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REVIEW OF SCIENTIFIC INSTRUMENTS **88**, 126106 (2017)

Note: Modified anvil design for improved reliability in DT-Cup experiments

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The Deformation T-Cup (DT-Cup) is a modified 6-8 multi-anvil apparatus capable of controlled strain-rate deformation experiments at pressures greater than 18 GPa. Controlled strain-rate deformation was enabled by replacing two of the eight cubic "second-stage" anvils with hexagonal cross section deformation anvils and modifying the "first-stage" wedges. However, with these modifications approximately two-thirds of experiments end with rupture of the hexagonal anvils. By replacing the hexagonal anvils with cubic anvils and, split, deformation wedge extensions, we restore the massive support to the deformation anvils that were inherent in the original multi-anvil design and prevent deformation anvil failure. With the modified parts, the DT-Cup has an experimental success rate that is similar to that of a standard hydrostatic 6-8 multi-anvil apparatus. https://doi.org/10.1063/1.5005885

The response of materials to differential stress and strain at high pressure and temperature is of great interest in the Earth sciences, physics, and chemistry. Initially high-pressure deformation experiments were performed by repurposing quasi-hydrostatic devices. For example, diamond-anvil cells[1,2] can be used as a low-strain deformation apparatus by bridging the sample between the diamonds.[3,4] The 6-8 multi-anvil has been used as a deformation apparatus by adding hard pistons to the assemblies.[5,6] Both apparatus though are limited to low strains; increases in strain are convolved with increases in pressure and the timing of strain is not readily controlled.

To enable deformation to large strains at constant pressure, multi-anvil apparatus have been modified to include secondary actuators that move some of the anvils independently of the others. The Deformation-DIA (D-DIA) is a modified cubic multi-anvil,[7,8] in which two of the anvils are made independent of the confining load, and load frames with 6 independent rams have been designed for the deformation of samples.[9] However, this style of apparatus is generally limited to between 10 and 15 GPa. Tsujino *et al.*[10] used the 6-8 geometry in a cubic press to deform bridgmanite but only to shear strains of ≈0.08. The Rotational Drickamer Apparatus (RDA)[11] is a rotational opposed anvil device that has been used to deform bridgmanite (Magnesium Silicate perovskite),[12] but it is limited by extremely small samples and large pressure, temperature, and strain gradients in the sample. Rotational diamond-anvils cells[13] have also been developed to deform samples in simple shear, but they have similar limitations to the RDA.

Although the 6-8 multi-anvil is able to generate higher pressures than cubic multi-anvils and on larger samples than opposed anvil devices, it was only recently modified as a deformation apparatus (the Deformation T-Cup or, DT-Cup).[14] In a hydrostatic 6-8 multi-anvil, the sample is contained within an octahedral pressure medium and compressed by 8 second-stage anvils each of which is a corner-truncated cube. The anvils are arranged such that each corner truncation acts on a face of the octahedral pressure medium. The edge-length of the corner truncation is smaller than the edge-length of the octahedron thus enabling the generation of high pressures by compression of the cell. To reduce the likelihood of sudden decompression in the pressure medium (a "blow-out"), pyrophyllite gaskets fill some of the gap between the anvils. The cubic anvils are made from super-hard materials, typically tungsten carbide, cubic boron nitride, or sintered diamond, and are compressed by 6 high strength steel first-stage anvils, commonly called "wedges". The success of the 6-8 multi-anvil apparatus is based on the principle of massive support. The tip and back faces of each cubic anvil are almost entirely in compression.

In the deformation, 6-8 design (the DT-Cup) controlled deformation to high strains is facilitated by replacing the two inner cubic anvils aligned with the compression axis of the load frame with hexagonal cross section anvils [Fig. 1(a)]. These hexagonal pistons are accommodated by removing material from the 1st stage wedges to leave hexagonal-prismatic holes along the compression axis and are backed by secondary actuators to enable deformation. Subsequent use of the DT-Cup has demonstrated that the hexagonal anvils are prone to failure at much lower forces than normal in hydrostatic 6-8 multi-anvils. In testing, two-thirds of experiments ended with the hexagonal anvils failing, and in a few cases, associated dilation of the wedges also prevented heating of the experiment. Here we discuss the design flaw and our solution to it.

