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CURRENT CHALLENGES IN DIAMOND ANVIL CELL DESIGN

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Current challenges in diamond anvil cell design are reviewed, especially as they relate to devices for X-ray diffraction. Cells for high- and low-temperature use are described, as are those for sideways X-ray scatter in diamond anvil cells, and for microscope use.

Introduction

The price of diamond imposes a practical limit on the size of anvil that may be used routinely in constructing dac's. Moreover, as the culet size is increased, the load that must be generated increases greatly and therefore poses engineering challenges. This leads directly to the main disadvantage of dac's, the small sample size that may be accommodated. However, in recent years there has been rapid development of measurement techniques, especially in the X-ray field. It is now possible to use a wide range of observational techniques with dac's. For example, a full single-crystal structure determination can now be performed on a crystal as small as 100 microns in side.

We consider that the major challenges in diamond anvil cell (dac) design, especially from a commercial viewpoint, are as follows.

1. To provide enough directions, and sufficiently large angles of observation whilst achieving the desired pressures and maintaining stability of anvil alignment. This challenge is especially acute in the X-ray field.

2. In addition, there are often physical constraints imposed by the sample space and environmental conditions. There is an increasing demand for non-magnetic dac's.

3. To these challenges must now be added the desire to combine high pressures with both cryogenic and very high temperatures. It is already possible to create in dac's the *P* and *T* combinations typical of those down to the centre of the earth itself.

4. Provision for continuous, and often remote, variation of pressure whilst the sample is held at either high or low temperatures.

There is now an awesome area of *P–T* space accessible to experimentalists. It is this market that Diacell Products Ltd seeks to serve in an innovative fashion.

The genaral features of dac's are well known. In this paper, the above challenges will be illustrated specifically by reference to developments in the field of dac's for use with X-ray diffraction.

General features of a dac for use in X-ray diffraction

Fig. 1 shows a section of the DXR-6 dac. The anvils are supported by plates of beryllium shaped to provide a constant absorption path to diffracted X-rays up to $2\theta =$ $= 45°$. There is a very small central hole in each plate to allow for (a) alignment and (b) the possibility of pressure estimation by the ruby R-line method.

