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MOUNTING METHODS FOR MINERAL GRAINS TO BE EXAMINED BY HIGH RESOLUTION ELECTRON MICROSCOPY

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ABSTRACT

Small mineral grains (~0.1–5 μm diameter) are given mechanical and electrical stability by embedding them in an evaporated carbon film. Larger grains (up to ~1 mm diameter) are embedded in a matrix of fine aluminum oxide powder bonded with epoxy resin and thinned by grinding followed by sputter-etching.

This note describes preparation methods which have proved extremely valuable in electron microscope studies of lunar fines in the size range 0.1 μm to 1 mm diameter. The procedures are applicable to all particulate materials with low solubilities in water and organic media. The examination of mineral grains in transmission has been greatly facilitated by the advent of the sputter-etching method, (Tighe, 1968 and Barber, 1970), and high voltage electron microscopes. However, the advantages of applying high resolution and selected area diffraction methods to minerals (McConnell, 1967) may be lost unless: (a) small grains can be held firmly and prevented from electrical charging in the electron beam; (b) large grains can be supported throughout their thinning by sputter-etching and subsequently made to meet conditions (a). Ways of achieving these ends will now be described.

(a) It is common practice to sandwich insulating particles between two evaporated carbon films affixed to support grids. Sometimes this is adequate but it provides less stability with equiaxed particles than with lamellae. Potential problems are avoided by embedding grains of diameter ~0.1 to 5 μm within a single carbon film. The fragments are scattered on a clean glass microscope slide which is carbon coated in an evaporator. It is desirable to place the carbon source to one side of the slide and rotate the latter during evaporation so that particles are 'shadowed' from all sides and totally coated. The film, typically 500 to 1000 Å thick, is then stripped from the slide by flotation onto water in the conventional way, so carrying the grains with it. Pieces of film are picked up on 200 mesh grids. Apart from giving particle stability and ease of handling, the method has other advantages. The grains can be chemically treated since the carbon film is permeable but unreactive. If the particles dissolve, the carbon envelopes remain as a record of their origi-

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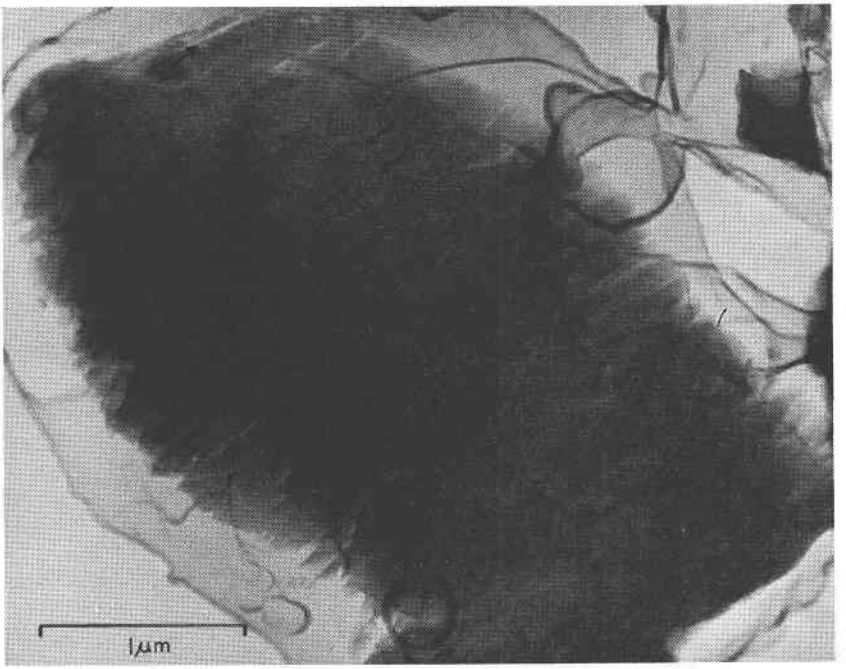

FIG. 1. Etched tracks of solar flare nuclei in a heavily etched (1 part HF:2 parts H_2SO_4:280 parts H_2O—1 minute) particle from the lunar soil encased in an evaporated carbon film. The ends of two of the tracks are arrowed. Note the carbon envelope showing the original shape of the particle and the earlier presence of two adhering smaller fragments of lunar dust.

nal sizes and shapes. Figure 1 shows a heavily etched particle from which approximately 0.2 μm has been removed, together with two smaller rounded particles. Because unetched lunar fines usually possess an amorphous skin, caused by solar wind irradiation (Dran *et al.* (1970), Barber *et al.* (1971)), the visibility of tracks by diffraction contrast in dark field is often poor. After sufficient etching to remove the amorphous layer but leave the tracks unetched, the track visibility is greatly improved. Using particle-bearing carbon films cemented to 200 mesh 'finder' grids, Barber *et al.* (1971) have been able to follow the progress of etching of solar flare particle tracks in lunar fines returned by the Apollo 11 and 12 missions. More recently, we have thus established that, with appropriate etching conditions, all of the tracks visible by diffraction contrast can be etched. Figure 2 shows a high density of finely etched tracks in a lunar pyroxene fragment.

