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# New diamond cell for single-crystal x-ray diffraction

Reinhard Boehler
*Max-Planck Institut für Chemie, Postfach 3060, D-55020 Mainz, Germany*

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A new design for a high-precision diamond cell is described. Two kinematically mounted steel disks are elastically deflected to generate pressure. This principle provides higher precision in the diamond anvil alignment than most sliding piston-cylinder or guide-pin devices at significantly lower cost. With this new diamond cell conical diamond anvils with an x-ray aperture of 85° were successfully tested to over 50 GPa using helium as a pressure medium. Anvil thickness of less than 1.4 mm provides high x-ray transmission and low background, a significant improvement compared to beryllium or diamond-disk backing plates. Because the diamond anvils are supported by tungsten carbide seats, samples and pressure media can be annealed by external or laser heating to provide hydrostatic pressure conditions. © *2006 American Institute of Physics.* [DOI: 10.1063/1.2372734]

## I. INTRODUCTION

There exist a large number of diamond cell designs for a broad range of applications (see Ref. 1). In general, diamond cells for megabar (100 GPa) pressures require costly high-precision machining for accurate diamond anvil alignment. The most commonly used design is a piston-cylinder device with tolerances on the clearance between the piston and the cylinder in the micrometer range (Mao-Bell type).[2] Standard industrial precision of 10–20 $\mu$m is not sufficient for achieving very high pressures and therefore the cost for diamond cells usually exceeds $5000.

For many applications such as single-crystal x-ray diffraction, double-sided laser heating, and IR and Brillouin spectroscopy, short symmetrical diamond cells with large apertures on both sides are needed. Usually, two plates holding the diamonds, guided with sliding pins, are pulled together with screws (Merrill-Bassett type).[3] Misalignment of the diamonds at high pressure is frequent due to slack in the guide pins.

For single-crystal x-ray measurements anvil supports made from beryllium, possessing high x-ray transmission, have been commonly used. However, beryllium has several disadvantages such as low yield strength of less than 0.5 GPa, which does not allow very high pressures due to plastic deformation and loss of anvil alignment. Additionally, beryllium causes strong and broad diffraction rings that often overlap with single-crystal diffraction peaks from the sample.[4] To circumvent this problem, diamond and cubic boron nitride (BN) backing plates have been recently used to replace the beryllium supports.[4,5] This approach significantly increased the pressure range and decreased the background, but several problems associated with these disks have been reported: bad optical transmission using type I diamond, high cost using type II diamond, still strong Bragg reflections and high Compton scattering due to large total diamond thickness, and anvil failure due to stress concentrations at the table of the anvil. The largest aperture using such backing plates has been 70°, and the highest pressures achieved in single-crystal x-ray diffraction measurements have been below 30 GPa.

In this study we describe a new design for a low-cost, high-precision diamond cell and present a version that is suitable for large-angle (up to 90°), single-crystal x-ray diffraction. The new design circumvents most of the problems associated with high-precision machining and misalignment of the anvils. This cell has no “loose” sliding parts and is both easy to built and to handle. Pressures of 100 GPa have been reached with standard conically supported anvils[6] with 0.3 mm culets without loss of anvil alignment after pressure cycling. Over 50 GPa have been reached with anvils with an aperture of 85° using helium as a pressure medium. Because relatively thin conical diamonds are used, problems associated with x-ray background will be significantly reduced. The anvils are supported by tungsten carbide and, thus, in contrast using beryllium seats, this cell can be heated, externally or by lasers, providing nearly hydrostatic pressure conditions necessary for single-crystal x-ray diffraction.

## II. CELL DESIGN

Figure 1 shows a schematic cross section of the diamond cell. It consists of two kinematically mounted steel plates (A). Parallelism and distance are adjustable with three alignment screws (B), which can be fixed by three setscrews (C). The two plates are elastically deflected in the center with three fine-thread screws (E) to apply pressure between the diamond anvils. One diamond anvil-seat unit (D) is laterally adjustable with three setscrews (F) to center the diamonds (lower plate) and the other is press fitted into the upper plate.

