used for X-ray diffraction studies, with the whole DAC cooled by commercial low-temperature gas-stream attachments. Alireza & Lonzarich (2009) built another miniature DAC for high-pressure magnetic measurements in a SQUID.

Temperatures of several thousand kelvin can be achieved by internal heating, where the sample absorbs the focused light beam of a laser (Bassett, 2001; Ming & Bassett, 1974; Shen *et al.*, 1996) or is heated by a thin wire passing through the chamber or its immediate surroundings, either in the gasket walls (Boehler *et al.*, 1986; Mao *et al.*, 1987; Zha & Bassett, 2003; Dubrovinsky *et al.*, 1998) or in the culets of intelligent diamond anvils (Bureau *et al.*, 2006). Composite resistance gaskets, with a platinum chamber wall acting as a 35 W resistance heater, can increase the temperature to over 2273 K (Miletich *et al.*, 2000, 2009). Laser beam(s) focused through the DAC anvil(s) onto the sample (Boehler *et al.*, 2001) can heat it to over 3273 K. This requires that the laser beam, or several beams, or a fraction of their energy, be absorbed in the sample. In order to increase the absorption, the sample can be mixed with another compound, for example gold powder. The main disadvantage of laser heating is inhomogeneous distribution of the temperature within the sample.

Much smaller temperature gradients, of a few kelvin at 2773 K, can be obtained in large-volume presses (LVPs). The multi-anvil LVP has traditionally been applied for the synthesis of diamond, which requires stable conditions of both high pressure and high temperature (Hazen, 1999; Liebermann, 2011). In the LVP, a resistance heater installed inside the chamber can provide stable control of the temperature for days, while the pressure is controlled by a hydraulic press. Owing to the large sample volume, the diffraction pattern can be quickly recorded. Most often, energy-dispersive diffraction is applied for the beams entering and leaving the pressure chamber through the gasket material between the anvils. LVPs are generally very large and heavy, which contrasts with the compact construction of the Paris–Edinburgh and Kurchatov–LLB pressure cells (Besson *et al.*, 1992; Goncharenko, 2004, 2006). Both these opposed-anvil cells can be placed in cryostats, and they can be used for either energy- or angle-dispersive diffraction of neutrons or X-rays. The Kurchatov–LLB cell has been optimized for neutron diffraction studies of magnetic structures at high pressure and low temperature (Goncharenko & Mirebeau, 1998; Goncharenko *et al.*, 1995).

2.7.6. Soft and biomaterials under pressure

Interest in the effects of pressure on biological materials is connected to the processing of food and the search for methods of modifying the structure of living tissue and its functions. Soft biological compounds, including proteins, membranes, surfactants, lipids, polymer mesophases and other macromolecular assemblies present in living tissue, are susceptible to pressure, which can affect the molecular conformation and arrangement with relatively low energies of transformation (Royer, 2002). Medium pressure suffices for protein coagulation, as observed for egg white at 0.5 GPa by Bridgman (1914). However, single crystals of egg-white lysozyme survived a pressure of several gigapascals (Katrusiak & Dauter, 1996; Fourme *et al.*, 2004), which was connected to the concentration of the mother liquor used as the hydrostatic fluid. Cells with externally generated pressures up to about 200 MPa for diffraction measurements on single crystals in a beryllium capsule (Kundrot & Richards, 1986) and on powders contained between beryllium windows (So *et al.*, 1992) have been built. Powder diffraction studies have also been

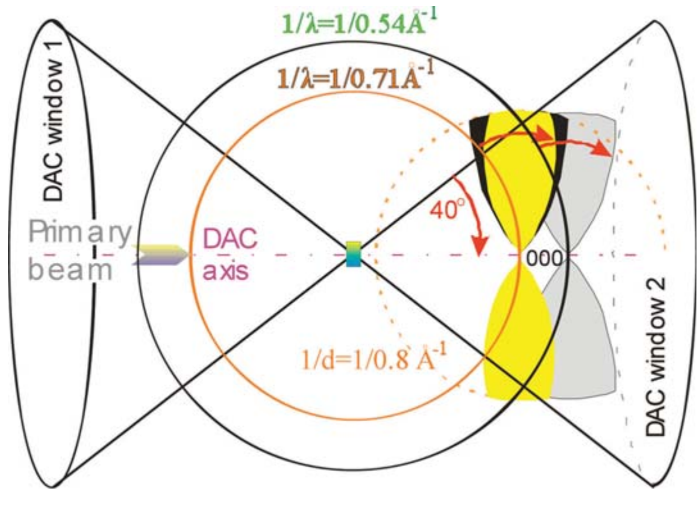

Figure 2.7.6
A diamond-anvil cell, showing the 40° half-angle opening of the conical windows and the reciprocal space accessed for a single-crystal sample and Mo $K\alpha$ or Ag $K\alpha$ radiation. In this schematic drawing, the window cones intersect at the disc-shaped sample (yellow–blue shaded rectangle) and around it the Ewald spheres of reciprocal radii corresponding to Mo $K\alpha$ and Ag $K\alpha$ wavelengths are drawn. The shape of the two yellow profiles meeting at the reciprocal 000 node is the cross section through the torus-like accessible volume of reciprocal space for Mo $K\alpha$ radiation; this torus is circularly symmetric about the DAC axis. The grey shape is likewise the accessible space for Ag $K\alpha$ radiation. Both are at the same resolution of $1/d_{hkl} = 1/0.8$ Å^{-1} (corresponding to θ angles of 26.4° for Mo $K\alpha$ radiation and 19.7° for Ag $K\alpha$). For a powdered sample, all reciprocal-space nodes contained within the resolution sphere (dotted circle) can be recorded. The DAC windows and the sample are shown at the initial 'zero' position, when the DAC axis coincides with the primary beam; the red arrows indicate the rotation of the DAC, sample and Ewald sphere to the limiting 40° angle.

performed on samples frozen under high pressure and recovered to ambient pressure (Gruner, 2004). High-pressure studies can be conveniently performed in the DAC, but because of the usually weak scattering of macromolecular samples, synchrotron radiation is preferred for such experiments (Fourme *et al.*, 2004; Katrusiak & Dauter, 1996).

2.7.7. Completeness of data

The steel parts of the DAC can restrict access of the incident beam to the sample and can obscure the exit of reflections. For a typical DAC working in transmission mode, the incident beam can be inclined to the DAC axis by up to about 25–40°, for the full opening of the window of 50–80°, respectively. In most DACs the collimator and detector sides are symmetric, so the opposing conical windows have the same opening angle. This limited access to the sample can affect the completeness of diffraction data for low-symmetry crystals, which can then pose considerable difficulties in solving and refining crystal structures from single-crystal measurements.

The restricted access of the primary and diffracted beams to the sample can conveniently be described by the concept of the reciprocal lattice (Fig. 2.7.6). The initial orientation of the crystal in the DAC defines the accessible region of the reciprocal lattice in such a way that the Ewald sphere can be inclined to the initial direction of the incident beam by up to the maximum window opening angle, denoted α_M. The sample can be accessed from both sides of the DAC (by rotating the DAC by 180°) and thus the accessible region of reciprocal space has the form of a round flat cushion, with surfaces touching at the cushion centre [described