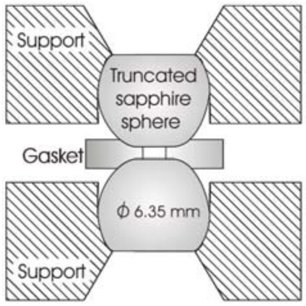

Figure 2.7.3
A schematic view of the opposed-sapphire anvil of the Kurchatov–LBB cell designed for neutron diffraction on magnetic materials (Goncharenko, 2004).

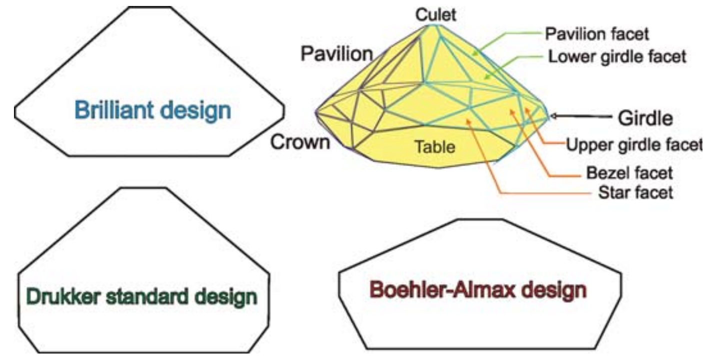

Figure 2.7.4
Cross sections of three types of diamond anvil used in high-pressure cells: the brilliant design (supported either on the table or on the crown rim), the Drukker design (supported on the table and crown) and the Boehler–Almax design (supported on the crown).

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.*,