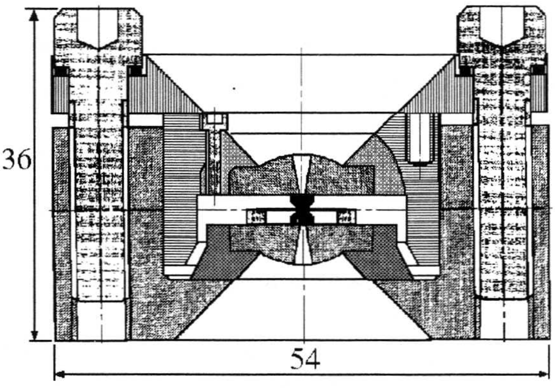

Fig. 1. The DXR-6 cell

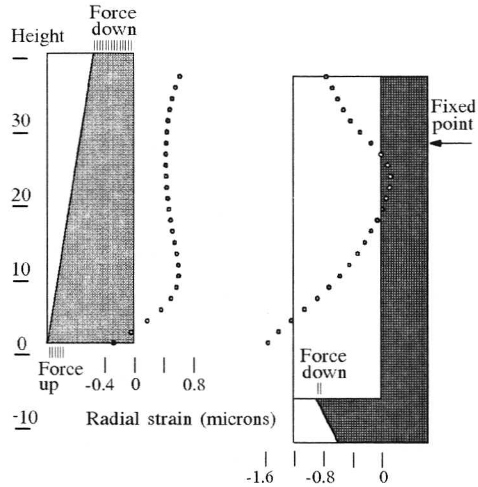

Fig. 2

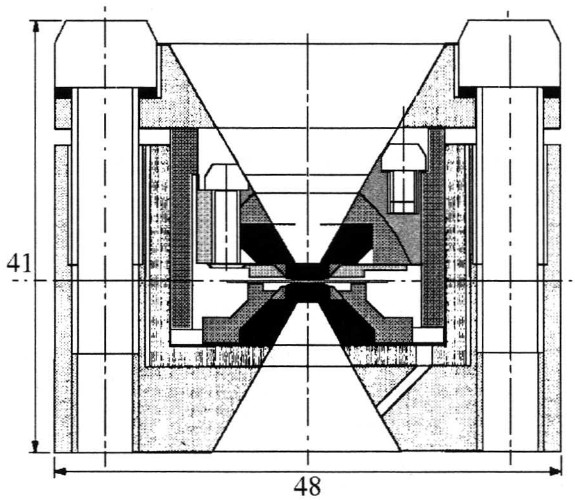

Fig. 4. The DXR-7 cell

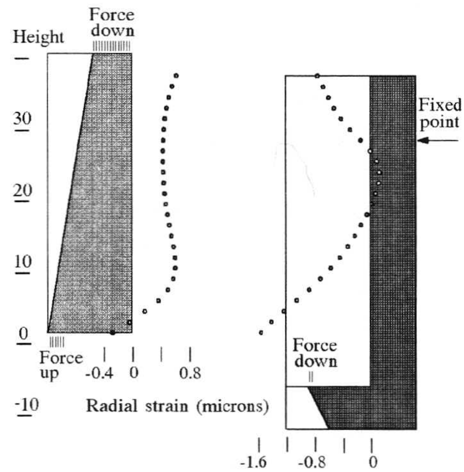

Fig. 3

Pressures of 0.95 Mbar have been achieved with this device, using anvils with culets of 200 rm diameter. At such pressures, the anvils sink into the beryllium plates by about 200 rm but then remain stable. Finite element calculations show that at these high applied loads, the components of the cell behave not unlike the leaf-spring of a motor vehicle: all of them yield a little, and destructive tensile and shear stresses are accommodated by being spread over more than just the anvil and its' beryllium support plate. This approach to design of high pressure devices contrasts with the Bridgman «massive support» philosophy, and becomes more attractive now that there is ready access to finite element analysis programs.

The maximum load that the DXR-6 can sustain is limited mainly by two factors: (a) friction associated with the bolts used to load the cell, and (b) distortion of the piston-cylinder assembly. Factor (a) could be reduced by using an external loading device, with the dac bolts used simply to clamp the cell once loaded. However, this is difficult if the dac is mounted on a goniometer.

We have estimated the distortions under load of both piston and cylinder using finite element methods. The results are shown in Fig. 2. The distortions are such that at the highest loads, there is interference between the two parts resulting in much increased friction. There are two ways around this problem.

(a) In the DXR-6 the piston-cylinder clearance is typically 2 rm. Increasing this by making the piston a little smaller relative to the cylinder would eliminate the interference but at the cost of poorer stability of alignment. (b) An alternative, see Fig. 3, is to ensure that the piston and cylinder are not load bearing. This is the approach taken in the DXR-7 cells, Fig. 4.

X-ray dac's without beryllium support plates

Cells with anvil supports made of beryllium perform well and are widely used but they suffer from the disadvantages associated with having beryllium in the scattered beam, namely absorption, and scatter which interferes with the data it is desired to collect. Moreover, the strength of beryllium falls rapidly with increase of temperature. These disadvantages are readily overcome by use of tungsten carbide support plates, but at some cost.

Mounting a diamond anvil of the same size (2.5 mm diameter) as is used in the DXR-6, over a radiation port in the carbide plate of the same size as is used for optical experiments (usually about 1 mm diameter), reduces the accessible 2θ angle to about 15°. This is acceptable for energy-dispersive studies but not for other kinds of experiment. Opening the radiation port further requires anvils of greater diameter and volume, and therefore also of cost. To achieve 2θ of 30° on carbide support requires a hole in the carbide of 2.1 mm diameter, and an anvil of 3.5 mm diameter at the girdle. In order to control destructive tensile stresses in the anvil base, it is necessary also to reduce the pavilion angle. All the parameters of the anvil, support plate, and of the piston-cylinder have been chosen following stress analyses. The initial aim is to reach 1 Mbar routinely. The result is the cell of Fig. 4.

X-ray dac's for use at very high temperatures

Temperatures of up to 3500°C have been reached in dac's using Nd-YAG-laser-heating of the sample. Typically, a sample spot of 10 to 20 μm diameter is heated. Whilst the other cell components can get seriously hot during extended runs, the temperatures generated are not such as to pose a threat to the mechanical integrity of the cell. The DXR-7 is suitable for such work.

