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Zen diamond-anvil low-pressure cell

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A diamond-anvil cell can be operated with only one anvil in order to generate modest pressures in relatively large volumes. We demonstrate it to pressures up to 2.5 GPa with gaskets of steel, brass, and other metals, with a sample chamber 0.25 mm in diameter by 0.25–0.9 mm depth, and with various pressure media. In this form the cell is very simple to operate and is useful for much work on biological systems and soft solids which requires pressures in the 1 GPa range. © *2000 American Institute of Physics.* [S0034-6748(00)04210-6]

I. INTRODUCTION

The diamond-anvil cell (DAC) is an opposed-anvil or Bridgman system.[1] It readily generates very high pressures of tens of GPa in a very small sample volume, typically 100 or 200 μm in diameter and only tens of microns thick. However, this is not very useful for much work on biological systems and soft solids, in which interesting transitions happen at relatively modest pressures below 1 GPa. The DAC is quite hard to control at such low pressures, and the small sample space can be a problem, especially if suspensions of relatively large particles are to be studied. For example, in studies of the gelation of starch under pressure, each starch particle can expand to more than 100 μm.[2] Whole live tardigrades have been studied under pressures to 2 GPa;[3] these are animals 100–500 μm long. With the standard opposed anvils, little can be done to enlarge the sample space to take such specimens, since the gasket becomes unstable if the sample hole is made much more than a third of the diamond culet diameter, or if the gasket thickness is increased beyond about a quarter of the culet diameter.[4]

Hydraulic piston-cylinder pressure systems, in contrast, allow large sample volumes but impose engineering difficulties and dangers. Seals and windows are unreliable, and stringent safety precautions must be taken against bursting and against the ejection of small parts which can travel like bullets and make holes through tables.[1] Even for the range up to 1 GPa, standard engineering safety factors cannot be used. Diamond-anvil cells are safe. Large-volume, low-pressure vessels can be very dangerous,[1] and the design and operation of such devices are best left to experienced high-pressure laboratories.

A DAC may be used to generate pressure with only a single anvil.[5] The other anvil and the gasket are replaced by a metal blank, and the cell then operates very much like a standard hardness-testing indenter. Surprisingly large pressures are generated—we have easily reached 2.5 GPa with a silver-steel blank—and the sample volume, a blind hole drilled in the blank, can be an order of magnitude larger than the standard DAC sample space. In this form the cell is very easy to use and, of course, it is completely safe.

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II. EXPERIMENT

A miniature cryogenic DAC was used, the full details of which are given in Ref. 6. The performance was evaluated using various metals, with different size sample holes and with different hydrostatic pressure media. The metals were normal bar stock from workshops, uncharacterized, except that we measured the Vickers hardness (Table I).

The sample hole was drilled without preindentation in the center of the flat surface of a blank 6 mm in diameter and 2.5 mm thick, chosen to be slightly larger than the diamond it replaces. With a 650 μm culet, we usually drilled the hole to a 250 μm diameter. The blank was glued to the piston of the cell, so as to leave the usual optical access through the bottom of the cell. The $X-Y$ adjustment was then used to center the diamond over the hole. The tilt adjustment of the cell is irrelevant in this configuration. A piece of ruby was placed inside the sample hole as an internal pressure gauge. The hole was filled with water or ethanol. The cell was then fully assembled, placed in the drive mechanism,[6] and force was applied. Condensed argon was also loaded as the pressure medium, using methods described elsewhere.[7] There was no significant difference in the behavior of the cell with the different pressure media.

The photoluminescence (PL) spectra of the internal ruby and a reference piece at atmospheric pressure were recorded on a Renishaw Raman microscope system equipped with a 632.8 nm HeNe laser. The shift between the peaks of the cell and the reference ruby were used to determine the pressure inside the sample hole. As a standard diagnostic, the pressure was plotted as a function of the force applied by a hydraulic ram. After the experiments the blanks were cut in half to examine the cross section of the sample hole.

TABLE I. Hardness and performance of the metals used for the gasket.