Figure 2 schematically shows a diamond anvil in its tungsten carbide support. The principle has been described earlier[6] and has now become a standard design in our and other laboratories. The conical bottom part and the girdle of the diamond are precision ground to within 1 $\mu$m in diameter and the conical carbide seat is perfectly matched[6] with a cone angle of 60°. A small amount of epoxy is used to fix the anvil in the seat. The culets are polished after the anvils are

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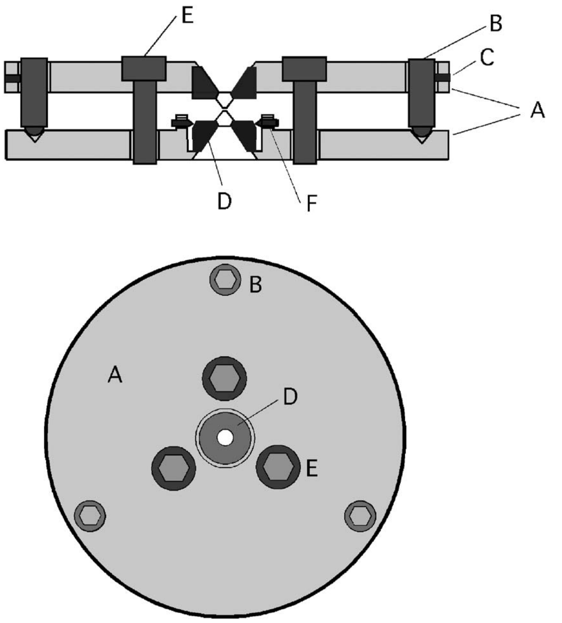

FIG. 1. Schematic drawing of the diamond cell. (A) Maraging steel plates (DIN 2709, HRC 55) with thickness of 5 mm and diameter of 50 mm, (B) alignment screws (kinematic mount) (M6/0.5 mm) fixed by set screws (C) (M3/0.5), (D) tungsten carbide supports for the diamond anvils, (E) screws to bend plates to generate pressure (DIN 2709, HRC 55, fine thread M5/0.35 mm), and (F) set screws for lateral alignment of diamond seat (M2.5).

inserted into their seats providing perfect parallelism with the base of the carbide seat. Table, girdle, and culet have diameters of 2.5, 3.3, and 0.3 mm, respectively, and the girdle height is 0.3 mm. The total height of the anvil is slightly less than 1.4 mm resulting in a facet angle of 15°. This anvil-seat arrangement provides an x-ray aperture of 85°.

## III. ALIGNMENT

The two steel plates are aligned with three adjustment screws (B in Fig. 1) with spherical ends resting in conical holes in the bottom plate. These screws are adjusted to provide a gap between the culets of 100–150 $\mu$m and parallelism of the plates. Accuracy in the gap is not important and can be measured either with a feeler gage or optically with a microscope. Parallelism of the plates can be easily adjusted to within a few thousandths of a degree. The adjustment screws are then fixed with setscrews. The two plates are elastically deflected with three screws made of the same maraging steel as the plates (fine thread M5/0.35 mm) using a gearbox with a gear ratio of 3. The large gear wheels for the three screws (E) in Fig. 1 have a diameter of 16 mm and 30 teeth. The driver rod in the center has a diameter of 5 mm and 11 teeth. When the two diamonds almost touch each other the lateral alignment of the diamonds is achieved with the three internal setscrews. The culets are then brought into contact in order to check their parallelism using the common interference fringe method. Corrections may be made with the adjustment screws, but in most cases this was not necessary.

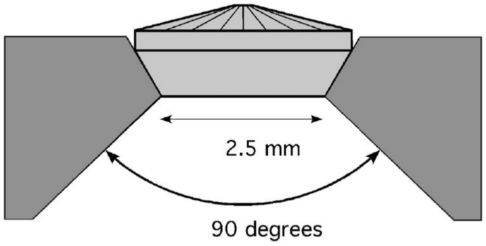

FIG. 2. Large aperture conical diamond anvil and tungsten carbide support. The conical parts of anvil and seat are ground to within 1 $\mu$m in diameter. The optical aperture is 90° and the x-ray aperture is 85°.

There is an additional benefit of this design that is worth mentioning. Due to the initial gap the two diamonds cannot be accidentally smashed together, which often causes diamond failure in any other diamond cell.

