

# Generation of Ultrahigh Pressure by Using Single-crystal Chemical Vapor Deposition Diamond Anvils

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## Introduction

The physical and chemical properties of materials can be radically altered as they are compressed. High-pressure research has been instrumental in the discovery of new phenomena and materials [1, 2]. The only tool for reaching ultrahigh (>100 GPa) static pressures has been the diamond anvil cell (DAC). The DAC generates pressure by applying force to two opposed single-crystal diamonds that are unavoidably damaged or destroyed either during the experiment or after releasing pressure from the megabar range [3-5]. Single-crystal diamonds found in nature or synthesized in high-pressure, high-temperature (HPHT) vessels are severely limited by size, availability, and cost issues. Various solutions have been proposed, such as the replacement of diamond with moissanite [6] or the use of a small replaceable diamond as the pressure-bearing tip on top of a larger diamond as the base (two-segment anvil) [7]. However, the moissanite anvils have so far been limited to below 60 GPa. General usage of the two-segment anvil has not been reported, probably because there are difficulties in aligning the two independent segments [7].

Chemical vapor deposition (CVD) homoepitaxial growth on diamond substrates has emerged as an extremely promising method for rapidly growing low-cost, large, single-crystal diamonds [8]. We used the CVD method to grow a large base onto a small diamond to make a single, regular-sized diamond anvil. We demonstrated that such anvils are comparable to high-quality natural and HPHT synthetic diamonds in reaching ultrahigh pressures of nearly 200 GPa. Since the pressures are significantly higher than the calibrated range of the commonly used ruby fluorescence scale, we resorted to the primary pressure standards based on the equations of state (EOSs) and densities of Ta and Re determined by synchrotron x-ray diffraction measurements.

## Methods and Materials

Single-crystal CVD diamonds were grown according to the method described in Ref. 7 on 1-mm-thick HPHT synthetic type Ib diamond substrates oriented with a {100} top surface to a final thickness of 2.5 mm. With the recently greatly accelerated growth rate of 100 mm/h [8], such plates could be produced in a single day. The plates were then cut into the commonly used size

and shape of a diamond anvil; the height of the anvils is 2.3 mm and the weight is 0.26 carat. The newly grown CVD side faces the anvil table, and the synthetic HPHT diamond side faces the beveled culet (Fig. 1). The volume of the CVD portion is approximately 90% of the diamond anvil, thus significantly enlarging the HPHT diamond, which would otherwise be too small for ultrahigh-pressure experiments.

An experiment was conducted in a DAC with a CVD anvil pressing against a natural diamond anvil. The diameter of the outer beveled culet (B in Fig. 1) for both diamonds was 300  $\mu\text{m}$ , and A- $\theta$  of the CVD was 100  $\mu\text{m}$ , 10° and of the natural diamond was 40  $\mu\text{m}$ , 9°. The CVD diamond was mounted on the piston side of a piston-cylinder DAC, and the natural diamond was mounted on its cylinder side [9]. A 30- $\mu\text{m}$  hole was drilled in a Re gasket into which the polycrystalline sample of Fe alloy with 8-wt% Si was loaded. A piece of Pt that was 5  $\mu\text{m}$  in size was added as an internal pressure standard. A lever arm apparatus was used to increase pressure [10]. Monochromatic synchrotron radiation  $\lambda$  of 0.4246  $\text{\AA}$  at beamline station 16-ID-B at the APS was used for angle-dispersive x-ray diffraction (ADX) measurements. The incoming x-ray was focused by a pair of bimorph Kirkpatrick-Baez (K-B) mirrors to  $10 \times 16 \mu\text{m}$  full width at half-maximum (FWHM) [11] and directed through the diamond axis; the diffracted beam was collected with a MAR charge-coupled device (CCD) detector (exposure time 30 seconds); and the powder diffraction images were processed by using the FIT2D software [12].

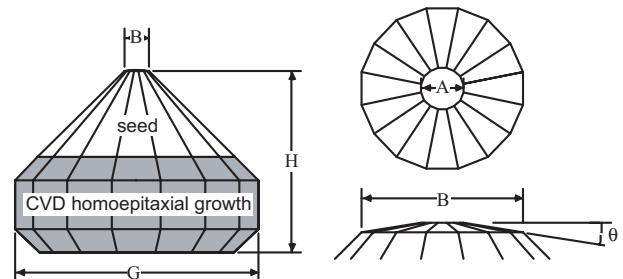


FIG. 1. Schematic of CVD diamond. CVD diamond grown on the HPHT diamond seed. A and B represent the central flat anvil diameter and outer beveled culet diameter, respectively.  $\theta$  is the bevel angle. G is the base diameter, and H is the diamond height. © 2003 by The American Institute of Physics.

