Structure factor ($S(Q)$) of GeO_2 glass at 92 GPa [13], $MgSiO_3$ glass at 88 GPa [15], Al_2O_3–SiO_2 glass at 131 GPa [16], and glassy carbon at 49 GPa [17] measured by using the double-stage large volume cell combined with multi-angle energy dispersive X-ray diffraction.

volume cell allows compression of a large volume sample of 0.5 mm in diameter and 0.2 mm in height, and it enabled us to measure structure factor of glassy carbon up to 49 GPa (Figure 4), although it required an acquisition time of ~9 h for the 1 structure factor measurement. These studies clearly show that the double-stage large volume cell experiment combined with multi-angle energy dispersive X-ray diffraction measurement opens a new way to investigate structure of amorphous materials under ultrahigh pressure conditions, which can play important roles in clarifying several unsolved scientific issues such as pressure-induced polyamorphic transformations of silicate magmas under >100 GPa pressure conditions of the Earth's core-mantle boundary.

Acknowledgements

We acknowledge two anonymous reviewers for valuable comments.

Disclosure statement

No potential conflict of interest was reported by the authors.

Funding

This study is supported by the National Science Foundation [award no. EAR-1722495]. This work was performed at HPCAT (Sector 16), Advanced Photon Source (APS), Argonne National Laboratory.

HPCAT operations are supported by DOE-NNSA's Office of Experimental Sciences. The Advanced Photon Source is a U.S. Department of Energy (DOE) Office of Science User Facility operated for the DOE Office of Science by Argonne National Laboratory [contract no. DE-AC02-06CH11357].

References

[1] Katsura T, Funakoshi K-i, Kubo A, et al. A large-volume high-pressure and high-temperature apparatus for in situ X-ray observation, 'SPEED-Mk. II'. Phys Earth Planet Int. 2004;143:497–506.
[2] Wang Y, Rivers M, Sutton S, et al. The large-volume high-pressure facility at GSECARS: a 'Swiss-army-knife' approach to synchrotron-based experimental studies. Phys Earth Planet Int. 2009;174:270–281.
[3] Ishii T, Yamazaki D, Tsujino N, et al. Pressure generation to 65 GPa in a Kawai-type multi-anvil apparatus with tungsten carbide anvils. High Press Res. 2017;37:507–515.
[4] Yamazaki D, Ito E, Yoshino T, et al. Over 1 Mbar generation in the Kawai-type multianvil apparatus and its application to compression of (Mg0. 92Fe0. 08) SiO3 perovskite and stishovite. Phys Earth Planet Int. 2014;228:262–267.
[5] Boehler R, Guthrie M, Molaison JJ, et al. Large-volume diamond cells for neutron diffraction above 90 GPa. High Press Res. 2013;33:546–554.
[6] Egami T. Structural study by energy dispersive X-ray diffraction. In: Güntherodt HJ, Beck H, editors. Glassy metals I. Berlin: Springer; 1981. p. 25–44.
[7] Kono Y, Park C, Kenney-Benson C, et al. Toward comprehensive studies of liquids at high pressures and high temperatures: combined structure, elastic wave velocity, and viscosity measurements in the Paris–Edinburgh cell. Phys Earth Planet Int. 2014;228:269–280.
[8] Klotz S, Hamel G, Frelat J. A new type of compact large-capacity press for neutron and X-ray scattering. High Press Res. 2004;24:219–223.
[9] Endo S, Ito K. Electrical resistance of αFe2O3 under ultrahigh static pressure. Solid State Commun. 1980;36:189–190.
[10] Kunimoto T, Irifune T, Sumiya H. Pressure generation in a 6-8-2 type multi-anvil system: a performance test for third-stage anvils with various diamonds. High Press Res. 2008;28:237–244.
[11] Kunimoto T, Irifune T. Pressure generation to 125 GPa using a 6-8-2 type multianvil apparatus with nano-polycrystalline diamond anvils. J Phys Conf Ser. 2010;215:012190. Epub 1.
[12] Tsuchiya T. First-principles prediction of the P-V-T equation of state of gold and the 660-km discontinuity in Earth's mantle. J Geophys Res. 2003;108:2462.
[13] Kono Y, Kenney-Benson C, Ikuta D, et al. Ultrahigh-pressure polyamorphism in GeO2 glass with coordination number> 6. Proc Natl Acad Sci USA. 2016;113:3436–3441.
[14] Funamori N, Sato T. A cubic boron nitride gasket for diamond-anvil experiments. Rev Sci Instrum. 2008;79:053903.
[15] Kono Y, Shibazaki Y, Kenney-Benson C, et al. Pressure-induced structural change in MgSiO3 glass at pressures near the Earth's core–mantle boundary. Proc Natl Acad Sci USA. 2018;115:1742–1747.
[16] Ohira I, Kono Y, Shibazaki Y, et al. Ultrahigh pressure structural changes in a 60 mol.% Al2O3–40 mol.% SiO2 glass. Geochem Perspect Lett. 2019;10.
[17] Shibazaki Y, Kono Y, Shen G. Compressed glassy carbon maintaining graphite-like structure with linkage formation between graphene layers. Sci Rep. 2019;9:7531.
[18] Dunstan DJ, Spain IL. Technology of diamond anvil high-pressure cells: I. Principles, design and construction. J Phys E Sci Instrum. 1989;22:913.
[19] O'Bannon Iii EF, Jenei Z, Cynn H, et al. Contributed review: culet diameter and the achievable pressure of a diamond anvil cell: implications for the upper pressure limit of a diamond anvil cell. Rev Sci Instrum. 2018;89:111501.
[20] Nakamoto Y, Sakata M, Sumiya H, et al. Note: high-pressure generation using nano-polycrystalline diamonds as anvil materials. Rev Sci Instrum. 2011;82:066104.