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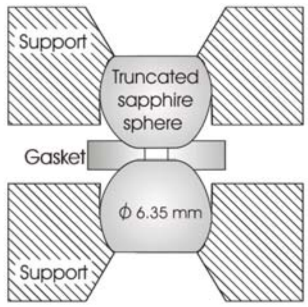


**Figure 2.7.3**
A schematic view of the opposed-sapphire anvil of the Kurchatov–LBB cell designed for neutron diffraction on magnetic materials (Goncharenko, 2004).

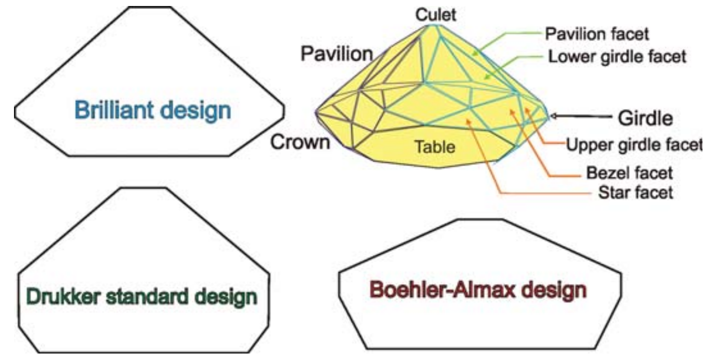


**Figure 2.7.4**
Cross sections of three types of diamond anvil used in high-pressure cells: the brilliant design (supported either on the table or on the crown rim), the Drukker design (supported on the table and crown) and the Boehler–Almax design (supported on the crown).

The sample can be either contained in a capsule or mixed with a pressure-transmitting pseudo-hydrostatic medium, which is inert and a weak absorber of X-rays. The sample is accessed by the X-ray beam between the anvils through a weakly absorbing sealing material, such as amorphous boron, magnesium oxide, corundum or pyrophyllite. Like the opposed-anvil presses, multi-anvil LVPs can be used effectively for X-ray diffraction at synchrotrons and neutron sources. However, these large installations also require (apart from intense primary beams) powerful translations for their precise centring relative to the primary beam and diffractometer axes. The main advantage of an LVP is stable and homogenous internal heating up to about 2000 K (Besson, 1997). Such stable conditions are particularly valuable for high-pressure synthesis and crystallization, for example of diamonds (Hazen, 1999).

### 2.7.4. The diamond-anvil cell (DAC)

The invention of the DAC revolutionized high-pressure studies, diversified their scope, greatly simplified the experimental procedures, increased the range of pressure and temperature, and initiated constant growth in the number of high-pressure structural studies, starting in the 1960s and continuing up to today. The DAC is built from a pair of opposing diamond anvils and a vice to generate their thrust. The sample is compressed between the culets of the anvils. Since its inception, the DAC has been modified and redesigned frequently, in order to adapt it to new experimental techniques or to take advantage of the parallel progress in scientific equipment. The original DAC built at the National Bureau of Standards (Maryland, USA) was used for infrared spectroscopy. Another DAC designed for powder diffraction experiments was made of beryllium, a relatively strong metal which weakly absorbs short-wavelength X-rays (Weir *et al.*, 1959; Bassett, 2009). The DAC, with steel frames and beryllium discs supporting the anvils, is still in use today.

The original and most efficient concept applied in the operation of the DAC was that the incident beam enters the pressure chamber through one diamond anvil and the reflections leave through the other anvil; this mode of operation is often referred to as transmission geometry. Together with the diamond anvils, Be discs constitute windows for the X-rays. However, beryllium has several disadvantages. It is the softest and weakest of the materials used in DAC construction, it softens at about 470 K, beryllium oxide is poisonous, and machining beryllium is difficult and expensive. Therefore, except for the pioneering DAC design by Weir *et al.* (1959), Be parts were initially limited to disc supports for the anvils. Moreover, polycrystalline Be discs produce broad reflection rings and a strong background, and the small central hole in the disc obscured optical observation of the sample. In many modern DACs the beryllium discs have been completely eliminated, and the diamond anvils are directly supported by steel or tungsten carbide platelets (Konno *et al.*, 1989; Ahsbahs, 2004; Boehler & De Hantsetters, 2004; Katrusiak, 2008). For this purpose new diamond anvils, exemplified in Fig. 2.7.4, were designed. Anvils of different sizes, culet dimensions, height-to-diameter ratios and other dimensions can be adjusted for the experimental requirements, such as the planned pressure range and the opening angles of the access windows.