There is, however, considerable interest in using dac's to temperatures up to 1000°C. This can be done by direct resistive heating, and avoids the enormously expensive systems needed for laser-heating and the accompanying pyrometry. Diamond is stable in air at these temperatures. There is some tarnishing of the exterior surfaces, but this may be avoided by flushing them with dry nitrogen to which a little hydrogen has been added. More seriously, above about 800°C tungsten carbide support plates begin to disintegrate as the binder drops out. We use a combination of internal and external resistive heaters.

X-ray dac's for very low-temperature use

Clamp cells for cryogenic X-ray (and other kinds of) use are simple to make. The challenge comes in designing such dac's in which the load can be varied whilst the device is at low temperature. One simple means of achieving this is to use gas-membrane drive, as in our DXR-GM cell. The membrane is filled with helium gas to a maximum pressure of 200 bar. At this pressure, helium undergoes a gas-to-solid transition at 5.2 K, thereby forming a lower temperature limit at which the device may be used. However, down to say 8 K, the DXR-GM device is proving very successful. The difficulty is to make a device which will operate below this temperature.

An alternative approach to continuous pressure variation whilst at low temperature is the miniature cryogenic dac (MCDAC) of Dunstan. In this device, load is applied to the dac using two piano wires that loop around the base of the dac and run through steel tubes. It is used routinely to 6 to 10 K, and has been taken to 2 K in superfluid liquid helium. However, although it is now quite widely used for optical and electrical studies, the geometry of this design is not compatible with the needs of X-ray diffraction, because the dac axis is parallel to that of the cryostat, and access is very limited.

We have devised an X-ray dac that uses only one drive wire and is estimated to reach as low as 1 K, see Fig. 5. The new feature is use of an intermediate load multi-

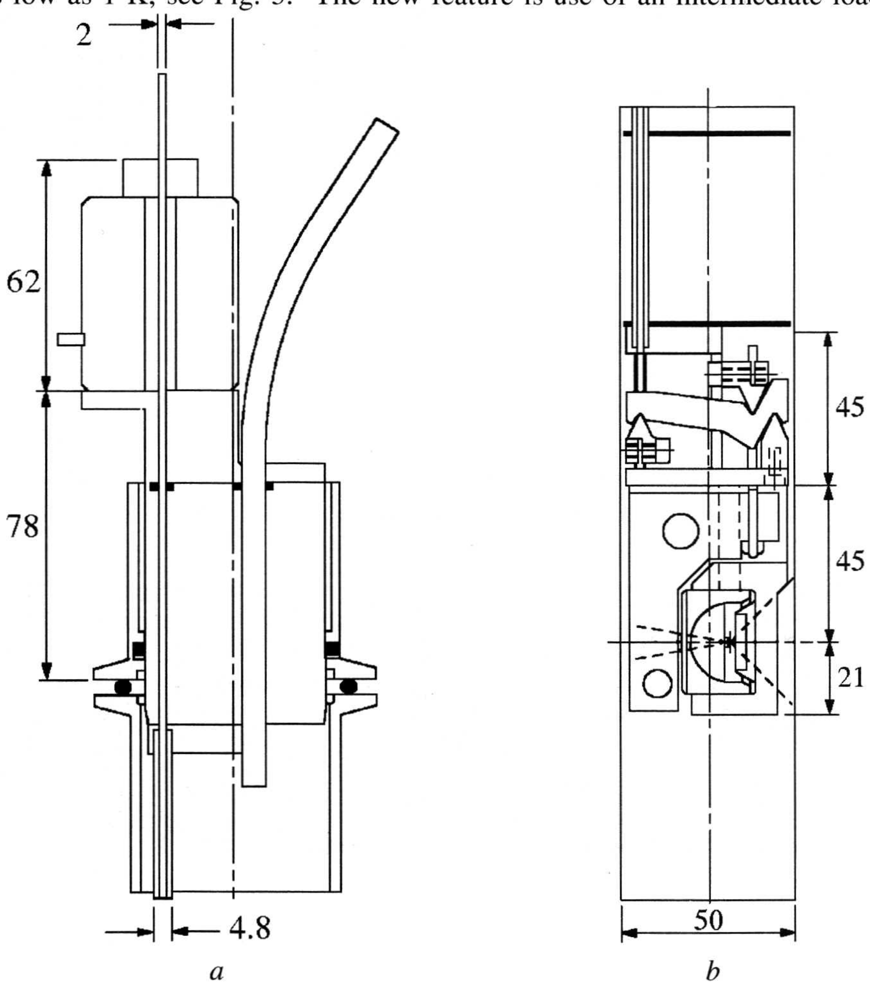

Fig. 5

plication stage in the form of a lever working on a pivot. This is low down in the cryostat. The device has been designed for X-ray use but can be modified for optical work.

