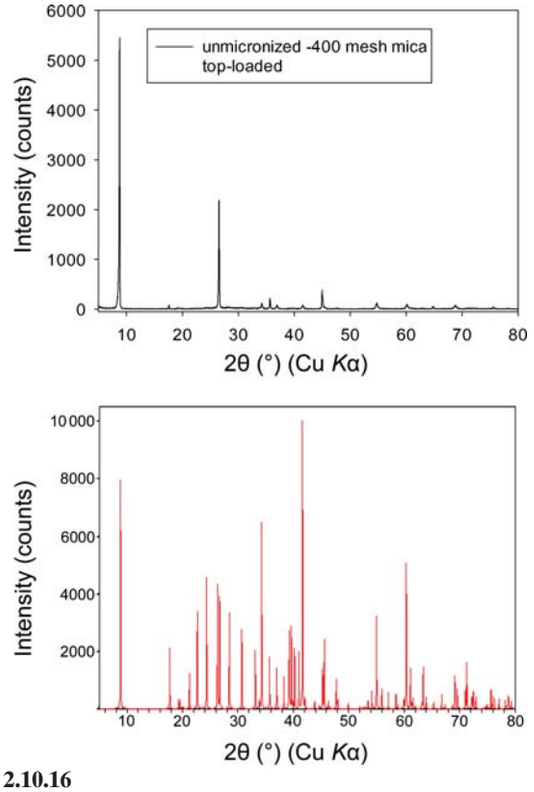

Figure 2.10.16
Top: diffraction pattern of top-loaded 400 mesh phlogopite mica. Bottom: calculated random pattern.

significant compression, yet remains flat. An example of a commercial back-loading holder is shown in Fig. 2.10.34. These holders are filled while upside down with the back removed. The cavity is filled with sample using minimal pressing, the back of the holder is replaced, and then the whole assembly including specimen is flipped the right way up. Generally, the deeper the holder the lower the compressive force on the analysed surface, but the trade-off is the requirement for large amounts of sample. Many of the samples exhibiting plate-like morphology possess low-angle reflections (such as mica and illite) so the sample area cannot be reduced too much to reduce sample volume, or beam overspill may occur.

A variation of the back-loading sample holder is the side-loading sample holder. These are less common, although the sample is still loaded against some surface in the same fashion as the back-loading variant. As the name implies, the difference is that the sample is introduced from a hole in the side as opposed to the back, and the hole is then plugged after filling.

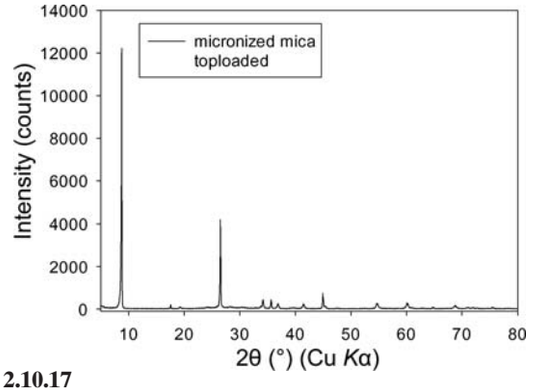

Figure 2.10.17
Diffraction pattern of top-loaded miconized phlogopite mica.

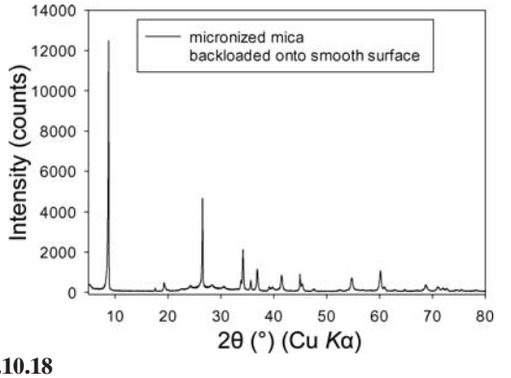

Figure 2.10.18
Diffraction pattern of miconized phlogopite mica when back-loaded onto a smooth surface.

Simple back-loading of samples in itself is not always sufficient for very platy samples such as the high-aspect-ratio mica used here. Fig. 2.10.18 shows the result from back-loading a micronized sample of the mica onto a smooth surface.

Although the result is improved, the specimen is still not a random powder. A useful approach in these circumstances is to make the surface of a back-loaded sample deliberately rough to break up the orientation of the plates. An easy way to achieve this is to load the sample onto the surface of sandpaper or a coarse ground glass slide. Sandpaper has the advantage of being disposable so avoiding cross-contamination among samples. Not all sandpaper has the desired jagged surface, so it may be necessary to experiment to find the best. The paper used for the data shown here was a 400-grit carborundum paper, the surface morphology of which is shown in Fig. 2.10.19. The rough surface will cause some slight defocusing in a parafocusing setup and reduce the count rates somewhat, but in many cases the advantages outweigh the disadvantages.

The result of back-loading the micronized mica onto the 400-grit carborundum paper is shown in Fig. 2.10.20. The dominance of the 00*l* reflections is reduced even further than when mounted onto a smooth surface. The approach is simple enough that it is used routinely in at least one laboratory dealing with large numbers of mining and mineral samples (Raudsepp, 2012). Back-loading samples is more time consuming than top-loading. Consequently, where high sample throughput is required, back-loading can be reserved for those samples where orientation is a problem.

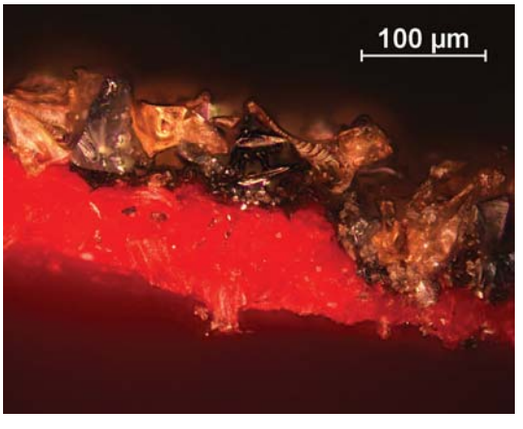

Figure 2.10.19
20× optical micrograph of a cross section of the 400-grit carborundum paper used for back-loaded mica.