Another DAC was independently designed for X-ray powder diffraction by Jamieson *et al.* (1959). In their DAC, the incident beam was perpendicular to the axis through the opposed anvils, and the primary beam passed along the sample contained and squeezed directly (no gasket was used) between the culets. The reflections were recorded on photographic film located on the other side of the DAC, perpendicular to the incident beam. This geometry was described as either panoramic, perpendicular or transverse. The transverse geometry is also used with beryllium or other weakly absorbing gaskets (Mao *et al.*, 1998). Other DACs, for example where both the incident beam and the reflections pass through one diamond anvil, were also designed (Denner *et al.*, 1978; Malinowski, 1987); however, the transmission geometry is most common owing to its advantages. In the transmission geometry the uniaxial support of the anvils leaves a window for optical observation of the sample, as well as for spectroscopic and diffractometric experiments along the cylindrical pressure chamber. Therefore, at present most DAC designs operate in transmission geometry.

The DAC construction can generally be described as a small vice generating thrust between opposed anvils. In the first DACs designed in the late 1950s, no gasket nor hydrostatic fluids were used and the sample was exposed to strong anisotropic stresses. Van Valkenburg (1962) enclosed the sample in a hole in a metal gasket, filled the hole with hydrostatic fluid and sealed it between the culets of the anvils. This most significant development of the miniature high-pressure chamber opened new possibilities for all sorts of studies under hydrostatic conditions, in particular powder and single-crystal diffraction studies. Since then, the gaskets have become an intrinsic part of the DAC. The hydrostatic conditions in the DAC have been used to grow *in situ* single crystals from the melts of neat compounds (Fourme, 1968; Piermarini *et al.*, 1969) and from solutions (Van Valkenburg *et al.*, 1971*a*,*b*). Now it is a common method for *in situ* crystallization under isothermal and isochoric conditions (Dziubek & Katrusiak, 2004; Bujak *et al.*, 2004; Fabbiani *et al.*, 2004; Fabbiani & Pulham, 2006; Budzianowski & Katrusiak, 2006*a*,*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 μm 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 (Letoullec *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.

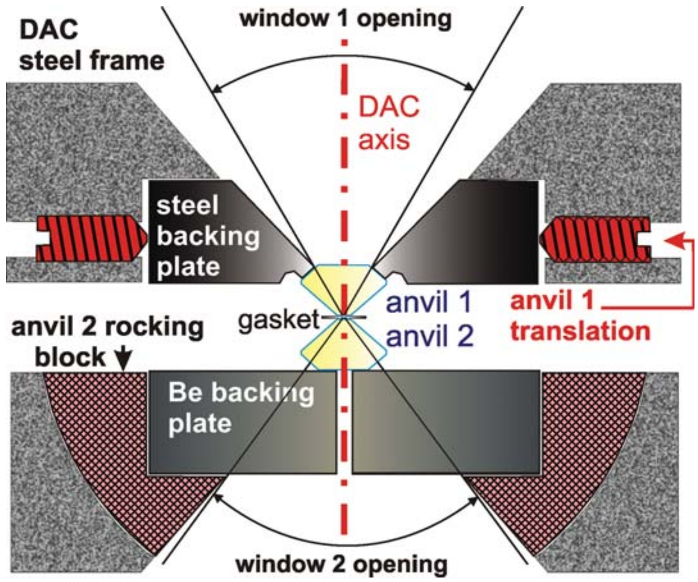


**Figure 2.7.5**
A cross section through the central part of a diamond-anvil cell, schematically showing the main elements applied in various designs. Usually, either beryllium backing plates or steel/tungsten carbide backing plates with conical windows are used. One of the plates can be translated and 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 hemi-cylindrical 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


references