X-ray dac's for use in sideways scatter geometry

There is increasing interest in X-ray diffraction experiments in dac's in which the beam is input in a direction normal, or nearly normal, to the axis of the two anvils. In such experiments the sample is contained within a beryllium gasket. Fig. 6 shows a schematic of a cell to meet such a need.

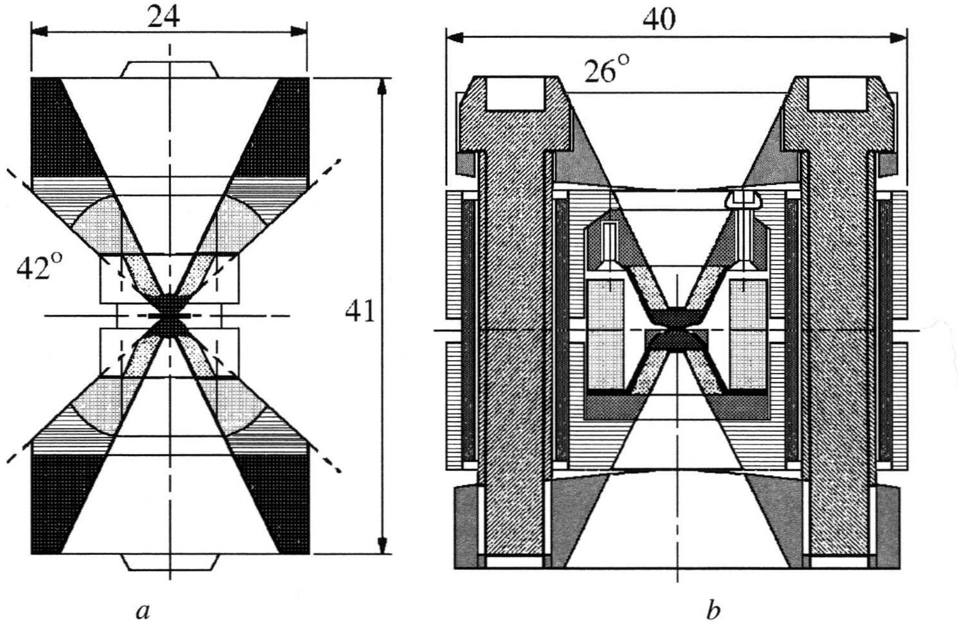

Fig. 6

Dac's for both X-ray and optical use

Some applications require dac's with which both optical and X-ray diffraction studies can be made. The non-beryllium type of X-ray dac is, of course, especially suitable also for optical and other studies. However, many optical, Raman and infrared

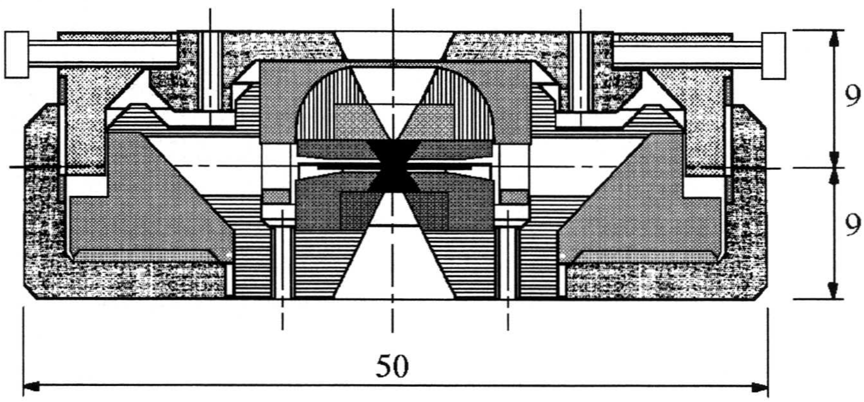

Fig. 7

investigations require use of a microscope. In Fig. 7 we show a dac that is only 18 mm thick along the axis and thus eminently suitable for microscope use. It may be configured for X-ray use only, for optical use only, or for both. It is expected to reach 1 Mbar with anvil culets of 0.2 mm.