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# High-pressure generation by a multiple anvil system with sintered diamond anvils

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High-pressure experiments using a multiple anvil high-pressure system with sintered diamond anvils are presented and discussed. Pressures in excess of 41 GPa were obtained, on the basis of the lattice constants of gold determined by the *in situ* x-ray diffraction technique using synchrotron radiation.

## INTRODUCTION

Among multiple anvil high-pressure apparatus, an octahedral assembly with eight anvils (MA8) which was originally designed by Kawai and Endo[1] is a convenient device for producing static pressures of $\sim 20$ GPa on a relatively large volume. Extensive work on phase equilibria and material synthesis mainly relevant to geophysical investigation has been carried out by using this apparatus.[2] These studies have been made using apparatus with tungsten carbide anvils. The strength of the tungsten carbide limits the pressure generated by the apparatus.[3] In order to increase the pressure limit, it is essential to use harder materials and, in this respect, sintered diamond is one of the most promising materials.

Sintered diamond was initially used by Bundy[4] for anvils of the Drickamer-type high-pressure cell, and was found to be suitable for an anvil material. Recently, Utsumi *et al.*[5] combined an opposed anvil device made of sintered diamond composites with a cubic anvil apparatus and sucessfully generated pressures up to 60 GPa as confirmed by x-ray diffraction experiments. Opposed anvil system, however, has a very limited sample volume which makes it very difficult to perform a high-pressure high-temperature experiment. On the other hand, the multiple anvil apparatus (MA8) provides a relatively large volume, and with the use of sintered diamond in MA8 it should be possible to conduct high-temperature experiments at pressures above 20 GPa.

The best way to calibrate the high pressure in this type of apparatus is to use the x-ray diffraction technique. A cubic-type high-pressure apparatus dedicated to synchrotron use (MAX80) has already been installed at the Photon Factory, the National Laboratory for High Energy Physics, and has been used for various kinds of high pressure and temperature experiments.[6] In the present study, an MA8 apparatus with sintered diamond anvils was combined with the MAX80 apparatus in order to expand the pressure range and to conduct x-ray diffraction experiments under higher-pressure conditions. Using the sintered diamond MA8/MAX80 system, we have obtained preliminary results of high-pressure x-ray diffraction experiments at room temperature, which are reported in this article.

## I. TECHNIQUE AND APPARATUS

Figure 1(a) shows the schematic diagram of the high-pressure system used in the present experiments. The system for the high-pressure x-ray study consists of the inner multiple anvil (MA8) and the outer cubic anvil (MA6) assembly. The edge length of the anvil head of the cubic anvil is 12 mm.

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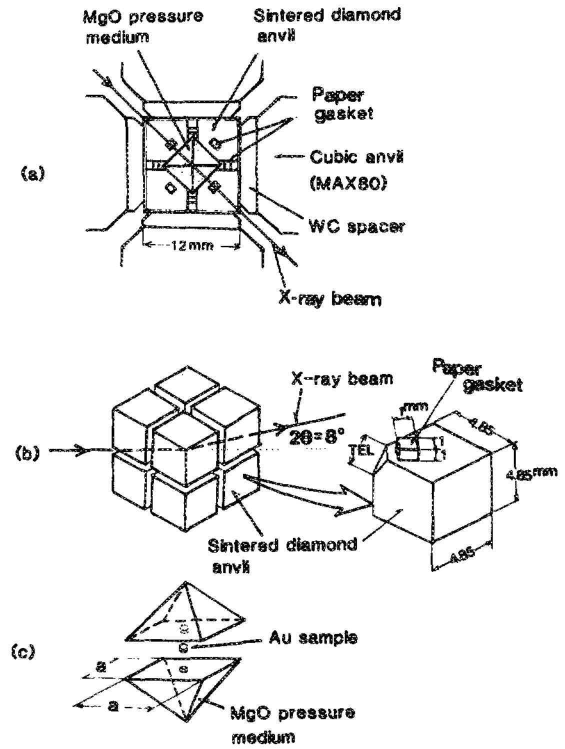


FIG. 1. Schematic diagram of the MA8/MAX80 high-pressure system. (a) Cross section of the MA8/MAX80 system; the size of the MAX80 cubic anvil head is 12 mm. (b) Paths of the incident and diffracted beams pass through the MA8 apparatus; the glancing angle $2\theta$ of the diffracted beam is 8°; the size of the sintered diamond anvil is 4.85 mm, and the truncated edge lengths (TEL) used in this experiment are 1.0 and 0.5 mm. (c) A schematic figure of the sample assembly; the edge length of the octahedral pressure medium, $a$, is 3.4 mm for the anvil with TEL = 1.0 mm, and $a$ = 1.5 mm for TEL = 0.5 mm.