In the DT-Cup, the hexagonal anvils fail by splitting vertically, top to bottom [Fig. 1(b)]. There is no warning (audible or otherwise) of imminent anvil rupture. The fractures are similar in every case, nucleating near the edge of the gaskets and propagating, in two or more sections, almost straight down to the base of the anvil. This failure can occur at any point during compression or heating (Fig. 2) but has not been observed during deformation. This is a breakage mode not seen in the hydrostatic 6-8 multi-anvil apparatus but it is the dominant mode of anvil failure in cubic presses.[15,16] To extend the working range of the anvils in cubic presses, they are compressed

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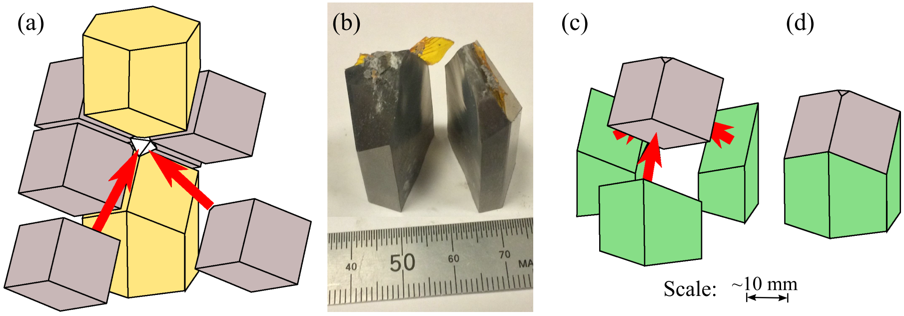

FIG. 1. Deformation anvils in the DT-Cup. (a) Arrangement of the 6 cubic and 2 hexagonal anvils in the DT-Cup, (b) example of broken anvil, and [(c) and (d)] replacement "deformation wedge extensions" with cubic anvil.

laterally by confining rings, but there is no space for anvil confining rings in the DT-Cup.

In addition to the anvil rupturing, a number of experiments would not heat or would self-quench during heating. Experiments are heated by applying electrical power to a cylindrical resistance furnace which connects through the hexagonal anvils. At low loads, the hexagonal anvils are a good fit into the first-stage wedges and they make a circuit through the furnace by contact with the wedges. During compression, dilation of the first-stage wedges breaks the contact between the hexagonal anvil and the wedges preventing heating. Non-heating of experiments was overcome by shorting the hexagonal anvil to the 1st stage wedges; numerous designs of which were trailed and had no discernible effect on the anvil failures. The breaking of the electrical circuit through the hexagonal anvils demonstrates that, although a close fit in the first-stage wedges at no load, the sides of the hexagonal anvil are unsupported at elevated loads.

The removal of massive support from the differential anvils is the cause of the anvil failure; therefore, returning the support will eliminate the problem. To do this, we returned the deformation anvils to their original cube form and designed "*deformation wedge extensions*" to go behind the cube [Fig. 1(c)]. The wedge extensions are made from steel with the same hardness as the wedges. In cross section, each piece is a 60° rhombic prism with an upper face inclined 35.26° to the prism axis. When assembled, the three wedge extensions and the cubic anvil are of the same size and shape as the hexagonal anvil they replace [Figs. 1(a) and 1(d)]. The deformation wedge extensions are held in place by the first-stage wedges, onto which they are pressed by the cubic anvil. All critical parts of the system are therefore in compression, returning the design to one utilising massive support. The pushing of the wedge extensions against the first-stage wedges makes a good electrical connection for the resistance furnace. An additional advantage of the deformation wedge extensions is that they present a significant cost saving over the hexagonal anvils: the wedge extensions are indefinitely reusable and cubic anvils are ~1/5th the cost of hexagonal anvils.

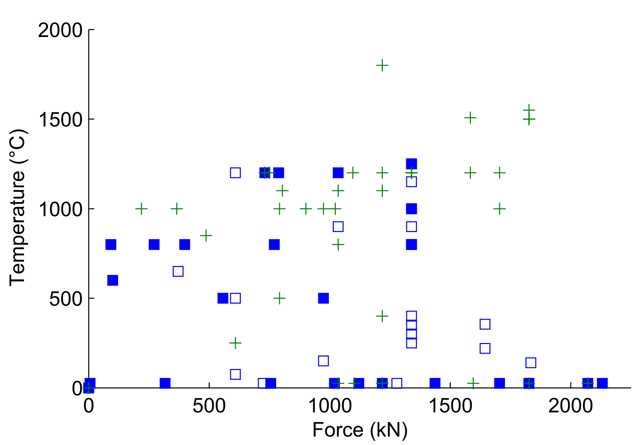

FIG. 2. Maximum load and temperature achieved in DT-Cup experiments with hexagonal anvils (squares) and replacement wedge extensions (+). Filled squares: experiment worked or failed for reasons unrelated to anvils (e.g., thermocouple broke); open squares: experiment ended with anvil failure. N.B. Some symbols represent more than one experiment.

