Pressure generation up to 55 GPa using a Drickamer-type apparatus with sintered diamond anvilstoward use of nano-polycrystalline diamond as anvils

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Abstract. Pressure calibration experiments were performed at room temperature for a Drickamer-type apparatus using a composite anvil system with a sintered diamond anvil, which can be replaced by nano-polycrystalline diamond anvil. The culet diameter and tapering angle are 1.0 mm and 30°, respectively. Transitions in Fe₂O₃ (50 GPa) and CoO (55 GPa) were observed at 39.6 and 38.5 kN, respectively, by electrical resistance measurements. Pressure of about 120 GPa can be generated at the maximum load of 98 kN by assuming the linear extrapolation of the obtained load-pressure relation. The Drickamer-type apparatus with nano-polycrystalline diamond anvils has potential to generate pressure covering the whole lower mantle.

1. Introduction

Drickamer-type apparatus is an opposed anvil type apparatus, which is composed of a pair of opposed anvils and a containment ring [1]. This apparatus has been used for electrical resistance measurements under high pressure and low temperature [2] since 1970s. This apparatus has also been used for X-ray diffraction experiments under high pressure and temperature [3, 4]. However, since 1980s, the most extensively used opposed anvil type apparatus is diamond anvil cell (DAC) because of dramatic improvements of intense synchrotron radiation for X-ray diffraction measurements under high pressure and of laser technology for high temperature generation in very small area (~100 μm²).

Irifune et al. [5] succeeded to synthesize nano-polycrystalline diamond (NPD) by direct conversion from graphite. NPD is harder and tougher than single crystal diamond. Therefore, the NPD is the most suitable material for anvils of high pressure devices. In Geodynamics Research Center, Ehime University, NPD materials with diameter exceeding 5 mm have already been synthesized and can be used as anvils for high pressure devices. We are trying to develop a Drickamer apparatus with NPD anvils, which can serve as a large DAC, allowing larger sample size of 0.1~0.2 mm in diameter. The larger sample size is suitable for internal resistive heating and experiments in complex rock compositions, such as pyrolite and MORB.

In the present study, we carried out preliminary high pressure generation tests of a Drickamer-type using a composite anvil system with a sintered diamond anvil, which can be replaced by a NPD anvil.

³ The original title of the present paper is “Plastic deformation of polycrystalline sintered diamond materials in the DDIA with HIMEDIA pistons”.

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Results of some pressure calibration experiments and the obtained load versus pressure curves are presented in this paper.

![schematic drawings of a Drickamer-type apparatus with composite anvils.](image)

**Figure 1.** Schematic drawings of a Drickamer-type apparatus with composite anvils. (a) whole view. 1, a containment ring; 2, a composite anvil; 3, gasket-sample assembly. (b) composite anvil. 1, a sintered diamond anvil; 2, tungsten carbide anvil backup; 3, stainless-steel support ring. (c) sample assembly for pressure calibration at room temperature. 1, a sintered diamond anvil; 2, pyrophyllite spacer; 3, pyrophyllite gasket; 4, single crystal MgO sample capsule; 5, Mo electrode; 6, pressure calibrant.

2. Experimental method

Pressure calibration experiments at room temperature were carried out for a Drickamer-type apparatus. This apparatus consists of the three major parts, a containment ring, opposed anvils, and gasket-sample assembly part (figure 1a). The containment ring (inner diameter: 10.15 mm; outer diameter: 40 mm; height: 19 mm) is made of a tool steel.

Figure 1b shows a schematic drawing of a composite anvil. The composite anvil consists of a sintered diamond anvil, a support ring made of a stainless steel, and tungsten carbide (WC) backup. The sintered diamond (SD) anvil with 1 mm culet diameter and tapering angle of 30° was made by Tomei Diamond Co. Ltd. The diameter and height of the WC backup (Fuji Die Co. Ltd.) is 10 mm and 6 mm, respectively. A copper foil with thickness of 0.03 mm was placed between the sintered diamond anvil and the WC backup to avoid local stress concentration. The side walls of the support ring and the WC backup were completely covered by a polyimide tape for electrical insulation and lubrication. The maximum load is 98 kN where pressure behind the SD anvil reaches 5 GPa.

We employed GaP, Fe$_2$O$_3$, and CoO as pressure calibrants. GaP is widely used as a pressure calibrant because it has a semiconductor to metal transition at 23 GPa. In Fe$_2$O$_3$, Pasternak et al. [6] reported presence of a first-order insulator-metal transition at 50 GPa and room temperature using diamond anvil cell (DAC). They performed electrical resistance measurement during compression and observed a precipitous decrease by about 6 orders of magnitude at this pressure. We employed this transition as a pressure fixed point at 50 GPa [6]. In CoO, Atou et al. [7] observed a drastic decrease of electric resistance by about 8 orders of magnitude between 43 and 63 GPa, which is concurrent with structural phase transition from a cubic to a rhombohedral phase. In their figure (Fig. 2 in [7]), the largest resistance decrease by about 4 orders of magnitude can be seen at 55 GPa. Therefore we employed this transition as a pressure fixed point at 55 GPa.

Figure 1c shows a schematic illustration of the sample assembly. A pyrophyllite gasket with thickness of 0.3 mm was employed. This gasket has a hole with 0.4 mm diameter at the center. Molybdenum disks (0.1 mm thick) as electrodes were embedded in the top and bottom part of the gasket hole. SD anvils were also used as electrodes because they are conductive. A sample capsule (outer diameter: 0.4 mm; inner diameter: 0.15 mm; height: 0.1 mm) made of single crystal MgO was used and the powered sample was packed into the chamber.

