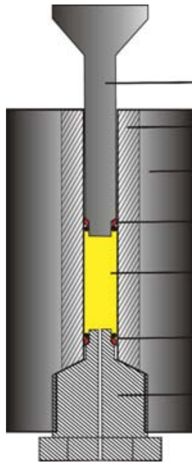

Figure 2.7.1
A cross section through a piston-and-cylinder device with a shrink-fitted double cylinder. In this design, the bottom piston (stopper F) is in the fixed position and only the top piston (A) is pushed into the cylinder by a hydraulic press. The main components of the device are: piston (A), inner cylinder (B), outer supporting shell (C), brass and rubber sealing rings (D), sample (E) and stopper (F).

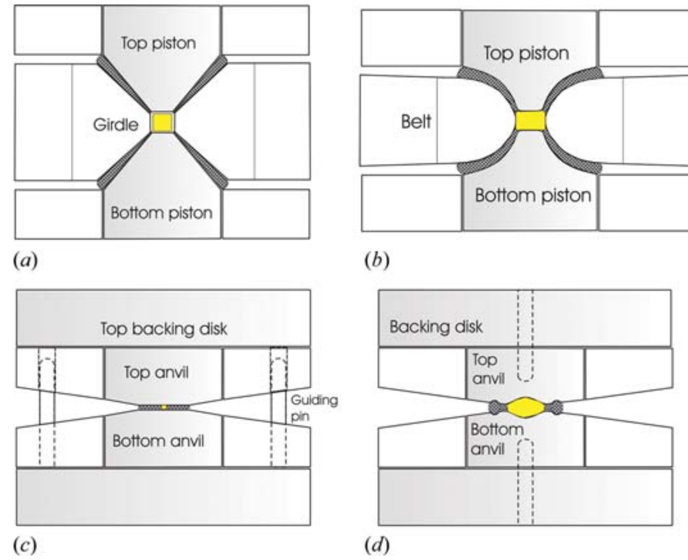

Figure 2.7.2
Cross sections of (*a*) the girdle anvil, (*b*) the belt anvil, (*c*) the Bridgman anvil and (*d*) the toroid anvil. The gaskets are dark grey, the tungsten carbide elements are pale grey and the sample chamber is yellow.

The PaC press can be considered as the prototype of other large-volume presses (LVPs). The PaC press consists of a cylinder closed at both ends with pistons, sealed with gaskets. The attainable pressure depends mainly on the tensile strength of the cylinder and of the gasket. The cylinder can be strengthened by the process of frettage, *i.e.* inducing tensile strain in the outer part and compressive strain inside (Onodera & Amita, 1991). This can be achieved by autofrettage, when a one-block cylinder is purposely overstrained to the point of plastic deformation and the deformation residues result in the required strain. Likewise, the cylinder can be built of shrink-fitted inner and outer tubes (the outer diameter of the inner tube is slightly larger than the inner diameter of the outer tube, which must be heated to assemble the cylinder) or of cone-shaped tubes (with a cone half-angle of *ca* 1°) pushed into one another to generate the frettaging strains. Alternatively, a coil of several layers of strained wire or tape can be wound around the cylinder, or the cylinder can be compressed externally to counteract its tensile strain simultaneously with the load being applied to the pistons (Baranowski & Bujnowski, 1970). The load against the cylinder walls can be reduced by containing the sample in a capsule of soft incompressible material, usually lead (Bridgman, 1964). Cylinder chambers with externally generated pressures up to 0.4 GPa (Blaschko & Ernst, 1974) and PaC cells capable of generating 2 GPa (Bloch *et al.*, 1976; McWhan *et al.*, 1974) have been used for neutron diffraction, and a beryllium cylinder has been used for X-ray diffraction on protein crystals to 100 MPa (Kundrot & Richards, 1986). The range of pressure up to a few hundred megapascals is often described as medium pressure. There are sample-environment chambers with externally generated medium pressure that are designed for in-house powder diffractometers operating in the Bragg–Brentano geometry (Koster van Groos *et al.*, 2003; Whitfield *et al.*, 2008).

If the cylinder length is reduced and the gasket reinforced by compression of the conical pistons, the girdle press is obtained (Fig. 2.7.2*a*). Its optimized modification is the belt apparatus (Fig. 2.7.2*b*). The girdle and belt presses generate pressures of about 10 GPa and can be internally heated to about 1500 K, and hence they have been used widely to synthesise materials. However, the opacity of the girdle/belt and anvils allows no access for X-ray or neutron beams between the anvils. This disadvantage is alleviated in the opposed-anvils press, operating on the massive-support principle, where the beams can pass through the gasket material. After the first record of a simple version of the opposed-anvils experiment in the mid 19th century performed in order to measure the effect of medium pressure on the electric conductibility of wires by Wartmann (1859), the opposed anvils were extensively developed and applied to much higher pressure by Bridgman (1935, 1941, 1952). He also equipped them with a pyrophyllite gasket separating the anvil faces (Bridgman, 1935), and in this form they are commonly known as Bridgman anvils (Fig. 2.7.2*c*). In the 1960s, the flat faces of the Bridgman anvils were modified to so-called toroidal anvils (Fig. 2.7.2*d*), where the sample space is considerably increased by hemispherical depressions at the anvil centre and surrounded by a groove supporting the gasket and preventing its extrusion (Khvostantsev, 1984; Khvostantsev *et al.*, 1977, 2004; Ivanov *et al.*, 1995). Anvils with a spherical sample cavity only, so-called Chechevitsa anvils, preceded the construction of toroidal anvils. Toroidal anvils were optimized for neutron diffraction by adding a small pneumatic press called the Paris–Edinburgh cell (Besson *et al.*, 1992). Toroidal anvils enabled neutron diffraction studies up to 50 GPa and 3000 K (Kunz, 2001; Zhao *et al.*, 1999, 2000; Redfern, 2002). Experiments on magnetic systems in a similar pressure range and at low temperature were performed in a very different design of opposed anvils, the sapphire Kurchatov–LBB cell, shown in Fig. 2.7.3 (Goncharenko & Mirebeau, 1998; Goncharenko, 2004).

More complex LVPs have been based on multi-anvil presses (Liebermann, 2011). These are usually very large devices capable of containing tens of cubic centimetres of sample. The sample is encapsulated and pressurized between anvils sealed with some kind of gasket. The attainable pressure depends on a number of factors, including the applied load and the strength of the anvils, which are made of steel, tungsten carbide, sapphire or sintered diamond; the maximum pressure depends inversely on the sample volume. Multi-anvil presses (Huppertz, 2004; Liebermann, 2011) – tetrahedral, trigonal–bipyramidal, cubic (Akimoto *et al.*, 1987) and octahedral (Onodera, 1987) – are optimized for larger sample volumes and for the high temperatures required for the synthesis of hard materials, especially diamond (Hazen, 1999). The multi-anvil presses are used for diffraction studies.

  references