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## ORIGINAL ARTICLE

# Recent advances of high-pressure generation in a multianvil apparatus using sintered diamond anvils

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**Abstract** The tried and tested multianvil apparatus has been widely used for high-pressure and high-temperature experimental studies in Earth science. As a result, many important results have been obtained for a better understanding of the components, structure and evolution of the Earth. Due to the strength limitation of materials, the attainable multianvil pressure is generally limited to about 30 GPa (corresponding to about 900 km of the depth in the Earth) when tungsten carbide cubes are adopted as second-stage anvils. Compared with tungsten carbide, the sintered diamond is a much harder material. The sintered diamond cubes were introduced as second-stage anvils in a 6–8 type multianvil apparatus in the 1980s, which largely enhanced the capacity of pressure generation in a large volume press. With the development of material synthesis and processing techniques, a large sintered diamond cube (14 mm) is now available. Recently, maximum attainable pressures reaching higher than 90 GPa (corresponding to about 2700 km of the depth in the Earth) have been generated at room temperature by adopting 14-mm sintered diamond anvils. Using this technique, a few researches have been carried out by the quenched method or combined with synchrotron radiation in situ observation. In this paper we review the properties of sintered diamond and the evolution of pressure generation using sintered diamond anvils. As-yet unsolved problems and perspectives for uses in Earth Science are also discussed.

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## 1. Introduction

High-pressure experimental techniques have been widely used in such fields as mineralogy, petrology, geophysics, and material science. Discerning the properties and behavior of the Earth and planetary minerals and rocks under high pressures provides indispensable information for the understanding of the chemical composition, structure, dynamics, and origin of the Earth and planets.

Generally, there are three major high-pressure devices used in high-pressure experimental studies for the Earth and planets, including the piston-cylinder apparatus, the multianvil apparatus



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(MAA), and the diamond-anvil cell (DAC). Each apparatus has its own advantages and unique applications. The piston-cylinder apparatus can provide accurate pressure measurements on a force-per-area basis. By adopting a 1/2-inch piston, pressure can be generated up to 4 GPa, corresponding to about 130 km depth of the Earth. Therefore, the piston-cylinder apparatus has been widely used for high-pressure and high-temperature experimental study under crustal and upper mantle conditions, as well as for synthesizing some starting materials for further experiments. The DAC can generate high pressures between two gem-quality single crystal diamonds (SCD), with the maximum reported pressure around 550 GPa (Xu et al., 1986), corresponding to the pressure of Jupiter's mantle. The DAC is widely used for investigating phase transitions and physical properties of materials at high pressures, combined with external or laser heating systems and optical and spectroscopic methods. The disadvantages of DAC include small sample size (on the order of  $\mu\text{m}$ ) and the inevitable presence of large pressure and temperature gradients through the sample. The MAA can routinely reach about 28 GPa by adopting tungsten carbide (WC) anvils, corresponding to about 750 km depth of the Earth; Katsura et al. (2004) once generated 31 GPa using WC anvils. The MAA has been widely employed for high-pressure and high-temperature experimental studies during the last several decades under upper mantle and uppermost lower mantle conditions. Its advantages include large sample size (on the order of mm), a quasi-hydrostatic pressure environment, uniform temperature field, and precise control of pressure–temperature (P–T) conditions, but the pressure is limited due to the hardness of WC. The pressure limitation in MAA makes it impossible to study the phase relations, physical and chemical properties of minerals and rocks in the deeper mantle, so that the advantages of MAA compared with DAC are not fully displayed.

With the development of material science and technology, sintered diamond (SD), which is much harder than WC, became available for use as an anvil material for the MAA (Endo et al., 1985, 1987; Utsumi et al., 1986). In the late 1980s, small cubic SD anvils were used for high-pressure generation to 41 GPa (Ohtani et al., 1989). The size of the SD anvil has been extended in the 1990s from a 5–10-mm to 14-mm edge length and this high-pressure technique has been developed and applied to study mineral properties and phase transitions in the MAA by using the traditional quenched method (Ito et al., 1998), or by combining with the synchrotron X-ray diffraction technique (Kato et al., 1992; Kondo et al., 1993; Funamori et al., 1996a, 1996b). High-pressure generation using SD anvils has been extended significantly, especially in

the last decade, including the Earth Science research field with many studies carried out (Ito, 2000; Ono et al., 2000, 2001; Irifune, 2002; Irifune et al., 2002; Ito and Kubo, 2002; Kubo et al., 2003; Yamazaki and Irifune, 2003; Ito et al., 2004; Ohtani, 2004; Sueda et al., 2004; Ito et al., 2005; Ito, 2006; Yamazaki et al., 2006; Ito, 2007; Stewart et al., 2007; Kubo et al., 2008; Shinmei et al., 2008; Tange et al., 2008; Ito et al., 2009, 2010; Katsura et al., 2009; Sueda et al., 2009; Yamazaki et al., 2011). In this paper we review the advances of high-pressure generation in the MAA by adopting the SD cubic anvil and we also discuss the problems and perspectives of this technique.