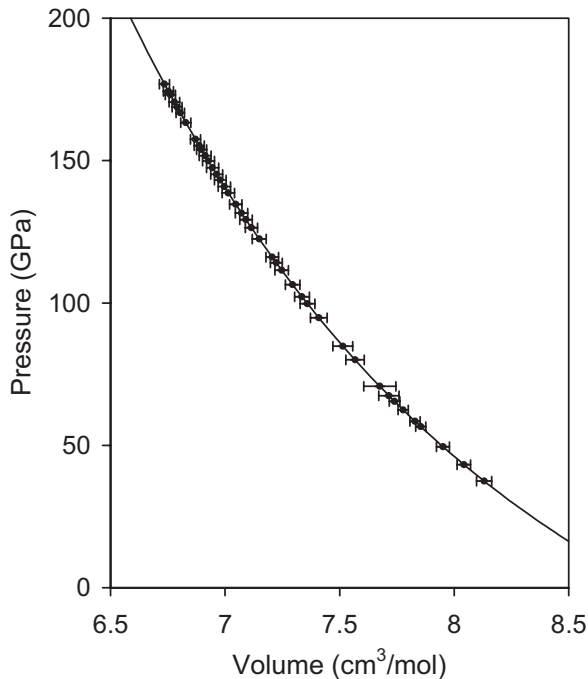


FIG. 2. Pressure versus volume plot for ultrahigh-pressure region of Re data. EOS curve from Refs. 4 and 15 is shown through the data. © 2003 by The American Institute of Physics.

## Results

The Fe with 8-wt% Si is bcc at ambient pressure and hcp above 40 GPa; it has not been studied above 100 GPa [13, 14]. We determined lattice parameters for the hcp Fe with 8-wt% Si from the  $d$ -spacings of three diffraction lines — 100, 101 and 102 — as a function of pressure. Because there was overlap of Fe with 8-wt% Si and Pt, which could introduce uncertainty in pressures, this report gives only the pressure in the Re gasket, which is slightly lower than the peak pressure at the center of the sample. Nevertheless, when the known EOS for Refs. 4 and 15 was used, the highest pressure reached was 177 GPa (Fig. 2). The lattice parameters for hcp Re were determined from four lines: 100, 002, 101, and 102.

## Discussion

The experiment terminated when the CVD diamond shattered at the maximum pressure. The opposing natural diamond, with a much smaller culet ( $B = 40 \mu\text{m}$ ), acted as an indenter and survived. The maximum pressure of 177 GPa reached by the CVD anvil is equal to that of natural diamond anvils of the same culet configurations ( $100 \mu\text{m}$ ,  $10^\circ$ ) within statistical errors. We demonstrated that single-crystal CVD diamonds can be used as anvils in DAC experiments to ultrahigh pressures of 200 GPa. The ability to grow large, high-quality CVD diamonds offers the possibility of

conducting ultrahigh-pressure research with larger sample volumes, and it enables the use of a full range of mainstream analytical techniques (e.g., neutron diffraction and spectroscopy, nuclear magnetic resonance, ultrasonic interferometry) that are currently unavailable because of the minuscule sample size at ultrahigh pressures.

## Acknowledgments

We thank M. Somayazulu and Y. Ding for assistance with the Fe with 8-wt% Si diffraction experiment. Support was provided by the National Science Foundation, Division of Earth Sciences (NSF-EAR); by the U.S. Department of Energy (DOE), Office of Science, Office of Basic Energy Sciences (BES) for the HP-CAT beamline at the APS at Argonne National Laboratory and for the National Synchrotron Light Source (NSLS) at Brookhaven National Laboratory; by the DOE National Nuclear Security Administration (NNSA) for the Carnegie/DOE Alliance Center; and by the W.M. Keck Foundation. Use of the APS was supported by DOE BES under Contract No. W-31-109-ENG-38. This work forms a portion of a manuscript that has been published in *Applied Physics Letters* **83**, 5190 (2003).

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