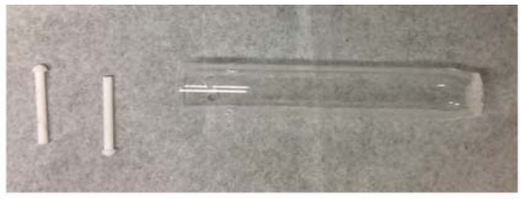

(*a*)

(*b*)

**Figure 2.10.51**
Two sample holders used for high-temperature studies. (*a*) A cell made of quartz with frits at the bottom to allow gas flow through the sample. (*b*) A holder on the right made of vanadium but using a boron nitride top with molybdenum bolt, nuts and washers to avoid melting due to eutectic formation. The fitting on the far left, which is made of stainless steel, is used to attach the boron nitride cap to the stick.

samples that react with vanadium, a thin layer of a noble metal such as gold can be vacuum deposited inside a vanadium can to stop it from reacting with the sample (Turner *et al.*, 1999). If this approach is used, it should be remembered that the melting point of gold is 1337 K, and when it is irradiated by neutrons it becomes activated with a half-life of 2.7 days. For experiments requiring hydrogen pressure at elevated temperature, Inconel (Fig. 2.10.50*a*) is often the material of choice (Bailey *et al.*, 2004). However, in this case for diffraction measurements one has to either exclude the Inconel peaks or make use of radial collimators to reduce the signal from the vessel itself. In spallation sources with large detector area coverage one can also do an experiment where only detectors at a scattering angle of 90° are used. For gas-absorption experiments at low temperatures, however, vanadium is still the material of choice. For pressure measurement in anvil-type cells, TiZr is used for the gasket.

For opposed-anvil pressure experiments the anvil materials can be either cubic tungsten carbide or boron nitride. The latter is preferred as boron is highly absorbing and does not contribute anvil reflections to the sample measurement, so therefore effectively works as an incident-beam collimator. The use of tungsten carbide is reserved for techniques where a 'through-gasket' approach is required, such as furnace measurements with a graphite heater where the use of a null scattering alloy as a gasket material is not possible. A recent development in high-pressure neutron scattering is the use of sintered diamond anvils, also called PCDs (from polycrystalline diamond). They allow the accessible pressure range to be doubled at the cost of adding very strong diamond reflections to the pattern.

However, for gas pressure cells aluminium is often used, as it can withstand higher pressure and Al absorbs neutrons only weakly as the absorption cross section of aluminium, $\sigma_{abs}$ = 0.231 b for a wavelength of 1.8 Å, is small. For high-temperature gas-flow experiments fused silica ('quartz') glass is generally used for sample containment. These holders can also have glass frits attached at one or both ends for easy flow of gas through the sample, as shown in Fig. 2.10.51(*a*).

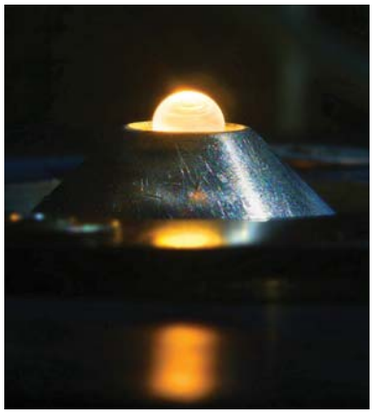

**Figure 2.10.52**
Aerodynamic levitation system to suspend melts at temperatures to 2773 K and beyond for neutron diffraction measurements.

A few very high intensity instruments are now able to carry out powder diffraction from milligramme quantities of sample. For these measurements vanadium cans produce too much background, as there is more vanadium in the beam than the sample. The use of thin-walled silica/glass or Kapton capillaries maybe more appropriate in those circumstances.

It is also important to remember that an exchange medium is used for low-temperature (heat transfer) and pressure (pressure transfer) measurements. Helium gas is generally used as a low-temperature exchange medium. Typically, cans are sealed with a flange and lid that supports an indium (or other soft metal) gasket. If the sample is air sensitive and has to be loaded in a glove box, one should try to use a helium-filled glove box. Argon- or nitrogen-filled glove boxes are more common but the freezing temperatures of argon and nitrogen are 84 K and 77 K, respectively. They will no longer work as exchange gases below these temperatures and, because of their rather large neutron-scattering lengths, new diffraction peaks will emerge at or below these temperatures. Similarly one should ensure that a pressure medium will remain hydrostatic for the pressure range for which it is being considered (Varga *et al.*, 2003).

It is also worth noting that cooling powder samples below 1 K relies entirely on thermal conduction through the walls of the sample holder and to the specimen itself. If great care is not taken, the specimen temperature may be far higher than that reported by a thermometer attached to the sample holder. At a minimum, the holder lid should be made from copper, as it is expected that the superconducting transitions in aluminium and vanadium would cause the walls of the sample holder to become thermally insulating and greatly reduce their ability to cool the sample. Of course, properly sealing the loose powder under an atmosphere of $^4$He is equally important. It is essential that the indium seal be installed correctly, as $^4$He undergoes a transition to a superfluid at 2.17 K and has effectively zero viscosity, and can easily escape from a poorly sealed can.

Levitation methods (*e.g.* gas flow, acoustic, electrostatic) as shown in Fig. 2.10.52 offer a containerless method, which eliminates altogether sample–container reaction problems and diffraction or additional background scattering from a sample container (Weber *et al.*, 2014). Levitated samples are typically used in conjunction with laser heating to achieve high temperatures, *in situ* melting of samples and prevention of heterogeneous nucleation.