(b) Since sputter-etching is best performed from two sides of a grain

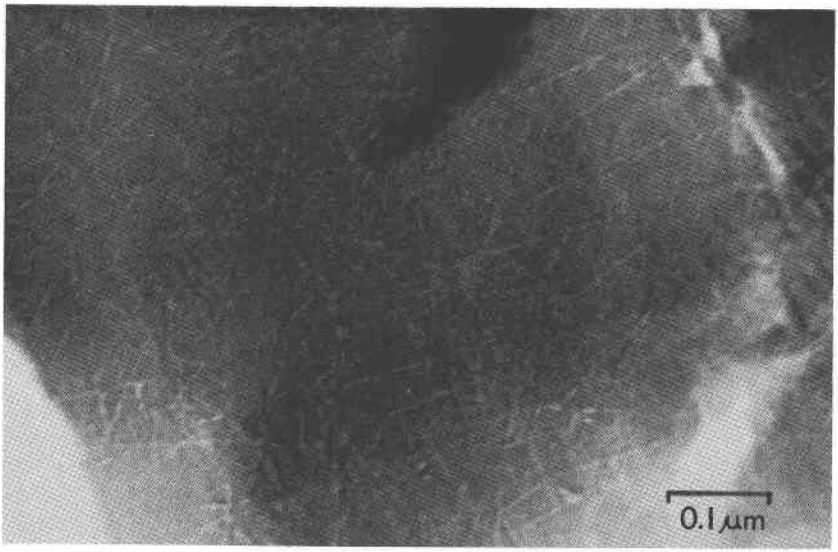

FIG. 2. Finely etched solar flare particle tracks (track density 6×10^{10}/cm^2) in a pyroxene fragment from the lunar soil.

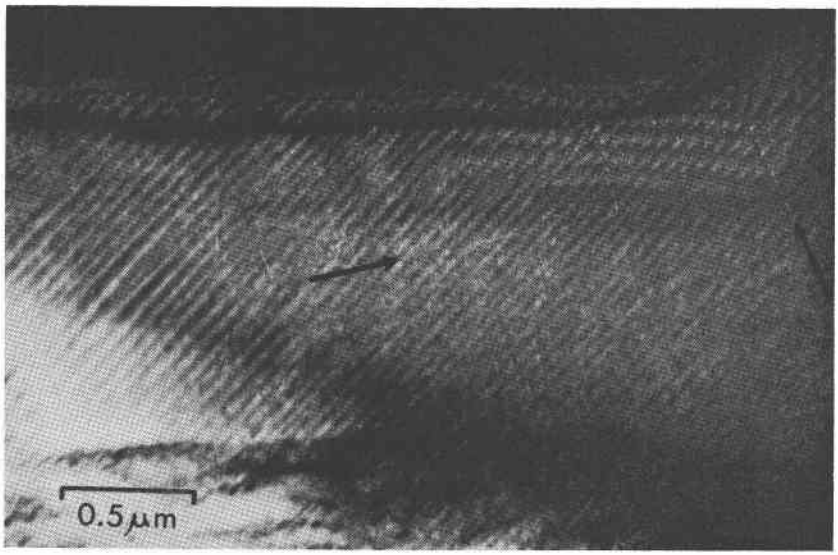

FIG. 3. Dark field micrograph of unetched solar flare particle tracks in a large (630 μm diameter) pyroxene fragment from the lunar soil which was sputter-etched after incorporation into a composite mount of epoxy resin and aluminum oxide powder. The exsolution lamellae in the pyroxene are seen as alternating black and white stripes. Two of the tracks are indicated by arrows.

simultaneously and minerals have been found to have low sputtering yields, the choice of a mounting medium for large grains is limited. Epoxy cements and plastics are not suitable because they sputter faster than minerals. For some purposes, it is feasible to use sealing glasses into which grains can be introduced by flash heating to ~500°C. But even if such heating can be tolerated, glass mounts are very brittle so that grinding, prior to ion-thinning, usually results in the loss of some samples. Moreover, the sputtering rates for glasses are not as low as those of the most resistant minerals, which include the pyroxenes.

A satisfactory mount has proved to be finely ground aluminum oxide packed tightly into low viscosity epoxy resin. Aluminum oxide has a sputtering yield less than most minerals, as shown by Wehner *et al.* (1963), and comparable to that of the clinopyroxenes for 6 kV argon ions. Thus, provided that the powder is closely compacted and in intimate contact with the sample grains, the composite mount will hold together down to thicknesses ~1 μm. The Spurr (1969) low viscosity diepoxide formulation has been used with 1 μm polishing alumina in work on lunar fines at Berkeley. Figure 3 is a dark field micrograph of tracks (unetched) near to the original surface of a 630 μm diameter particle in a sputter-etched composite mount.

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