

## 2. INSTRUMENTATION AND SAMPLE PREPARATION

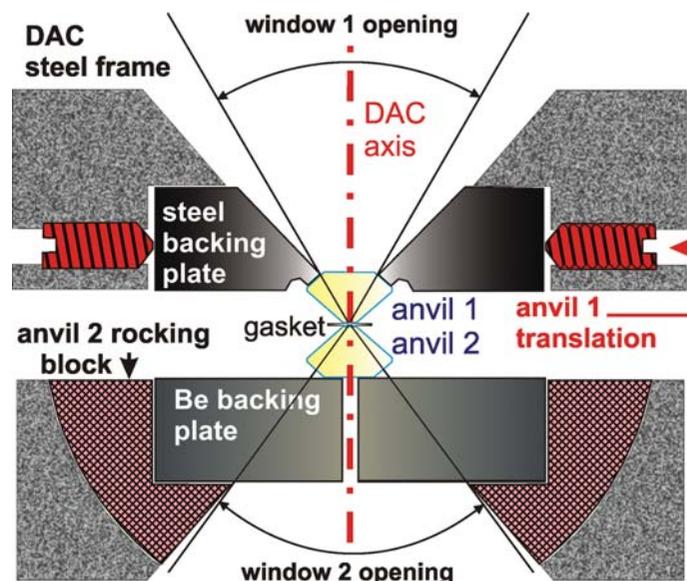
2004; Fabbiani *et al.*, 2004; Fabbiani & Pulham, 2006; Budzianowski & Katrusiak, 2006a,b; Dziubek *et al.*, 2007; Paliwoda *et al.*, 2012; Sikora & Katrusiak, 2013).

The original designs of the DAC (Weir *et al.*, 1959; Jamieson *et al.*, 1959) were later adapted to various purposes. Significant modifications take advantage of new designs of diamond anvils and their supports. Initially, the brilliant-cut diamonds of traditional design, but with the culet ground off to form a flat thrust surface parallel to the table, were used (Fig. 2.7.4). Culets of 0.8 mm in size can be used to about 10 GPa, 0.4 mm culets to about 50 GPa, 0.1 mm culets to about 100 GPa and 0.02 mm (20  $\mu\text{m}$  in diameter) or even smaller (Akahama *et al.*, 2014; Akahama & Kawamura, 2010; Dalladay-Simpson *et al.*, 2016) culets can be used for the megabar 200–400 GPa range. The megabar range requires bevels on the culets to protect their edges from very high strain and damage. The bevels are about 6–7° off the culet plane and the ratio of bevel-to-culet diameters is between 10 and 20. Also, the gasket material, the hole diameter and its height, being a fraction of the hole diameter, are of primary importance. Double bevels can be used to release the strain further, but it appears that a value of about 400 GPa is the maximum pressure attainable in the conventional DAC (c-DAC).

The pressure limits of the c-DAC are surpassed in a double-stage DAC (ds-DAC), in a toroidal DAC (t-DAC) or by shock compression. In the ds-DAC a pair of small anvils, constituting a microscopic DAC (m-DAC, also described as second-stage anvils), is contained inside the c-DAC. The micro-anvils are prepared from diamond or amorphous diamond using the focused ion-beam technique (Sakai *et al.*, 2015, 2018). For another type of ds-DAC, employing microscopic diamond hemispheres (Dubrovinsky *et al.*, 2012; Dubrovinskaia *et al.*, 2016), pressures exceeding 1 TPa have been reported. In the t-DAC, each diamond culet is modified in such a way that an ion-beam-eroded groove surrounds the central micro culet (Dewaele *et al.*, 2018; Jenei *et al.*, 2018; Mao *et al.*, 2018).

At present, the DAC most commonly applied in laboratories is a miniature Merrill–Bassett DAC, where the anvils are installed on two triangular frames driven by three screws along three sliding pins (Merrill & Bassett, 1974). Analogous designs with two or four thrust-generating screws are also in use. The original Merrill–Bassett DAC was equipped with a pair of brilliant-cut 0.2 carat diamonds with polished culets (Fig. 2.7.4) and the anvils were supported on Be discs. The Merrill–Bassett DAC is optimized for use with automatic diffractometers. It contains no rocking blocks but allows translation of one of the anvils. The light weight and small size allow the Merrill–Bassett cell to be routinely used on single-crystal diffractometers. This simple DAC design is suitable for experiments up to about 10 GPa. Dedicated DACs for higher pressure have rocking supports for the diamonds, in the form of either hemispheres or half-cylinders (Fig. 2.7.5).

A very fine adjustment of the anvils and fine and remote pressure control can be obtained in a membrane DAC, where the thrust is generated by a metal membrane operated with gaseous helium or nitrogen (Letoulec *et al.*, 1988; Chervin *et al.*, 1995). Owing to the ideally coaxial thrust generation by the membrane and the stable supports of the anvils, usually in the form of a piston and cylinder, the membrane DAC is suitable for generating pressures of hundreds of gigapascals. The membrane DAC can be operated remotely through a flexible metal capillary, which is advantageous for spectroscopy and both powder and single-crystal diffraction experiments at synchrotrons.



**Figure 2.7.5**

A cross section through the central part of a diamond-anvil cell, schematically showing the main elements applied in various designs. Usually, either beryllium backing plates or steel/tungsten carbide backing plates with conical windows are used. One of the plates can be translated and the other rocked in all directions (the hemispherical rocking mechanism). In other designs, one of the anvils can be rocked around and translated along one axis, and the other anvil rocked and translated in the perpendicular direction (two perpendicular hemicylindrical mechanisms). The usual thickness of the beryllium plate is 3 mm or more, and most constructions allow a window opening of about 40° to the DAC axis. The thickness of the diamond window (the table-to-culet distance) is usually about 1.5 mm.

### 2.7.5. Variable-temperature high-pressure devices

One of the most common interests in extreme conditions combines high pressure and high temperature. Several techniques for simultaneously controlling both pressure and temperature have been developed (Fei & Wang, 2000). The DAC can be heated externally (with respect to the sample chamber between the anvils' culets) when the entire DAC is placed in an oven or in a hot stream of air from an electrical heater (Fourme, 1968; Allan & Clark, 1999; Podsiadło & Katrusiak, 2008; Bujak *et al.*, 2008). External resistance-wire heaters placed immediately around the diamond anvils and the gasket are often used (Bassett & Takahashi, 1965; Takahashi *et al.*, 1982; Adams & Christy, 1992; Eremets, 1996; Moore *et al.*, 1970; Hazen & Finger, 1982; Besson, 1997; Dubrovinskaia & Dubrovinsky, 2003; Fei & Wang, 2000). External heating can routinely operate up to about 673 K. Its main advantages are stability, reliable measurement of temperature and high homogeneity of temperature in the chamber. The disadvantages include the relatively low temperature range and the large mass of the DAC mechanical parts that are heated. Their thermal expansion can cause loss of pressure. This does not apply to the membrane DAC, where a constant thrust from the membrane is transmitted to the anvils. A sophisticated externally heated DAC in an atmosphere of inert gases is capable of operating between 83 and 1473 K (Bassett *et al.*, 1993).

A very small turnbuckle DAC, about 6 mm in diameter, was originally constructed of plastic and hardened beryllium–copper alloy (BERYLCO 25) in order to perform magnetic measurements at low temperature in the small bore of a superconductive quantum interference device (SQUID) (Graf *et al.*, 2011; Giriat *et al.*, 2010). These are at present the smallest designs that can be