Metal	Micro-Vickers hardness (GPa)	Maximum pressure (GPa)
Aluminum	1.08	1.02
Brass	1.62	1.47
Silver steel	2.42	2.56
Mild steel	2.72	2.03
Stainless steel	3.49	1.86

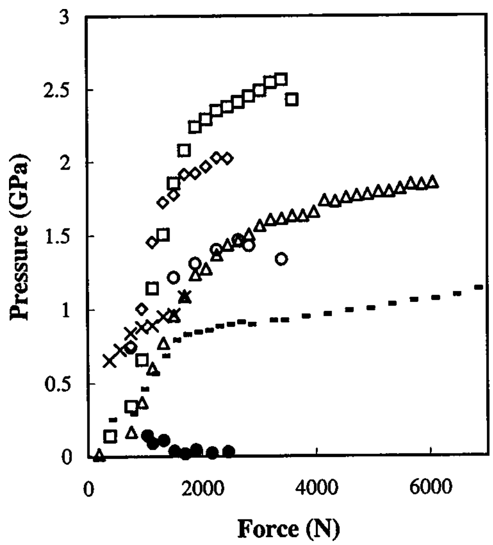

FIG. 1. Force–pressure plots for lead (●), aluminum (×), brass (○), silver steel (□), mild steel (◇), stainless steel (△), and the mushroomed stainless in Fig. 2(c) (−) blanks with 0.25 mm diam and 0.30 mm deep holes filled with ethanol.

A. Gasket material

Blanks made from lead, brass, aluminum, silver steel, mild steel, and stainless steel were tested. Using ethanol as the pressure medium, in a 300 μm deep, 250 μm diam sample hole, the maximum pressures reached are given in Table I and compared with the Vickers hardness of the metals. Typical force–pressure plots are shown in Fig. 1 and show very similar behavior, scaling roughly with the strength of the metal. Experiments were terminated when the sample hole began to enlarge, judged by visual inspection under a microscope.

The sample hole was remarkably well behaved. In most cases, it reduced slightly in both diameter and depth [Figs. 2(a) and 2(b)] but retained a fairly constant shape. In one case only, in stainless steel, the hole mushroomed [Fig. 2(c)] and this corresponded to a much lower pressure for a given force (Fig. 1).

B. Sample hole depth

Using brass blanks and a 250 μm diam hole filled with ethanol, the effect of sample hole depth was investigated. From the force–pressure plots of Fig. 3, maximum pressure is obtained with a 300 μm deep hole and deeper holes yield lower pressures. As seen in Fig. 2(b), even the deepest hole retained its shape satisfactorily.

C. Pressure medium

Using brass blanks and a 250 μm diam hole 300 μm deep, all three pressure media, water, ethanol, and argon, yielded very similar force–pressure plots, reaching about 1.5 GPa (Fig. 4). No data were obtained for argon below 1.1 GPa, because the cell has to be closed to an initial pressure sufficient to trap the condensed argon. With water and ethanol, experiments can be started at 1 bar (0.1 MPa) and the pressure raised gradually.

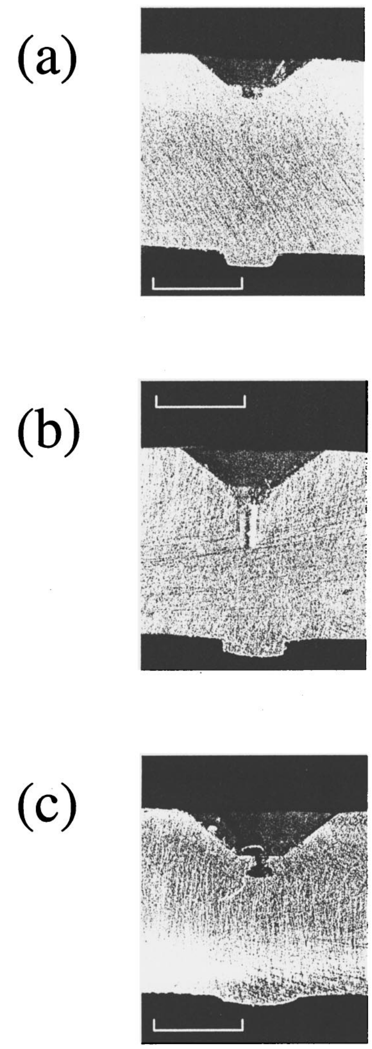

FIG. 2. Three of the blanks used are shown sectioned after the pressure run. The brass holes (a), (b) deformed in a very satisfactory way, becoming slightly shallower and slightly smaller in diameter. The stainless blank (c) behaved very poorly, mushrooming out and generating no more than 1.1 GPa. The holes were 0.25 mm in diameter, filled with ethanol, and had depths of 0.30 (a), 0.90 (b), and 0.30 (c) mm. The scale bar in each photograph is 1 mm long.

