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NEW ANVIL DESIGNS IN DIAMOND-CELLS

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New diamond anvils with conical support are introduced. Compared to conventional anvils the new design offers superior alignment stability, larger aperture, and reduced cost owing to significantly smaller anvil diameters. Except for table and culet, all surfaces are precision ground on a lathe, which lowers cost compared to faceted anvils. The conical design allows for steel supports, which are significantly easier and cheaper to manufacture than tungsten carbide supports. Conical support also prevents seat damage upon diamond failure. An additional new feature of the anvils is the roughened outer portion of the culet, which increases friction between the anvils and the gasket. This increases the height to diameter ratio of the pressure cell and prevents bonding between gasket and diamond, which causes ring cracks during pressure release. This technique replaces complicated diamond coating procedures. The anvils have been extensively tested for culets ranging from 0.1 to 1 mm diameter up to megabar pressures. A new anvil shape with cup-shaped culets to further increase the cell volume and gasket stability is also introduced.

Keywords: Diamond anvils; High pressure; X-ray diffraction

The basic design of diamond anvils in diamond-anvil cells has been essentially unchanged for several decades [1]. However, several applications call for design improvement with regards to cell volume, aperture, absorption, and stability. In conventional diamond cells, the anvils have brilliant or modified brilliant shapes. Tungsten carbide plates with a central conical opening generally support the large flat table of these anvils. Large windows require large table diameters owing to the limited shear strength of the seat material, resulting in anvils typically 3–4 mm in diameter with 1/4 to 1/3 carat weight. Recently, single crystal diamond supports have been employed in various forms [2] allowing for smaller anvils. However, the overall increased thickness of the diamond window plus anvil presents a problem in some applications such as X-ray diffraction, where absorption in the diamond plays a role. Additionally, catastrophic anvil failure may cause costly damage to the diamond support plate.

We present a modified simple design for diamond anvils that offers a number of improvements compared to conventional anvils. The principles of the design are shown in Figure 1 in comparison with a conventional design. Without loss of pressure range and cell volume (culet diameter), the size of conical anvils can be significantly decreased. The conical anvils have diameters of typically 2–2.5 mm, heights of 1.5–2 mm, and weights

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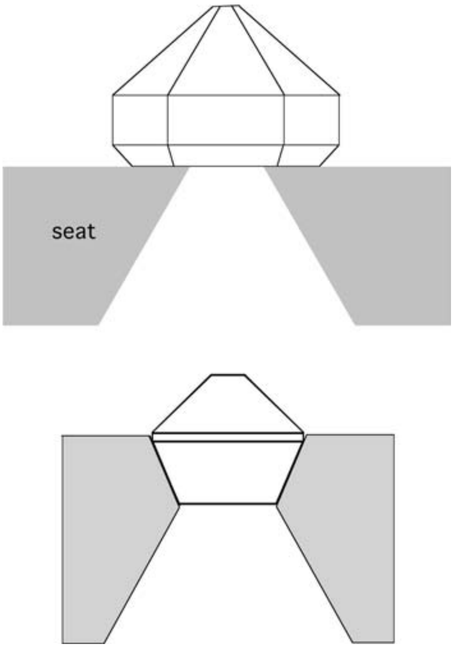

FIGURE 1 Schematic view of conventional (top) and conical (bottom) high-pressure anvils.

of 0.05–0.15 ct. Diameters of the table (window) range from 1.3 to 2.3 mm (see later). The bottom cone included angle is 60° and the top cone angle ranges from 60° to 130°. Bottom and top cone and the girdle are ground (not polished) on a lathe using commercial ceramic bond diamond grinding wheels. Precision in the diameters within 1 μm is routinely obtained. Figure 2 shows three examples which have been tested to pressures up to 1 Mbar with a culet

