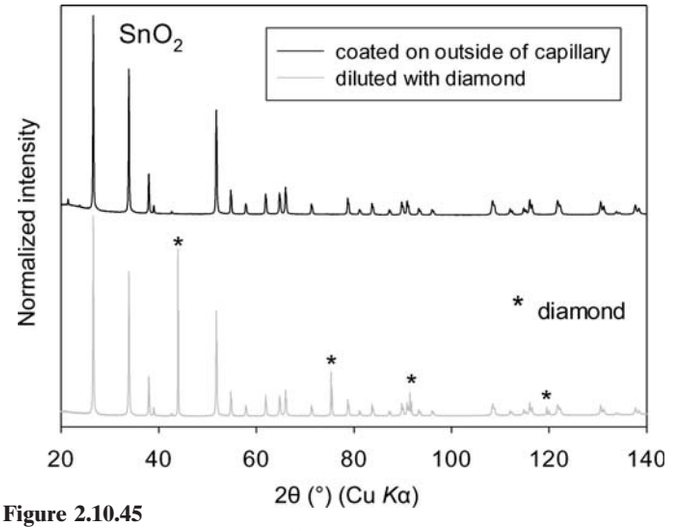

Figure 2.10.45
Comparison of data from SnO_2 when diluted with diamond inside a 0.3 mm capillary and pure SnO_2 coated on the outside of a 0.3 mm capillary.

Figure 2.10.47
Platform and pin mounts for capillary samples.

Figure 2.10.48
A 0.5mm capillary secured into a standard brass capillary pin using dental wax at both ends of the pin.

diameter capillary than optimal to retain reasonable resolution. Consequently, with organic samples where capillaries of 0.8 mm diameter are commonly used, it is highly recommended that an instrument with a primary focusing monochromator (or mirror) is used; the focus should be at the detector. Where the diffractometer is θ–θ geometry it is best to still collect capillary data as if it were a θ–2θ Debye–Scherrer instrument, simply by collecting 'detector scans' or the equivalent in the data-collection software. This has no effect on the data in a perfect situation, but it means that the sample illumination is constant over all diffracting angles even if there is a misalignment of the primary beam with respect to the capillary axis (caused either by misaligned optics, a misaligned capillary stage, or both). In addition, a correction for capillary displacement can be applied to data collected in conventional Debye–Scherrer geometry (Klug & Alexander, 1954) as the x and y displacements relative to the incident beam are constant over all 2θ angles.

Polymer capillaries are becoming increasingly common and are the standard at many synchrotron beamlines. They are easy to seal, but the lack of a funnel can make smaller sizes trickier to fill. A number of polymers can be used for capillaries, *e.g.* Mylar [poly(ethylene terephthalate) – PET] and Kapton [poly(oxydiphenylene pyromellitimide)]. The background from the capillary material itself is often more noticeable with a laboratory diffractometer than for higher-energy synchrotron instruments. A comparison of the background with a Cu $K\alpha$ focusing mirror laboratory diffractometer from 0.5 mm quartz, soda lime glass, PET and polyimide capillaries is shown in Fig. 2.10.46. A study of the different options for polymer capillaries in the laboratory environment was published by Reibenspies & Bhuvanesh (2006), which highlighted the awkward reflection with polyimide visible just above 5° 2θ in Fig. 2.10.46. It is also worth noting that the walls of polymer capillaries are not as stiff as those of quartz capillaries. If a low-temperature or other experiment might produce an internal vacuum (*i.e.* freezing a liquid sample), a polymer capillary can deform from a perfect cylinder, which may cause problems.

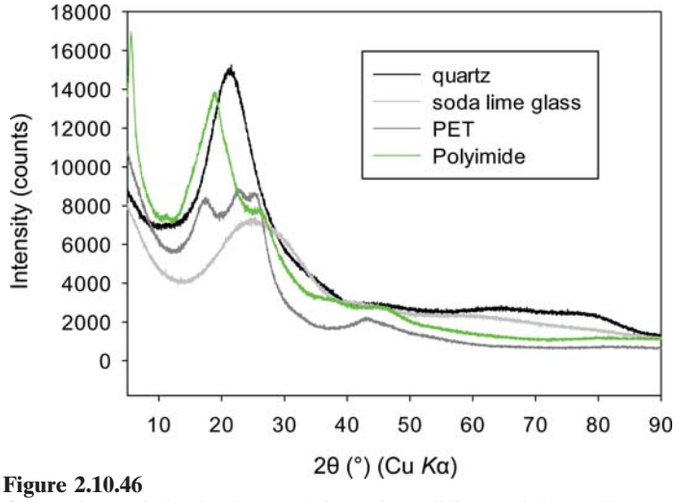

Figure 2.10.46
Comparison of the background from four different 0.5 mm-diameter capillaries. The quartz and glass capillaries are commercial capillaries for diffraction analysis. PET and Kapton capillary tubing are available from a number of different suppliers and are not made specifically for diffraction.

Mounting the filled capillary on the goniometer head can be achieved in different ways. Most commonly a hollow brass pin is used, but flat platforms are available (Fig. 2.10.47). The various pins/platforms are a standard size, so they should fit no matter where they are sourced from. The flat platforms have a hole in the middle, but it is only suitable for inserting small-diameter capillaries. Large-diameter capillaries must be affixed to the platform surface with wax and are vulnerable to sagging with horizontal goniometers because of the lack of support. The brass pins will accept larger capillaries and are to be preferred with respect to improved support for the capillary where the capillary is held at both ends of the brass pin (Fig. 2.10.48). Fixing the capillary onto the base is often done using wax or clay, although epoxy may be preferable if elevated temperatures are to be used. Coarse alignment is usually performed using a small desktop microscope before final alignment on the system. It is important to try to get the capillary rotating as straight as possible before mounting on the system, as removing tilt errors is much more difficult with the higher-magnification alignment scope mounted on the goniometer. Final alignment of a capillary is an exercise requiring patience. Never try to align out errors in two directions at once. Even if repeated attempts are necessary to stop the goniometer head in the correct position (Fig. 2.10.49), only correct errors perpendicular to the view in the scope. Ideally, the final alignment should only require correction of a side-to-side