Testing of the new deformation anvils shows that they behave in exactly the same way as a traditional non-deformation 6-8 multi-anvil. With the new design, there have been no experimental failures related to anvil failure even at conditions beyond those investigated with the hexagonal anvils (Fig. 2). With the new design, we have deformed bridgmanite samples to >20% strain at pressures in excess of 24 GPa (end load of 1750 kN), 1500 °C, and strain-rates ~2×10^{-5} s^{-1}. The overall performance and experimental failure rate due to anvil breakage of the apparatus are similar to those of hydrostatic 6-8 multi-anvil devices (e.g., T-Cup).

Finite element analysis (FEA) of the hexagonal anvils and their replacements confirms our understanding of the system's mechanics. Both the hexagonal anvil and the wedge extensions were modelled in ANSYS Mechanical v17.1.[17] The wedge extension model was of a 16 mm tungsten carbide cube with 3 mm corner truncation backed by the three hardened steel wedge extensions. The pieces in the model had a total height of 34.93 mm. The model of the hexagonal anvil was the same shape as the first model but made from a single piece of tungsten carbide. The material properties of the tungsten carbide were the approximate properties of the TJS-01 tungsten carbide from Fujiloy Co., Japan (Young's modulus 6.4×10^{11} Pa; Poisson's ratio 0.21) and those of the steel for hardened steel (Young's modulus 2×10^{11} Pa; Poisson's ratio 0.3). A pressure of 20 GPa was applied to the truncated anvil tips, and gaskets were modelled with 10 GPa and 1 GPa areas extended down the face of the cube to a total distance of 7.84 mm from the edge of the truncation. The basal surface of models was allowed to slip freely. The sides on the hexagonal anvil were unconstrained but the sides of the wedge extensions were constrained by an elastic support and the wedge extension–cube interface was a free slip surface. Comparable results to those presented were obtained in models with fixed basal surfaces and models which ignored the force from gaskets.

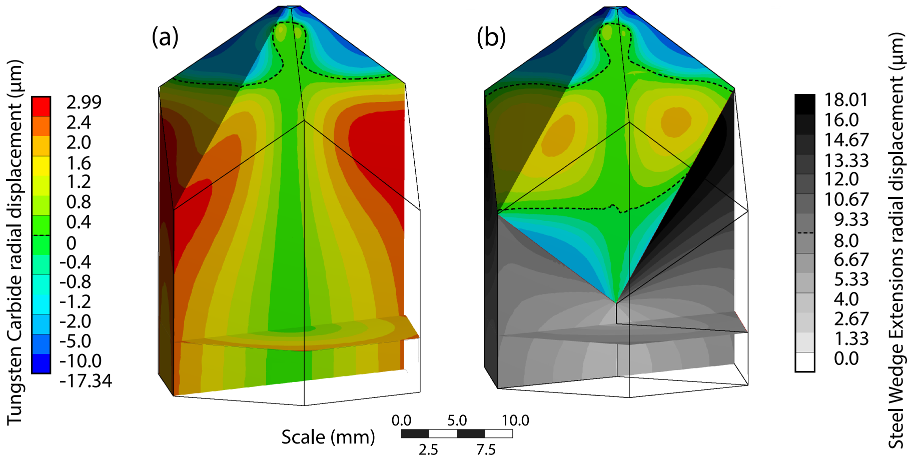

FIG. 3. Radial deformation in (a) hexagonal anvil and (b) replacement cubic anvil and deformation wedge extensions, from finite element analysis. The red-blue colour scale applies to tungsten carbide parts and the grey scale applies to steel components. The dashed line is zero radial displacement iso-surface in the tungsten carbide components. For details of the models, see the text.

The output of the model shows that the maximum compression is at the tip of the anvils (Fig. 3) and that the body of the hexagonal anvil dilates radially under loading [Fig. 3(a)]. Under the conditions of the model, below the gaskets, the entire body of the hexagonal anvil is in radial extension with a maximum radial dilation of ≈3.0 μm. The radial dilation of the body implies that if a crack is initiated in the anvil, it will propagate vertically through the anvil. The tungsten carbide anvil in the replacement design in contrast is under significantly more compression and the maximum radial dilation is 1.8 μm [Fig. 3(b)], although the steel wedge extensions dilate much more. This 1.8 μm is 60% less than that in the hexagonal anvil and only half the vertical profile of the anvil is in radial extension.

The deformation 6-8 multi-anvil (DT-Cup), as reported by Hunt *et al.*,[14] is capable of performing deformation experiments at pressures in excess of 18 GPa but with a subsequently discovered, unacceptably high failure rate. These failures were primarily due to vertical cracking of the unsupported hexagonal anvils. The failure mode of the hexagonal anvils and the FEA demonstrate that the removal of massive support for the hexagonal anvil is the primary cause of failure. By replacing the hexagonal anvils with three deformation wedge extensions and a cubic anvil, we have returned the massive support present in the original 6-8 geometry. Thus the improved design restores the inner geometry to that of the original hydrostatic design while retaining an even extending the ability to deform samples to high strains.

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