Tungsten carbide spacers with 4 mm thickness are placed between the first (MA6) and the second (MA8) stages. Sintered diamonds are used for the anvils of the MA8 system of the second stage. Various materials such as cobalt metal have been used as a catalyst for diamond sintering. Silicon carbide, SiC, is also used as a binder in some composites. Silicon carbide has a small mean atomic weight, and the sintered diamond composites with the SiC binder have excellent transparency for the x-ray beam. Incident x-rays can pass through the anvil-gap to the sample; however, the diffracted x-ray must pass through the diamond anvil of MA8 in the present double-stage system as shown in Fig. 1(b). Therefore, we selected the sintered diamond composite containing SiC rather than metals as a binder.

The sintered diamond blocks (SYNDAX L555, De Beers Diamond Co. Ltd.) are polished to form cubes, and their corners are truncated to form the anvil head of the MA8 apparatus. One corner of each cube is truncated to form the anvil heads to accomodate the octahedral pressure medium shown in Fig. 1(c). A side of a sintered diamond cube after polishing has 4.85 mm in length. The truncated edge lengths (TEL) of the anvils used for the present experiments are 0.5 and 1.0 mm.

Pressure attainable for a given press load by the multiple anvil high-pressure apparatus can be controlled mainly by the mechanical properties (e.g., material, porosity) and geometry (size) of the pressure medium and preformed gasket. Since there are few theoretical guidelines to choose the optimum conditions for pressure medium and gasket, we must determine the best conditions by the experiments. Semisintered magnesia, MgO, with porosity of ~30% is used as the pressure medium in the present experiments. The edge length of the octahedron of the magnesia pressure medium is 3.4 mm for the anvil with TEL = 1.0 mm, while it is 1.5 mm for the anvil with TEL = 0.5 mm. The preformed gasket made of cardboard ($1\times1\times1$ mm$^3$) is placed between the sintered diamond anvils [see Fig. 1(a) and (b)]. Since the edge length of the magnesia pressure medium is about three times larger than that of the anvil heads (TEL) of the apparatus, a large part of the pressure medium is extruded into the anvil gaps (see Fig. 5). Thus, the extruded magnesia pressure medium together with the preformed gasket made of cardboard works as the gasket to support anvils.

The powdered sample of gold was filled in a hole (0.5 mm in diameter and 0.5 mm in depth) placed in the center of the pressure medium [Fig. 1(c)]. The pressure generated was determined by measuring the lattice constant of gold and by comparing the data with the equation of state of gold given by Jamieson *et al.*[7] Experiments were performed at the synchrotron radiation laboratory of the Accumulation Ring facility, the National Laboratory for High Energy Physics. X-ray energy up to 150 keV could be used by the synchrotron radiation from a bending magnet with the operating condition of 6.5 GeV and 5–20 mA. The energy dispersive method was employed for the present experiments. Diffraction from the sample was detected by a solid-state detector set with a glancing angle ($2\theta$) of 8°. Typical measuring time to obtain one diffraction pattern was about 1000 s. The experiments were conducted at ambient temperature.

## II. EXPERIMENTAL RESULTS AND DISCUSSION

Figure 2(a) and 2(b) show the x-ray diffraction patterns of the gold sample at various pressures for the anvils

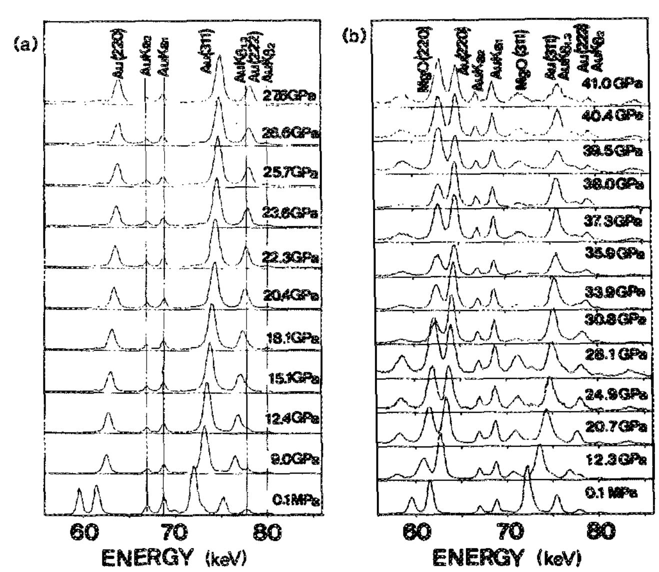


FIG. 2. X-ray diffraction patterns of gold at various pressures. (a) Run No. 2, TEL of the anvil is 1.0 mm. (b) Run No. 3, TEL of the anvil is 0.5 mm.





