Note: An anvil-preformed gasket system to extend the pressure range for large volume cubic presses

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An anvil-preformed gasket system has been developed to extend the pressure range for the widely used large volume cubic press without sacrificing the sample volume. The relationship of the sample chamber pressure versus press load for this system was calibrated at room temperature using transitions in Bi, Tl, and Ba. With similar sample volumes (8–11 mm in diameter and 8 mm in length), the anvil-preformed gasket system can generate pressures up to about 8.5 GPa, significantly higher than 6 GPa, which was generally the maximum pressure for the conventional anvil-gasket system. The details on the optimized design for the anvil-preformed gasket system are given in this note. © *2010 American Institute of Physics.* [doi:10.1063/1.3488606]

Over the past 50 years, large volume cubic presses have been widely used in the field of diamond industry to generate pressures up to about 6 GPa with the tungsten carbide (WC) anvil-gasket system.[1,2] In 2008, the production of industrial diamond amounted to about 5.0×10^9 carats (1000 tons) in China alone, and the main equipment used was the large volume cubic press.[3] In order to meet the needs of scientific research, the pressure range of the cubic press has been extended to over 10 GPa by decreasing the anvil truncation size.[4] Using anvils with the truncation of 3 mm, for example, a sample pressure of 13 GPa was reached.[5] By replacing the WC with sintered diamond, the pressure range could be extended to over 18 GPa.[5–7] Recently, the maximum pressure generated in the cubic anvil (DIA-type) apparatus has been extended up to about 25 GPa by optimizing the pressure medium and by using harder WC anvils (the truncated edge length is 2.5 mm).[8] With such small anvil truncations, however, the sample volume at these conditions is very small, and the temperature gradient is very large. The small sample size makes a number of physical property measurements very challenging, such as thermal diffusivity, electrical conductivity, and elastic properties using ultrasonic method.[9] In addition, industrial applications require large assembly volume for synthesizing bulk materials.

In the large volume cubic press system, the sintered WC is used as anvil material, and the pyrophyllite is typically used as the gasket material. When the anvil face is larger than $23.5\times23.5\ \text{mm}^2$ (the corresponding sample chamber is of 8–11 mm in diameter and 8 mm in length), it is often found that the maximum pressures for the anvil-gasket system are about 6 GPa. This is because the relationship between the cell pressure and the press load departs from linearity as the cell pressures advance beyond 4 GPa.[2] A finite element calculation showed that the value of the von Mises stress along the bevel of the anvil reaches about 11 GPa when the cell pressure is 5.6 GPa.[10] This means that the loading force is largely consumed by compressing the region around the gaskets so that the working hydraulic oil pressure has to be very high in order to obtain higher cell pressure. This will increase the risk of breaking the anvils and will eventually lead to a high cost of industrial products. Here, we describe an anvil-preformed gasket system that has been designed with the aim of providing significantly higher maximum pressures than those of the conventional anvil-gasket systems but with similar sample volumes. Our experiments show that this system can reach a cell pressure of about 8.5 GPa under an applied load of 8 MN.

The conventional anvil-gasket system consists of six WC anvils and one pyrophyllite cube, as shown in Fig. 1(a). The six WC anvils are mounted on six pistons that are pushed by six hydraulic rams. In the cubic press, six anvils define a center cubic cavity, in which the pyrophyllite cube is compressed. To operate the cubic press, the six anvils are simultaneously forced by six hydraulic rams. The motion of the six anvils compresses the gaskets and the cube so that the chamber pressure continues to build up in the pyrophyllite cube with a decrease in the volume of the pyrophyllite cube and the thickness of the gaskets.

In our system, called the anvil-preformed gasket system here, a pyrophyllite block with 12 preformed gaskets is used, as shown in Fig. 1(b). The 12 preformed gaskets were machined from the edge part of the six faces of the pyrophyllite cube, each preformed gasket has a 41° bevel whose width is

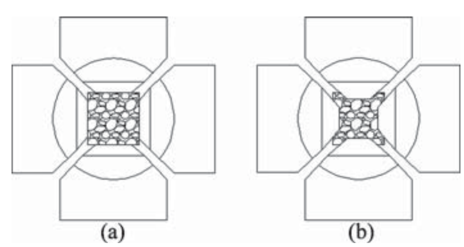

FIG. 1. (a) Anvil-gasket system. (b) Anvil-preformed gasket system.

