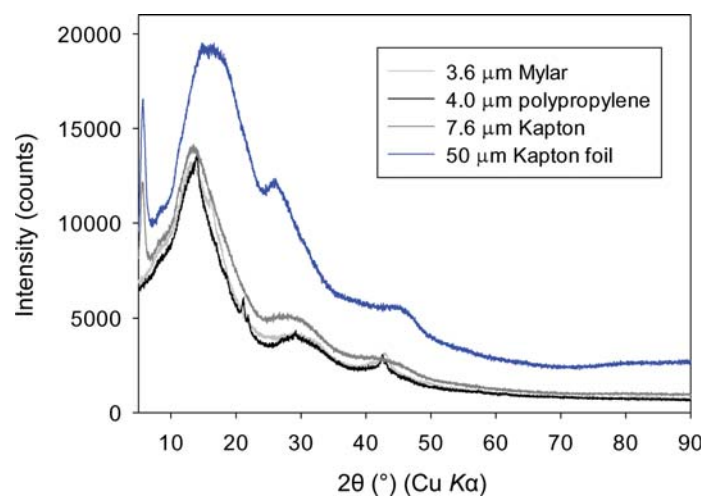


## 2. INSTRUMENTATION AND SAMPLE PREPARATION

**Figure 2.10.37**

Parts prior to assembly of a transmission foil sample in the holder. In this instance, micronized quartz is held as a loose powder between two 50  $\mu\text{m}$  Kapton foils while the upper foil is stretched into place by the black clip.

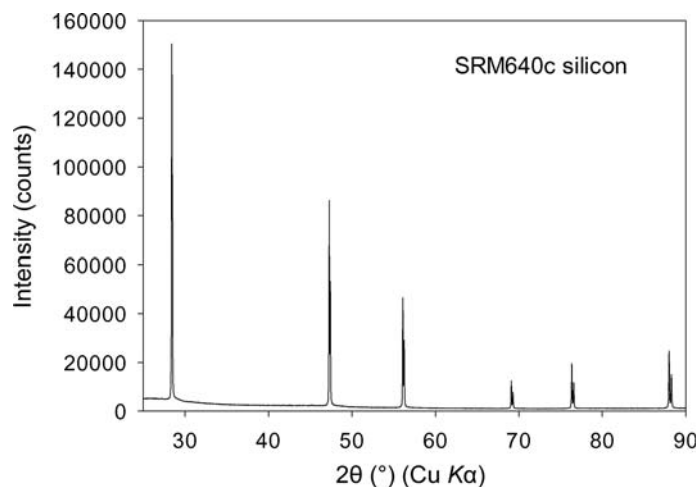
**Figure 2.10.38**

Transmission data from double layers (as used for powder samples) of different polymer substrate films. They include 3.6  $\mu\text{m}$  Mylar, 4.0  $\mu\text{m}$  polypropylene and 7.6  $\mu\text{m}$  XRF films, and a thicker 50  $\mu\text{m}$  Kapton foil.

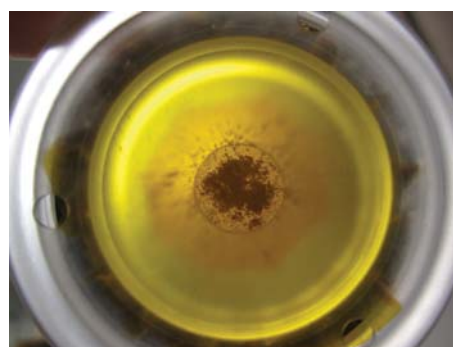
for reflection geometry, there are a number of ways to prepare the thin layer required. Loose powders may be trapped between two foils as in Fig. 2.10.39, or alternatively a slurry or smear mount may be used in a similar way to reflection geometry. Although the sample may adhere sufficiently such that a single foil can be used, it may be necessary to use a sandwich in the same way as a loose powder. For instance, slurries do not usually adhere well to Kapton foils, so it is often better to sacrifice a little intensity from the additional Kapton attenuation and ensure the sample does not fall away during data collection. Lack of adhesion could be regarded as an advantage with regards to recovery of valuable samples. Where an adhesive is used, the same considerations as with a smear mount in reflection still apply with regards to background *etc.*

Ideally the sample thickness should be perfectly uniform, but in practice this will rarely be achieved. Commonly a specimen in visible light transmission will appear something like that seen in Fig. 2.10.40. Rotation is used to average out inhomogeneity in the specimen.

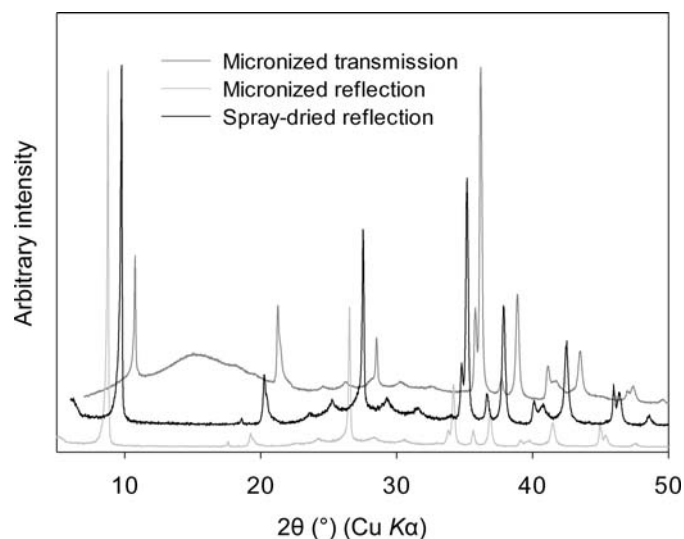
Sedimentation during slurry mounting and compression of powders between two foils can lead to preferential orientation

**Figure 2.10.39**

Diffraction pattern from loose SRM640c powder between two 50  $\mu\text{m}$  Kapton foils.

**Figure 2.10.40**

Transmitted light view of a micronized quartz sample through 50  $\mu\text{m}$  Kapton foils.

**Figure 2.10.41**

Comparison of data from micronized 40S mica taken in reflection and transmission geometry, and spray-dried material in reflection geometry. For improved clarity the spray-dried and transmission data sets are translated by  $+1^\circ$  and  $+2^\circ$   $2\theta$  respectively.

in foil transmission samples just as with flat-plate reflection specimens. Although the physical effect is the same for plate-like crystallites, it should be remembered that the crystallite orientation with respect to the beam is rotated by  $90^\circ$ , so the