The Drickamer-type anvil assembly was compressed by a small hydraulic press, RP-50 (Ryosen Engineers Co. Ltd.), whose height and weight are 170 mm and 8.5 kg, respectively. In addition to the pressure calibration runs using the composite anvil with SD anvil, we carried out experiments using whole WC anvils whose culet diameter and tapering angle are the same as those of the SD anvil. In these experiments, we used ZnS (15.6 GPa) and GaP as pressure calibrants.
3. Results and discussion

Figure 2a shows an example of resistance measurements as a function of load in Fe$_2$O$_3$. In this case, resistance was higher than $10^9$ ohm below 20 kN. Above 20 kN, we observed gradual decrease of resistance from $\sim 10^8$ to $\sim 10^5$ ohm. This behavior is similar to that observed by Pasternak et al. [6]. At 39.6 kN, we observed a large decrease of resistance by about 4 orders of magnitude. Using this observation, we judged that pressure of 50 GPa was generated at 39.6 kN.

An example of resistance measurements with applied load in CoO is shown in Figure 2b. Resistance increased gradually from $\sim 10^6$ to $\sim 10^7$ ohm between 0 and 30 kN. Above 30 kN, resistance remained constant and a precipitous decrease by about 7 orders of magnitude occurred at 38.5 kN. The magnitude of resistance drop is similar to that observed by Atou et al. [7]. We judged that pressure of 55 GPa was generated at 38.5 kN.

The transition in Fe$_2$O$_3$ at 50 GPa was observed at 39.6 kN, whereas the transition in CoO at 55 GPa was observed at 38.5 kN. One of the possible explanations of this discrepancy is that uncertainty of transition pressures in Fe$_2$O$_3$ and CoO. For example, in Fe$_2$O$_3$, Ito et al. [8] reported that a large resistance decrease (3 orders of magnitude) was observed at 54±1 GPa using a Kawai-type apparatus with sintered diamond anvils. This difference between Pasternak et al. [6] and Ito et al. [8] might be explained by difference of pressure standard (ruby fluorescence in [6]; an equation of state of MgO in [8]) and of stress state produced by the different ways of compression. Regarding CoO, as far as we know, there has been no study except Atou’s study. More experimental studies should be performed to make Fe$_2$O$_3$ and CoO reliable pressure calibrants. Small machining errors of sample assembly parts can also explain this discrepancy. It has been well known that distance between two opposed anvil surfaces strongly affects pressure generation efficiency [9]. If the distance in the CoO run was shorter than that in the Fe$_2$O$_3$ run because of the machining error, the discrepancy, the higher pressure was generated at the lower load, could be explained.

Microscope photographs of the recovered samples assemblies are shown in Figure 3 (a: the run using WC anvil for observation of GaP transition at 23 GPa; b: the run using SD anvil for observation of CoO transition at 55 GPa). In these photographs, molybdenum electrodes are seen as white parts. In figure 3a, diameter of molybdenum electrodes is 0.590 mm, and thickness of the sample assembly is 0.125 mm. In figure 3b, the diameter is 0.927 mm and the thickness is 0.116 mm. The sample assembly recovered from the run of CoO is larger in diameter and thinner than that recovered from the run of GaP. This can be attributed to difference in experienced pressure and in anvil material. The WC anvils were plastically deformed after the run for GaP transition, whereas we did not observe any plastic deformation for SD anvils. In addition, in both the figures, we can clearly see that a molybdenum disk is completely separated from the other by the MgO capsule and the sample, which ensures that resistance measurements of the samples were successfully carried out.

Figure 3. Examples of microscope photographs of recovered samples. (a) GaP run using WC anvils. (b) CoO run using SD anvils. Scale bars represent 0.5 mm.
Figure 4 shows a summary of load-pressure relations obtained in the present study and comparison with that of a previous study [10]. Using WC anvil with culet diameter of 1 mm and tapering angle of 30°, pressure of 23 GPa was generated at 31.7 kN, whereas pressure of about 50 GPa was generated at about 40 kN using SD anvil with the same culet diameter and tapering angle. Pressure generation efficiency of SD anvil is almost twice as that of WC anvil. This can be mainly attributed to difference in compressional strength of anvils. A load-pressure relation obtained by Akahama et al. [10] is also shown in this figure. They obtained this relation in a Drickamer-type apparatus using SD anvils with 0.9 mm culet diameter and tapering angle of 15°. The relation obtained by Akahama et al. [10] is less efficient than that obtained for SD anvil in the present study, which might be attributed to difference of tapering angle; anvil with steeper tapering angle can generate higher pressure. If we can extrapolate the load-pressure relation of SD anvil linearly, pressure of about 120 GPa can be generated at the maximum load of 98 kN. Since hardness of NPD is higher than that of any materials, load-pressure relation of NPD anvil can be more efficient than that obtained for the SD anvil. In addition, NPD material with diameter larger than 5 mm can be synthesized in GRC, Ehime University. Use of the anvils with larger diameter makes the maximum load higher (see figure 4). Therefore, use of NPD anvils with diameter larger than 5 mm has potential to generate pressure covering the whole lower mantle, up to 135 GPa. We are planning to carry our Drickamer apparatus with NPD anvils into a synchrotron facility to measure pressures above 55 GPa using pressure markers such as gold and periclase.

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