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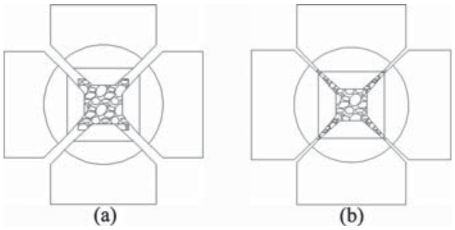

FIG. 2. Gaskets at the beginning (a) and at the end (b) of the pressurizing stroke.

6 mm. In the anvil-gasket system, self-formed gaskets are produced by the extrusion of the pressure medium during the compression process, so the symmetrically deformed region under high pressure is much smaller than the pyrophyllite cube. Using 23.5 mm truncation anvils and a pyrophyllite cube with edge length of 32.5 mm, for example, the symmetrically deformed region is about $18\times18\times18$ mm^3. In the anvil-preformed gasket system, upon applying force to the anvils, the preformed gasket thins and flows away from the center along the anvil's bevels (Fig. 2); the symmetrically deformed size/anvil truncation size can be larger in this system. Table I lists the symmetrically deformed region for the anvil-preformed gasket system and anvil-gasket system with different anvil truncations under 4 GPa. The size of the symmetrically deformed region could be shown by a simple demonstrational experiment: (1) divide a pressure medium into two parts and draw a square mesh on its inner surface. (2) Compress the pair of half pressure medium and check the variation of the mesh in the recovered medium. It can be seen from Table I that the symmetrically deformed region in the pressure medium under high pressure is larger for the anvil-preformed gasket system than that for the anvil-gasket system. Figure 3 is a schematic diagram of the sample assembly for the anvil-preformed gasket system, in which the diameter of the sample chamber can be in the range of 8–11 mm.

Table II lists the relationship of the cell pressure versus press load for the anvil-preformed gasket system (14.5 mm truncation anvils) and anvil-gasket system (23.5 mm truncation anvils) with similar sample volumes.[11] It can be seen from Table II that with the anvil-preformed gasket system, the Bi III–V phase transition has been observed at a press load of 6.5 MN, which has not been observed with the anvil-gasket system even at the press load of 9 MN. Figure 4 shows the pressure calibration curves for the anvil-preformed gasket system and anvil-gasket system with different anvils. It appears that the cell pressure versus press load curve reaches a plateau at about 5.5 GPa for the anvil-gasket system when 23.5 mm truncation anvils are used. The plateau most likely occurs at loads where both extrusion and compressibility of the gaskets are significantly reduced and a large proportion of the additional load becomes supported by the gaskets.[9] Using the anvil-preformed gasket system (14.5 mm truncation anvils) with a similar symmetrically deformed region, a higher pressure-generating efficiency has been observed in this study. With a moderate extrapolation of the cell pressure-press load relationship established here, the cubic press is expected to achieve a pressure of 8.5 GPa at the press load of about 8 MN. For the purpose of comparison, we tested the anvil-gasket system using the same truncation anvil (14.5 mm truncation anvils). The pressure generation efficiency at this condition was found to be better than that with the anvil-preformed gasket system; nevertheless, the six anvils broke at a press load of 5.2 MN (Bi III–V phase transition was not observed) and the symmetrically deformed region is much smaller. Anvil break, which limits the maximum pressure for the anvil-gasket system, may be due to reduction in the area of the gasket. A reduction in the area of the gasket increases the average pressure at the anvil bevels. The open triangle in Fig. 4 is an estimate pressure value for the anvil break. The curves in Fig. 4 show that the anvil-preformed gasket system is an excellent method to improve the maximum pressure in the cubic press without reducing the sample volumes.

In the present study, with similar sample volumes (8–11 mm in diameter and 8 mm in length), the maximum achievable pressure for the large volume cubic press was extended to about 8.5 GPa by modifying the geometry of the pyrophyllite cube in the pressure cell. On the basis of the relationship $P=F/S$ (F is the applied force that allocated to the anvil face, S is the area of the anvil face, and P is the pressure on the faces of the pyrophyllite cube), for a given

TABLE I. Symmetrically deformed region for the anvil-preformed gasket system and the anvil-gasket system with different anvil truncations under 4 GPa.