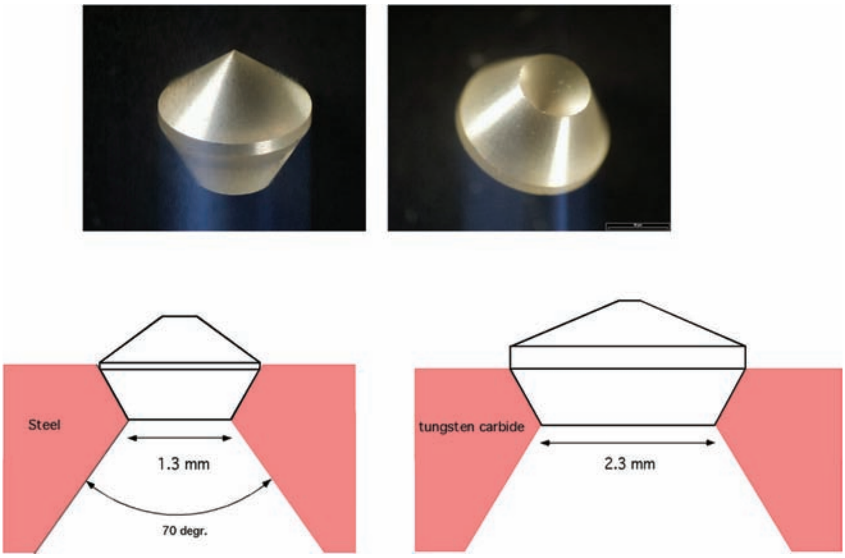

FIGURE 2 Top: photographs of a conical diamond anvil (top left: top view, top right: bottom view). Bottom: cross section of a ‘standard’ anvil (left) and an anvil with very large X-ray aperture (75°).

diameter of 300 μm. Culet and bevel size and bevel angle can be chosen as in any conventional anvil. The seat (support) material is steel ranging from hardnesses HRC 55 (maraging steel, DIN 2709) to HRC 69 (for example high speed steel, Uddeholm VANADIS 60). The steel seats are machined before heat-treatment, and the conical seat is ground in after heat-treatment with a conical polycrystalline diamond (PCD) tool that has exactly the same cone angle as the diamonds. In order to avoid wear of the tool, we use boron carbide powder with 10 μm grid sizes. We successfully tested anvils with diameters slightly <2 mm using tungsten carbide seats. However, the cost reduction of the diamond is insignificant compared to the increased difficulty in machining tungsten carbide. Steel support may also be easily machined to match the requirements for individual diamond cells. Our steel supports have outer diameters ranging from 10 to 17 mm and thicknesses from 5 to 7 mm. The conical opening of the seat is typically 70–75°.

Conical diamond-seat design provides improved alignment stability in diamond cells. In conventional diamond cells, the flat table resting on a flat support (Fig. 1, top) requires either mechanical clamping of the diamond or adhesives (for example epoxy). While mechanical clamping may be complicated, adhesives are often ineffective at both high and low temperatures because both the anvil and seat are polished, thus, requiring frequent alignment of the anvils. We found that with a conical design, the anvils can undergo many load cycles to the maximum loads without intermediate alignment of the anvils. For the anvils and pressure ranges described earlier, the pressures on the seats were below the elastic limit (yield strengths) of the seat materials. In principle, owing to the high precision matching of seat and conical anvils, adhesives are not necessarily required and the diamonds will remain aligned after the experiment. However, we found it more convenient to use a small amount of epoxy adhesive. A high temperature, one-component epoxy (Hysol, Loctite) provided a strong, durable bond even after long laser-heating experiments owing to the surface roughness of both anvil and seat.

An important improvement of the new anvil-seat design is the lack of seat damage upon diamond failure. In a conventional design, diamond failure in most cases (especially at very high loads) results in a complete destruction of the anvil(s). The diamond fragments embed in the seat material and often damage the tungsten carbide support beyond repair. With our new design, after extensive testing of pressure limits and routine work, none of the seats has been damaged yet. The reason is that cracks in the diamond never fully propagate through the entire anvil, but stop where the seat radially supports the diamond.