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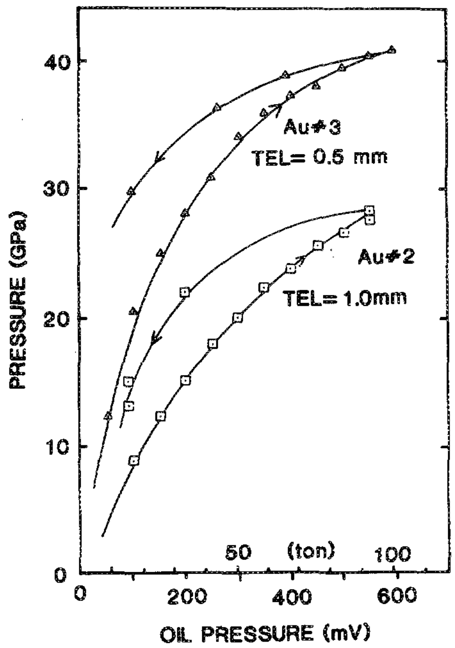



FIG. 3. Relationship between press loads and the generated pressures for the anvils with TEL = 1.0 and 0.5 mm.

with TEL = 1.0 and 0.5 mm, respectively. By utilizing high-energy x ray from the accumulation ring, diffraction peaks are observed between the energy range from 50 to 90 keV. We can precisely determine the lattice constant of gold by referring to the characteristic x-ray peak of gold observed in this energy range. Diffraction peaks only from gold were observed in the experiments using anvils with TEL = 1.0 mm, whereas those from gold and MgO (the pressure medium) were observed in experiments with TEL = 0.5 mm

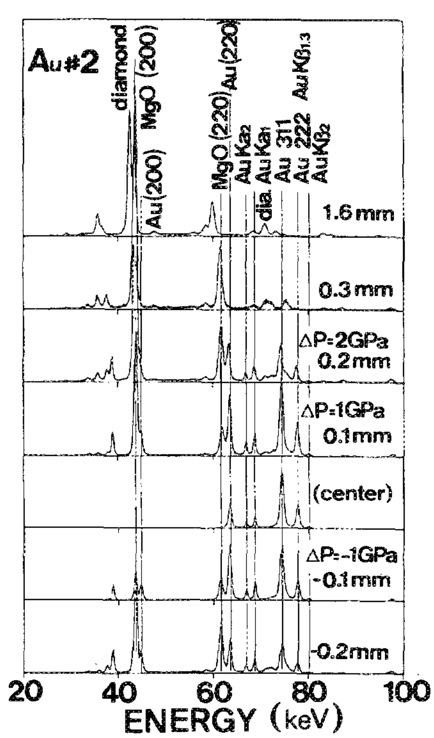
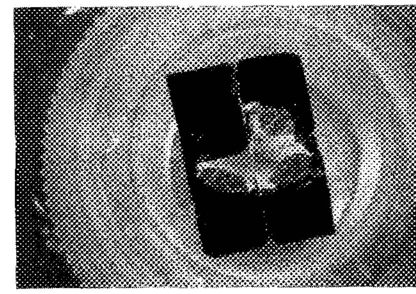



FIG. 4. Change of the diffraction patterns of gold with the beam position at 22 GPa for the anvils with TEL = 1.0 mm (Run No. 2).



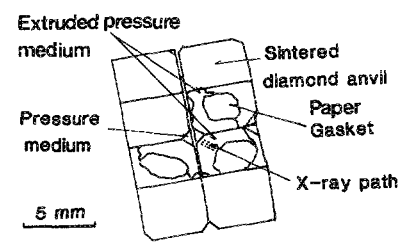



FIG. 5. Photograph of the MA8 apparatus after composition. The path of the intense x-ray beam is observed in the extruded magnesia pressure medium as the dark area of the radiation damage.

anvils because of the small sample volume. The relationship between pressure and press loads for the anvils with TEL = 1.0 and 0.5 mm are shown in Fig. 3. Pressures up to 28 GPa for TEL = 1.0-mm anvil and up to 41 GPa for TEL = 0.5-mm anvil were obtainable in the present experiments. The press loads necessary to produce these pressures were about 100 tons.

The change in the x-ray diffraction pattern at 22 GPa with the beam position to the sample is shown in Fig. 4 for the anvils with TEL = 1.0 mm. When the beam passed through the center of the pressure medium, only the diffraction pattern of gold is observed. When the beam position was shifted from the center, diffraction patterns from pressure medium, MgO, and/or anvil (diamond) are observed.

The sintered diamond anvils scarcely show plastic deformation around the top of the anvil heads, and a very small crack was observed on the surface of one anvil after generating 41 GPa. A photograph of the MA8 apparatus after compression is shown in Fig. 5. The path of the intense x-ray beam of the synchrotron radiation is clearly observed in the extruded pressure medium of magnesia as the dark area of radiation damage in magnesia (see Fig. 5).

Present results indicate that the MA8 apparatus with sintered diamond is suitable for generation of pressures up to 40 GPa. The sintered diamond containing silicon carbide as a binder has a remarkable transparency for the x-ray beam of synchrotron radiation, and is, therefore, suitable as the anvil material for carrying out high-pressure x-ray diffraction studies. The MA8/MAX80 high-pressure system with sintered diamond is now operational and available for *in situ* x-ray diffraction study at room temperature and pressures above 20 GPa. We believe that the present system could be used to conduct x-ray diffraction studies at high temperatures and at pressures above 20 GPa, since the pressure medium has sufficient volume to accommodate an internal heating arrangement.



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