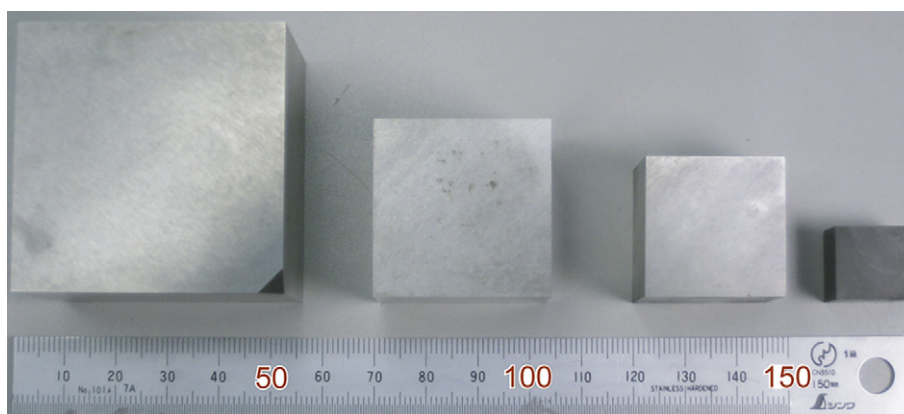
## 2. Properties of the sintered diamond anvil

We have used a 14-mm SD cubic anvil together with WC anvils of various sizes (Fig. 1). The basic procedure for the synthesis of SD, which is usually made of fine diamond powder with cobalt as a binder, was reported by Pope et al. (1972). Some properties of SD have been tested by Horton (1979). The density of SD is larger than pure diamond due to the presence of cobalt. Kondo et al. (1993) reported that the calculated bulk modulus of SD is 410 GPa, which is almost the same as that of an SCD (420 GPa), and the estimated compressive strength of SD is more than 12 GPa, twice that of the strongest WC grades. Sung and Sung (1996) reported that the Knoop hardness for SD is 5000  $\text{kg}/\text{mm}^2$ , whereas that for WC is 2400  $\text{kg}/\text{mm}^2$ .

The effect of cobalt binder content on the elastic properties of SD has been investigated by Kono et al. (2010). The results showed that a lower cobalt content implied higher elastic stiffness, which corresponds to a higher efficiency of pressure generation in the MAA as reported by Tange et al. (2008). The surface roughness of the SD anvil also has an effect on the efficiency of pressure generation in the MAA (Goto et al., 2002). A smaller surface roughness displays a higher efficiency, with the effect being large at low pressures and diminishing gradually at higher pressures.

## 3. Pressure generation

The pressure capability of a Kawai-cell assembly (including 8 truncated anvils, an octahedral pressure medium and pyrophyllite gaskets) depends first on the truncation size of the anvil; the smaller the truncation, the higher the potential pressure. The simplest measure of the theoretically attainable pressure is equal to the ram force divided by the area of 4 truncations. However, the attainable pressure is much lower ( $\geq 50$  percent) than the

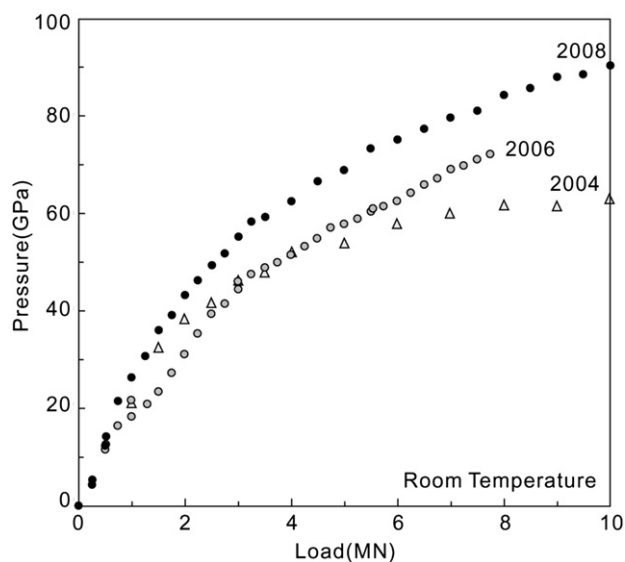


**Figure 1** Photographs of a sintered diamond anvil (14 mm) and tungsten carbide anvils (from left to right: 46 mm, 32 mm and 26 mm).