In order to test the strength of the cones and their supports, we performed a number of runs with large culet diameters (830 and 700 μm). A test of the pressure limits with such large culets using conventional anvil design would require rather large and costly diamonds. The conical anvils had an outer (girdle) diameter of 2.4 mm, 1.8 mm height, and a window (table) diameter of 1.3 mm. The tungsten gaskets had hole diameters of 220 and 200 μm, respectively, an initial thickness of 100 μm, and a final thickness (measured after the run) of 50 μm. The pressure medium in both cases was argon. For these large culets, pressures reached a plateau most likely due to cupping. Further increase of load did not result in a noticeable pressure increase. The pressure limits were 22 and 33 GPa, respectively. The forces on the diamonds in these runs were measured only qualitatively and the force–pressure plots are shown in Figure 3. However, this also illustrates that even with very large diamond anvils, pressures for a given sample volume may be limited, and thus, proposals to reach high pressures with large sample volumes by using larger diamonds in conventional designs should be looked at very critically.

For many applications in the diamond-cell, in particular in laser-heating experiments where the heated sample has to be thermally insulated from the highly conductive diamond, large height-to-diameter ratios of the cell are required [3]. Rare gases and some

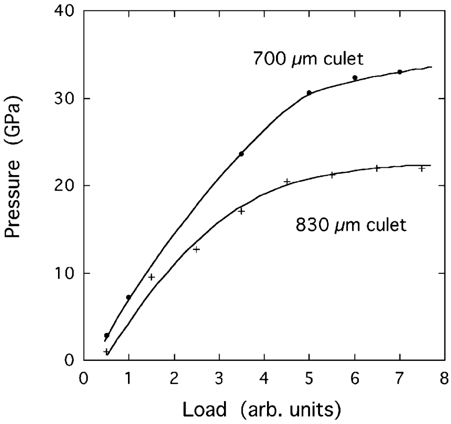

FIGURE 3 Pressure as a function of load for large culets. Forces were not measured absolutely. Pressures reach plateaus at about 22 and 33 GPa.

alkali halides have favorable properties as pressure media with respect to low conductivity and chemical inertness to many samples, but these materials are very compressible and require the use of metallic gaskets. The limited strength of metallic gaskets causes significant radial extrusion and, thus, thinning and asymmetric deformation of the pressure chamber. In the past, we have significantly reduced this extrusion and deformation by coating the gaskets with micron-sized diamond powder. Moreover, this procedure also prevents the often-observed bonding (welding) between the gasket and the polished diamond that may be the main cause for the occurrence of ring cracks upon pressure release. Additionally, thick gaskets reduce stress concentrations at the edges of the culets when cupping occurs at high pressures.

Applying uniform coatings on the gasket, however, is difficult and time consuming. We noticed a similar improvement in the gasket stability when the outer portion of the polished culet was roughened. Roughening can be achieved by mechanical grinding or by ultrasonic drilling. Mechanical roughening is achieved with an unpolished-lapped disc of PCD (name) with a grain size of 2 or 10 μm. The disc is moved in a circular motion at an angle of 2° while the diamond is rotating. The result is shown in Figure 4A. This procedure has proven not to influence the strength (pressure limit) of the anvils. The culet can also be roughened ultra-

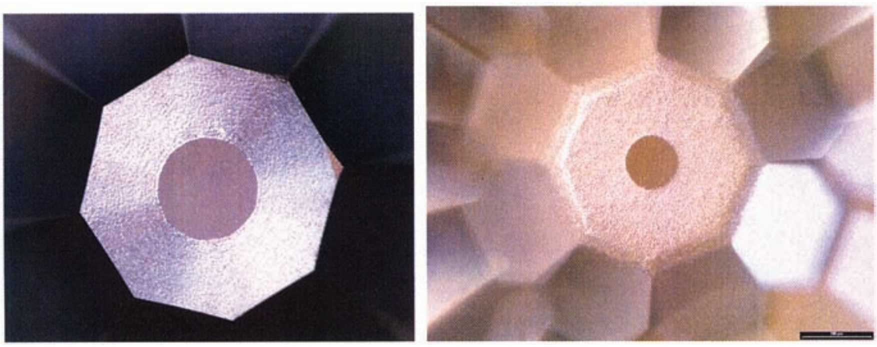

FIGURE 4 Photographs of roughened culets. Left: culet was ground at an angle of 2° with a PCD disk. Right: culet was drilled ultrasonically with a hollow tungsten carbide drill. Drill depth is 2 μm. Outer culet diameters are about 400 μm.