System	Edge length of the anvil (mm)	Edge length of the pyrophyllite (mm)	Symmetrically deformed region (mm^3)
Anvil-gasket system	23.5	32.5	$18\times18\times18$
Anvil-gasket system	14.5	24.5	$11\times11\times11$
Anvil-preformed gasket system	14.5	32.5	$17\times17\times17$

TABLE II. Pressure calibration for the anvil-preformed gasket system (14.5 mm truncation anvils) and the anvil-gasket system (23.5 mm truncation anvils).

Calibrated material	Phase change type	Sample chamber pressure (GPa)	Press load (MN)	
			Anvil-preformed gasket system	Anvil-gasket system
Bi	I–II	2.55	1.2	1.5
Tl	II–III	3.67	2.0	2.6
Ba	I–II	5.5	3.5	5.3
Bi	III–V	7.7	6.5	⋯

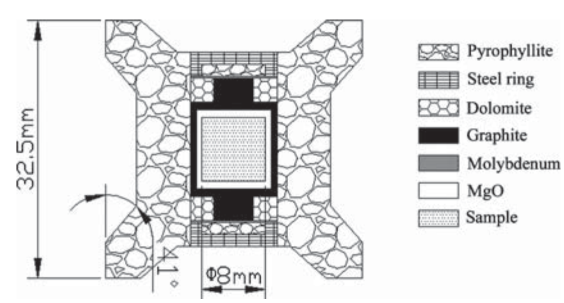

FIG. 3. Cell assembly used in the anvil-preformed gasket system.

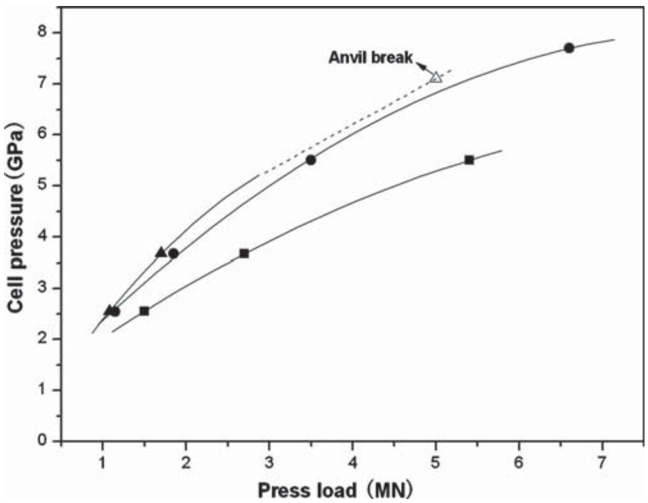

FIG. 4. Relation of the cell pressure vs. the press load. The line (-●-) represents the values obtained using the anvil-preformed gasket system (14.5 mm truncation anvils). The line (-■-) represents the values obtained using the anvil-gasket system (23.5 mm truncation anvils). The line (-▲-) represents the values obtained using the anvil-gasket system (14.5 mm truncation anvils).

sample volume, the anvil-preformed gasket system has a smaller anvil truncation in comparison with the anvil-gasket system. Thus, the anvil-preformed gasket system is able to achieve higher sample pressures than the conventional anvil-gasket system under the same press load. The six anvils inserted into the pyrophyllite cube replace some relatively soft pyrophyllite material with hard tungsten carbide, which automatically leads to a higher pressure for the same load since the resulting compound bulk modulus of the cell is higher ($dp=-BdV/V$).[12]

Some materials, having some special properties sintered under a static pressure of about 8.0 GPa, such as the translucent cubic boron nitride composites, have been synthesized at a pressure of about 7.7 GPa, and nanopolycrystalline diamond with superhardness and greatly enhanced fracture toughness has been synthesized at a pressure of about 8 GPa.[13–15] The anvil-preformed gasket system is being used at present to synthesize bulk superhard material under a high pressure of about 8.5 GPa (at a press load of 8 MN) in our laboratory, such as nanopolycrystalline diamond composites and B_6O-TiB_2 composites.

In this work, we designed and tested an anvil-preformed gasket system for the large volume cubic press. The relationship of the sample chamber pressure versus press load was established at room temperature. The data show that the anvil-preformed gasket system can generate pressures up to about 8.5 GPa while maintaining the sample volume, significantly higher than 6 GPa, which was generally the maximum pressure for the conventional anvil-gasket system.

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