## IV. TESTS

A number of tests were performed with varying gasket thicknesses and initial gap between the diamonds. A cell with a "standard" anvil-seat arrangement with a 70° optical aperture[6] was cycled to megabar pressures with a 0.3 mm culet and an $Al_2O_3$ pressure medium. No loss of diamond alignment was detected.

In the first test using an anvil-seat arrangement with 90° optical aperture and 85° x-ray aperture, the gap between the diamonds was set to 100 $\mu$m. A tungsten gasket of 210 $\mu$m thickness was used. In order to test the force requirements, this gasket was preindented to a thickness of 75 $\mu$m with a hydraulic press and a force of 9 kN. The gasket hole with 120 $\mu$m diameter was filled with sodium chloride and several ruby chips. After a pressure cycle to 33 GPa the diamonds were still perfectly aligned.

In a second test using the same anvil-seat unit, the culet size was enlarged to 400 $\mu$m and the initial gap between the diamonds was set to 150 $\mu$m. A tungsten gasket with 210 $\mu$m thickness was preindented to a thickness of 60 $\mu$m using a force of 14 kN. Helium was loaded as a pressure medium using a 0.3 GPa membrane-compressor gas loader. The sample was a single crystal of AlOOH with dimensions of $60\times 60\times 15$ $\mu m^3$. At 15.4 GPa the gasket hole (initially 150 $\mu$m diameter) deformed significantly, but after unloading the crystal was undamaged.

In a subsequent run the 400 $\mu$m culet was beveled to 380 $\mu$m at 7°. The gap was 100 $\mu$m. A 140 $\mu$m tungsten gasket was preindented to 43 $\mu$m using the three M5 screws (see Fig. 1) gearbox. A single crystal of AlOOH with the dimensions $30\times 20\times 10$ $\mu m^3$ was loaded in helium into the 130 $\mu$m diameter gasket hole using the 0.3 GPa gas compressor. The cell was then pressurized to 51.5 GPa. The gasket hole shrunk to 75 $\mu$m in diameter. Both hole and crystal were perfectly centered on the culet and the crystal was intact (see Fig. 3). We believe that this is the highest pressure achieved in a diamond cell with such a large x-ray aperture using such thin anvils. No diamond failure has occurred thus far using this new diamond cell and we therefore do not know the upper limit in pressure and the potential of even larger apertures.

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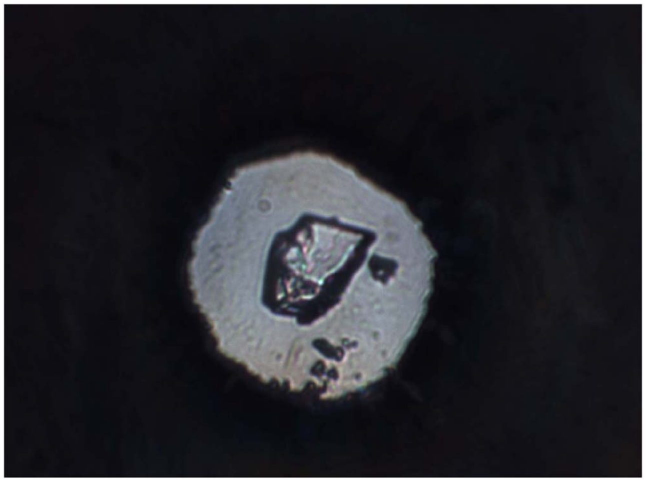

FIG. 3. Picture of an AlOOH crystal with dimensions $30 \times 20 \times 10\ \mu m^3$ in a helium pressure medium at 51.5 GPa.

## V. CONCLUSION

The new design for a diamond cell offers a number of improvements and advantages compared to previous designs. Its simplicity significantly lowers manufacturing costs, especially because it only requires standard industrial precision (for example, tolerances of about 0.01–0.02 mm). The kinematic principle provides the high precision and reproducibility in diamond anvil alignment necessary for megabar pressure application. The cell is easy to handle and to align, and diamond anvils cannot be accidentally broken because they can only be brought into contact by applying the high force necessary to bend the plates. The cell has been successfully tested to over 100 GPa using standard aperture conical diamond anvils and, additionally, to 50 GPa with an x-ray aperture of 85° using very thin diamond anvils and a helium pressure medium. The limits of this cell have not yet been reached.

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