sonically with a hollow drill. We used a modified ultrasonic homogenizer (HD 2070, Bandelin). The result is shown in Figure 4B, where about 2 μm of the polished surface have been removed. The final gasket thickness is a function of initial thickness, pressure, and culet size and pressure medium. We do not provide a quantitative study here, but we routinely measured final gasket thicknesses between 30 and 40 μm for tungsten gaskets and argon pressure medium with outer culet diameters from 400 to 500 μm in the pressure range to 50 GPa.

Volumes of the pressure chamber can be further increased with an anvil shape shown schematically in Figure 5. A shallow hole is ultrasonically drilled in the culet of the 'back' diamond. This allows for flow of the gasket towards the center, partially into the hole. During several test of this design using a variety of dimensions, we noticed a significant improvement of the gasket stability with increased height of the sample chamber. For example, with a culet diameter of 450 μm, a hole diameter of 250 μm, and a hole depth of 25 μm the final gasket thickness at 54 GPa was over 40 μm. Thus, the pressure chamber had a height of over 60 μm with a diameter of 120 μm. The gasket is very stable even during pressure release, but the drilled anvil developed cracks after several pressure cycles. This is most likely due to micro cracks produced by the process of ultrasonic drilling. If the drilling

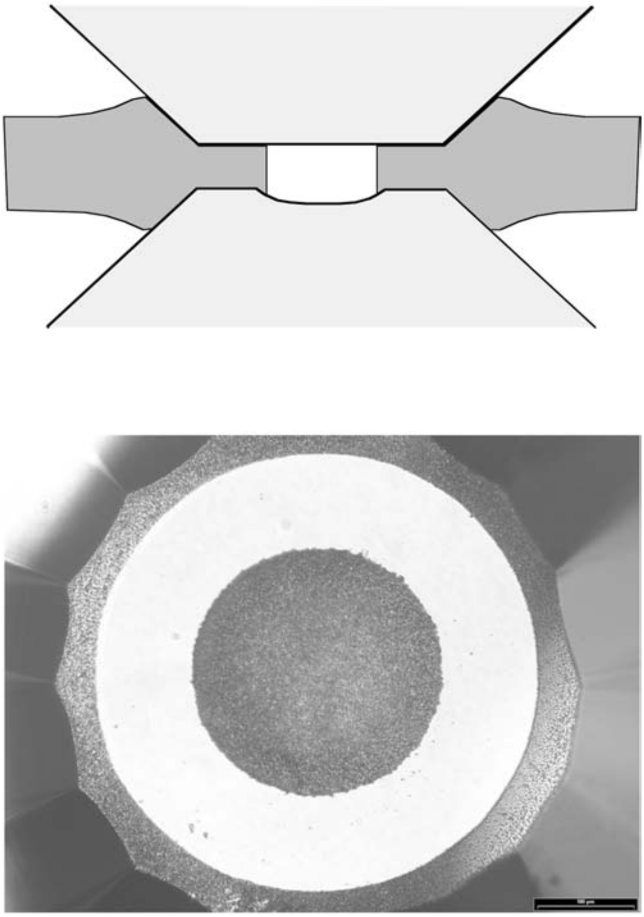

FIGURE 5 New anvil design with a cup-shaped recess in the culet. During compression a portion of the gasket flows towards the center. This design yields higher gasket stability and increased cell height.

process can be improved using other methods, this design looks very promising for routine application.

In summary, we made several improvements on diamond anvils with respect to alignment stability, the volume of the sample chamber and cost reduction on both anvils and anvil supports. Conical design adds stability and reduces cost of both anvils and supports. It allows for significantly larger aperture making such diamond cells suitable to significantly increase the pressure range in single crystal X-ray diffraction with reduced anvil absorption due to reduced anvil thickness. Modified culets result in larger cell volumes and improved gasket stability. These modifications can be made with simple tools.

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