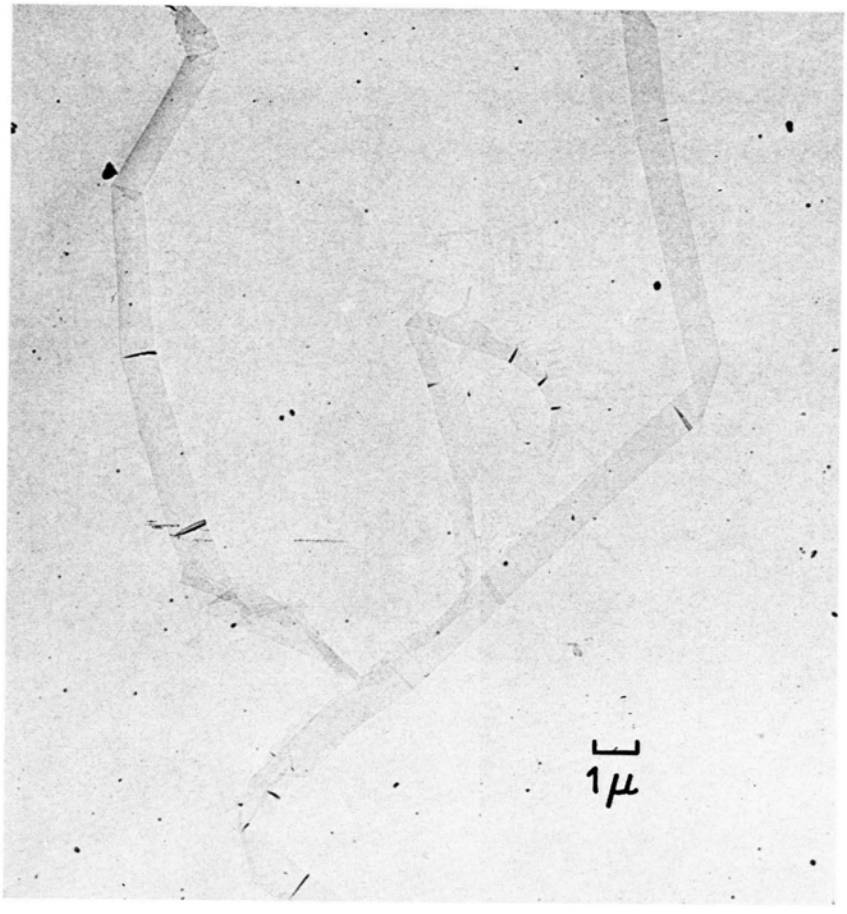

Fig. 1. Electron shadow image of rectorite crystals.

Fig. 2. Shadow image of part of a rectorite crystal and its diffraction pattern. The bright spot locates the diffracting area exactly.

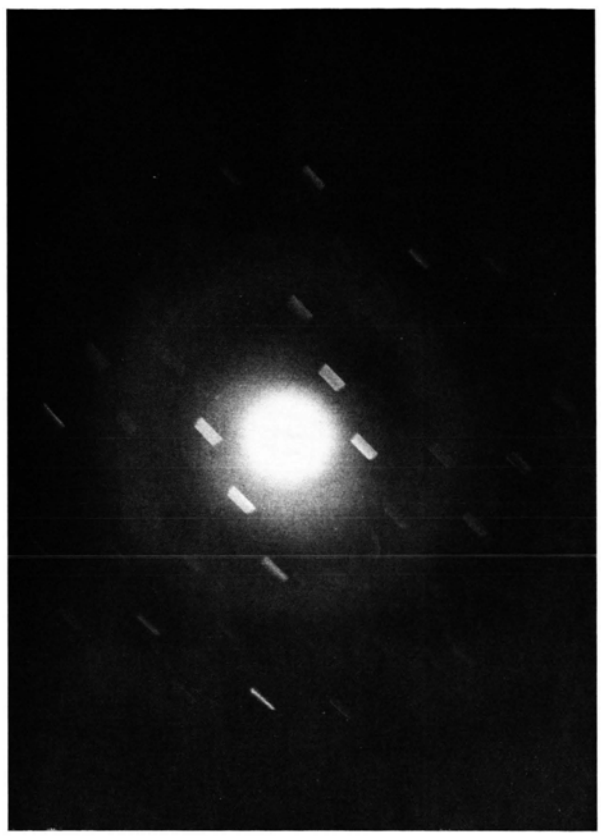

Fig. 3. Low-magnification multiple images from which the relative orientation of crystal edges and axes can be determined.

Selected-Area Diffraction in the Shadow Electron Microscope

W. C. T. Dowell

Division of Chemical Physics, CSIRO, Clayton, Victoria, Australia *

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A method of performing selected-area diffraction in the shadow electron microscope is described. The procedure is simple and the diffracting region is accurately located.

Methods of using the facilities of either conventional or scanning transmission electron microscopes for selected-area electron diffraction are well established [1–7] but to date no report has appeared describing any way of using a shadow electron microscope for this purpose. The method described here has both the virtues of being simple and of offering an unambiguous definition of the diffracting area of crystal. The diameter of this region is of the order of 100 nm.

The shadow microscope is operated as a defocussed convergent-beam electron diffraction camera with a very small angle of illumination so that the recorded pattern is a zero-order image of the selected area of crystal surrounded by the excited diffracted orders.

Figure 1 ** shows a shadow image of ribbons of a very thin crystal of the clay rectorite recorded without a defining aperture. If a defining aperture of several hundred microns diameter is used in order to somewhat limit the irradiated area of crystal, an image of small extent can be recorded near one edge of a photographic plate or film. In our instrument, a converted Elmiskop 1 electron microscope, this is achieved by racking the plate forward just sufficiently to allow part of it to intercept the direct beam. A very small aperture, 3 to 10 μ in diameter, is then substituted for the larger defining aperture and its image superimposed on the preceding image. The photographic plate is then racked fully forward and the diffraction pattern recorded. If the source, the crystal and the small defining aperture remain stationary, the diffracting area of crystal is exactly defined. Figure 2 shows the projected image and the diffraction pattern as they appear together on the photographic plate. The bright spot in the circle outlined by the smaller defining aperture shows the area of crystal generating the diffraction pattern.

Generally one uses the underfocus mode, which provides sharper images. The plane of the convergent-beam source is then between the crystal and the recording plane. The image of the crystal is inverted in this case, but the diffraction pattern is not rotated. Low-magnification images formed by various diffracted beams can, as in Fig. 3, which is also a pattern taken from rectorite, show the relative orientation of crystal axes and edges. At very high magnifications, however, the crystal is situated near the caustic of the objective lens. In this case the projected image but not the diffraction pattern suffers rotation and distortion.

When the crystal is within the caustic, the area irradiated through a small defining aperture is about 2 nm in diameter [8] in our instrument, when it is focussed for convergent-beam electron diffraction [9]. If the electron-optical system is well centred, this small diffracting region can be located with good accuracy by using subsequent shadow micrographs taken at reduced lens excitation. This latter method is indirect, however, and cannot be described as selected-area diffraction.

* P.O. Box 160, Clayton, Victoria, Australia 3168. Reprint requests to Dr. W. C. T. Dowell, CSIRO, Division of Chemical Physics, P.O. Box 160, *Clayton/Victoria*, Australien 3168.

** Figures 1–3 on page 1434 a, b.

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