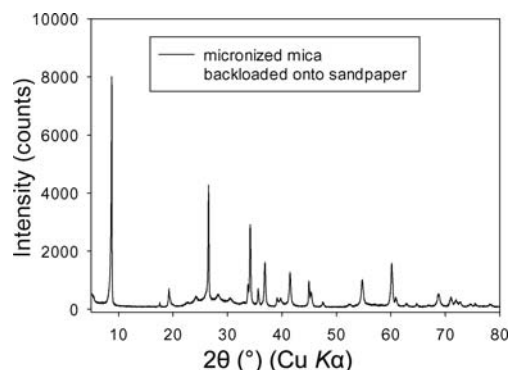
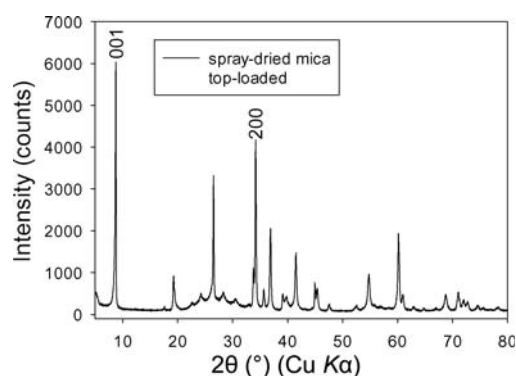


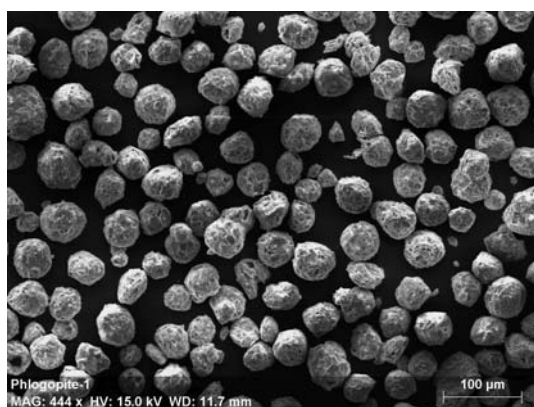
## 2. INSTRUMENTATION AND SAMPLE PREPARATION

**Figure 2.10.20**

Diffraction pattern of micronized phlogopite mica when back-loaded onto 400-grit carborundum paper.

**Figure 2.10.21**

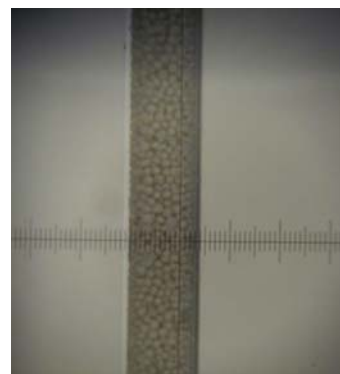
Diffraction pattern of top-loaded spray-dried phlogopite mica. The sample was not pressed; instead, a flat surface was produced by lightly scraping off excess material with a microspatula.

**Figure 2.10.22**

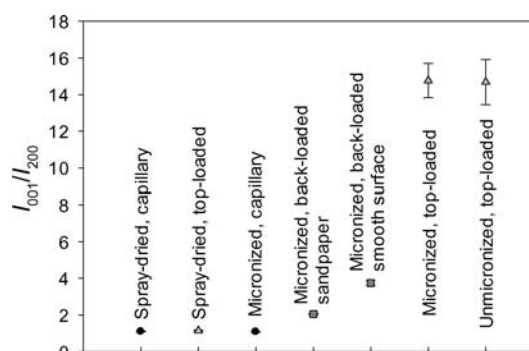
SEM micrograph of spray-dried micronized phlogopite mica (courtesy of M. Raudsepp, University of British Columbia).

Without resorting to transmission measurements, preferred orientation from platy samples may be almost, if not completely, eliminated by spray drying micronized samples (Hillier, 1999, 2002; see Fig. 2.10.21). This process produces spherical agglomerates (Fig. 2.10.22) that have no tendency to orient if handled gently. The disadvantage is that a relatively large amount of sample is often required because of inefficient sample recovery. Equipment optimized to reduce sample loss for spray-dried XRD samples may be bought in kit form (<http://www.claysandminerals.com/spraydrykit>), or constructed in house using a small air-brush and heated oven.

One potential practical problem when using spray-dried material with  $\theta$ - $2\theta$  geometry instruments is that the spherical

**Figure 2.10.23**

View through the alignment scope of the spherical spray-dried mica inside a 0.5 mm capillary.

**Figure 2.10.24**

Plot of the ratio of the integrated intensities of the 001/200 reflections of the mica using different sample-preparation techniques.

**Figure 2.10.25**

SEM micrograph of wollastonite needles.

particles can start to roll out of the specimen holder at higher  $2\theta$  angles (Raudsepp, 2012). The effectiveness of spray drying can be seen as the relative intensities from the top-loaded spray-dried material are almost identical to those in data obtained from the capillary experiments. The spray-dried spheres are very delicate and pressing of the sample must be avoided where possible.

It is worth noting that the platy nature of this mica was so extreme that the micronized mica tended to orient slightly inside the capillary if too much energy was applied during the filling process (e.g. using ultrasonics). Arguably, a capillary measurement using a spray-dried material is the ultimate precaution against preferred orientation effects, and the excellent flow characteristics of the spheres mean that the agglomerates remain