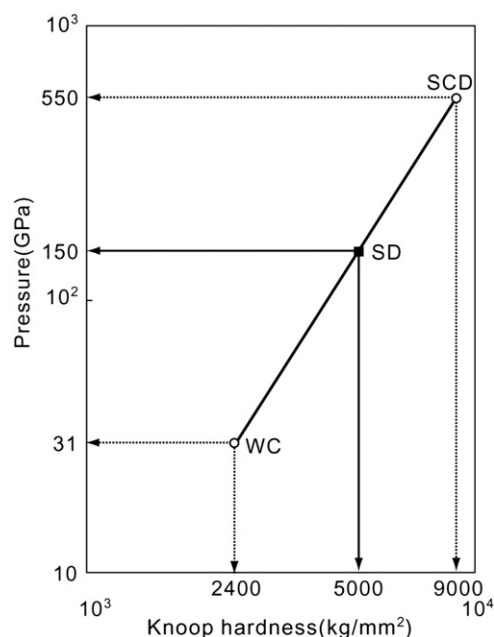
theoretical value because of the gasketing. Putting all the force on the truncations alone would break the anvils, so a pyrophyllite gasket seal is created between anvils. Additionally, the grade or quality of anvil and the design of the assembly and gasket are other factors that affect the attainable pressure.

Since the hardness of an SD anvil is much higher than common WC anvils, the generated pressure in the MAA has largely been extended by its adoption. The first trial to use cubic SD anvils was performed by Ohtani et al. (1989) in the DIA-type cubic-anvil press (Inoue and Asada, 1973) at the High Energy Accelerator Research Organization (KEK), Japan. Ohtani et al. (1989) used 4.85-mm SD cubes with 0.5- and 1.0-mm truncations, and the maximum generated pressure reached 41 GPa based on the Au scale of Jamieson et al. (1982). Adopting 10-mm SD cubes with 2-mm truncation, Kondo et al. (1993) generated a pressure up to 30 GPa by using Fe–V alloys as calibrants. Funamori et al. (1996a,b) used 9.5-mm SD cubes with 2-mm and 1.5-mm truncation to generate pressure up to 30 GPa based on the Au scale of Jamieson et al. (1982) by means of a synchrotron in situ X-ray diffraction method.

Since the late 1990s, a large-sized SD cubic anvil of 14-mm edge length has been available. So far the 14-mm SD cube has commonly been used as second-stage anvils in the MAA. Using this type of SD anvil with 3-mm truncations, Ito et al. (1998) generated pressure reaching 37 GPa. In addition, by means of the quenching method, some phase equilibrium and melting experimental studies have been done (Ito et al., 1998, 2004; Ono et al., 2001). It is the best way to determine the generated pressure based on some equations of state for selected pressure calibrants, e.g., Au, Pt, MgO, combined with synchrotron in situ X-ray diffraction. These studies were carried out using the DIA-type MAA, SPEED-1500 (Utsumi et al., 1998) and SPEED-MkII (Katsura et al., 2004), installed at a bending magnet beamline BL04B1 of the Spring-8 synchrotron radiation facility in Japan. The generated pressures reached 63 GPa and 72 GPa in 2004 and 2006, respectively. Tange et al. (2008) reported a generated pressure up to 80 GPa based on the Au scale of Tsuchiya (2003). Details of the experimental procedure are described elsewhere (Ito et al., 2005; Ito, 2007). Quite recently, Ito et al. (2010) reported a new record up to 90.4 GPa, which was



**Figure 2** Pressure generation using 14-mm sintered diamond cubes in the multianvil apparatus (modified from Ito et al., 2010).

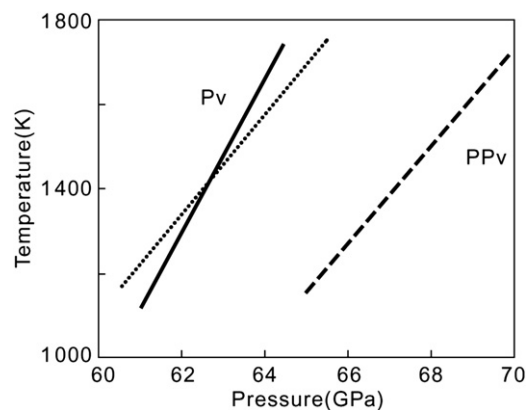


**Figure 3** Relationship between Knoop hardness and maximum pressure (data points of hardness from Sung and Sung (1996)). WC: tungsten carbide; SD: sintered diamond; SCD: single crystal diamond.