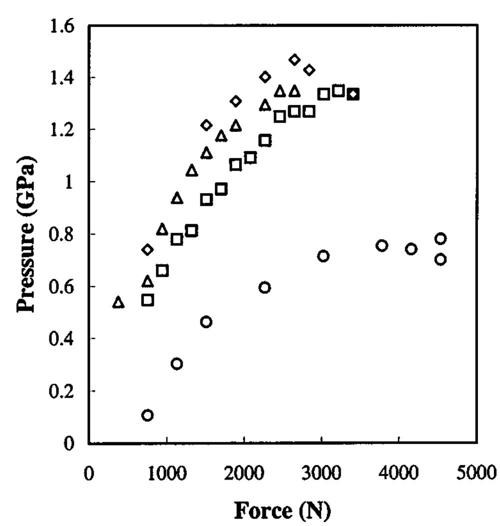

FIG. 3. Force–pressure plots for brass blanks 0.25 mm in diameter and 0.26 (△), 0.30 (◇), 0.50 (□), and 0.90 (○) mm deep holes filled with ethanol.

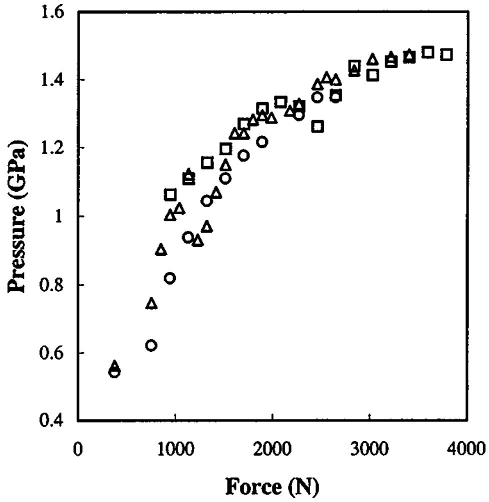

FIG. 4. Force–pressure plots for brass blanks with holes 0.25 mm in diameter and 0.25 mm deep, filled with ethanol (○), water (△), and condensed argon (□).

III. DISCUSSION

Unlike the standard DAC, the Zen DAC does not require precision machining and accurate alignment. It does not appear to be possible to break the diamond by mishandling the cell. And unlike piston-cylinder devices, it has no seals or windows that fail. It therefore provides a very easy route to attain remarkably high pressures. It would not be expected that brass, with a tensile strength of about 0.2 GPa, could support an internal pressure of 1.5 GPa. However, the situation under the diamond is not unlike indentation hardness testing. We find that for the brass, aluminum, and silver steel blanks the maximum pressure does correlate well with the Vickers hardness (see Table I for data measured on a micro-Vickers instrument), although the mild and stainless steels perform less well on this criterion.

Alternatively, we may make a comparison with an autofrettaged pressure vessel. Autofrettage, that is, plastic deformation of the material of the walls of the vessel, allows indefinitely high pressures to be sustained, increasing with the ratio of the outside diameter (b) to inside diameter (a) as $\sigma_Y(b-a)/a$ for cylindrical vessels and $\sigma_Y(b-a)^2/a^2$ for spherical vessels, which is approximately our geometry here except for the deepest sample holes. Autofrettage is very dangerous in conventional pressure vessels, but can be used here as both the risk and the dangers of explosion are zero.

In this form the diamond-anvil cell is useful for work requiring low pressures, as in biology. It is limited to optical measurements in the backscattering configuration, but this permits photoluminescence and Raman spectroscopy as well as direct observation through a microscope. There is much scope for varying both the maximum pressure and the controllability at low pressure, the slope of the $P-F$ curve, by choosing the metal and the depth of the sample hole to suit the system under study.

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[5] ''What is the sound of one hand clapping?'' traditional Zen Buddhist riddle.
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