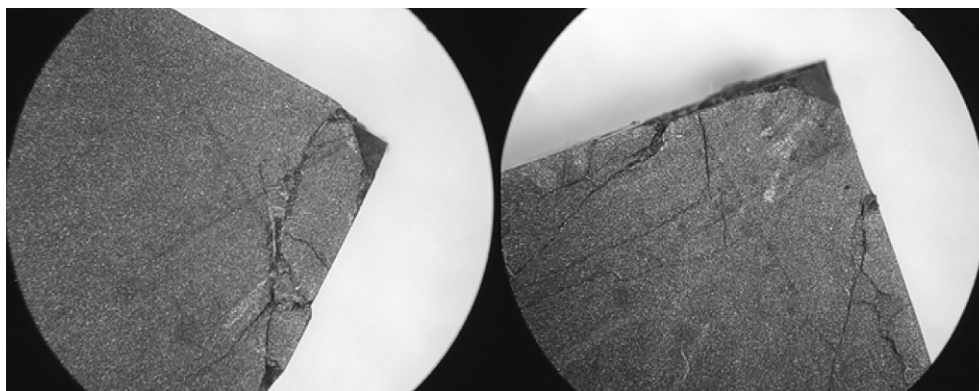
reached in 2008, based on the Au scale of Anderson et al. (1989) as shown in Fig. 2. It should be noted that the generated pressure is read as 95.5 GPa based on the Au scale of Tsuchiya (2003), just short of 100 GPa. Ideally, based on the Knoop hardness of SD (Sung and Sung, 1996), it is theoretically possible to reach 150 GPa (Fig. 3).

#### 4. Problems and prospects

By measuring the volumes of pressure markers, the generated pressure is precisely determined through the equations of state. Many pressure markers can be used. However, it should be pointed out that one pressure marker can give different pressure values according to different scales. As described by Ito et al. (2010), adopting the pressure scales of Au reported by Anderson et al.



**Figure 4** Phase boundary between perovskite (Pv) and post-perovskite (PPv) in MgGeO<sub>3</sub> determined by using different pressure scales of Au. The solid, dot and dashed lines represent the boundaries determined by Au scales of Anderson et al. (1989), Shim et al. (2002) and Tsuchiya (2003).



**Figure 5** Photos of subsidence that occurred close to the truncation of the sintered diamond anvils.

(1989), Shim et al. (2002) and Tsuchiya (2003), the generated highest pressure can vary within 5% over pressures of 60–70 GPa. This is clearly shown in the determination of the perovskite to post-perovskite phase boundary of  $\text{MgGeO}_3$ , as shown in Fig. 4. Therefore, one urgent task is to establish reliable pressure scales.

During compression of the cell assembly of the SD anvils, blowout can sometimes happen and the experiment is immediately terminated. Compared with the WC assembly, the SD assembly blows out much more frequently. The blowout may be caused by a large pressure gradient through the pressure medium and gaskets. Thus, preheating on compression might be an efficient way to prevent blowout.

The quality of the SD anvil is another issue in high-pressure generation. In some experiments, such as M492 mentioned by Ito et al. (2010), pressure drastically dropped and suddenly blew out during a further increased load. Then two recovered anvils showed that serious subsidence occurred at the top portion close to the truncation, as illustrated in Fig. 5. In this case, the subsidence caused the pressure drop and the blowout. Therefore, it is important to improve the quality of the SD anvil to generate higher pressure. Also, a larger high-quality SD cube is necessary for a larger sample volume.

Compared with the WC anvil, the SD anvil is expensive (about \$2000 per cube for a 14-mm cube). This cost is therefore an obstacle to the widespread usage of SD anvils in the MAA, and necessitates looking for a cheaper way to produce SD anvils.

By adopting SD as the second-stage anvils, many studies in Earth Science can be carried out — as follows:

- (1) Phase relation is a traditional topic in Earth science. A few minerals have been investigated, but many minerals have not been examined in the deep mantle by using SD anvils in MAA, and the information is essential for constitution of deep mantle. The quenching method and in situ X-ray diffraction experiments can yield detailed phase relations of single-mineral and many-mineral systems;
- (2) Physical and chemical properties of minerals are important for understanding the evolution and dynamics of Earth. There still a significant lack of knowledge of some physical and chemical properties of mantle minerals, which limits precise modeling of the deep mantle;
- (3) Melting experiments of mantle minerals are fundamental for magma ocean differentiation. Liquidus phase relations and element partitioning of mantle minerals have not been well examined, but can be done by using SD in MAA.

## 5. Conclusions

Pressures generated in a multianvil apparatus (MAA) have been significantly improved by using the SD anvil in the last decade. This technical development makes it possible to investigate the deeper regions of the Earth's interior much more quantitatively than before. Studies have been done on phase transitions, physical properties, and melting experiments of a few important minerals, but many experimental issues are still unresolved. An improved knowledge of minerals under deeper mantle conditions is essential to an understanding of the structure, dynamics and evolution of the Earth. Therefore, use of the SD anvil for high-pressure experimental studies will be widely increased in the future.

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