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# Demountable polished extra-thin sections and their use in transmission electron microscopy

THE advantages of polished ultra-thin sections (PUTS) in the study of very fine-grained materials, such as occur in some meteorites, have been illustrated by Fredriksson *et al.* (1978) whose technique is based on the earlier work of Beauchamp and Williford (1974). An essential feature of such methods for friable and heterogeneous materials is the use of a medium, usually an epoxy resin, to consolidate and partially impregnate them. Normally one polished side of the specimen is bonded to a glass slide during preparation, and the finished PUTS are integral with the slide on completion. PUTS are typically 2–5 microns in thickness.

Transmission electron microscopy (TEM) is now frequently used in the study of minerals, and although it has numerous potential applications in the earth sciences (Champness, 1977), it is particularly valuable for studying very fine-grained assemblages, with features below the limits of optical resolution. For TEM the thickness of specimens must be reduced to about 0.1–0.3 microns and this is now routinely performed for minerals by ion-beam or sputter-thinning (Barber, 1970), usually starting from sections about 30–50 microns thick, depending on their friability. Standard petrographic thin sections can be used provided they were originally bonded to glass slides with a soluble cement.

Because ion-beam thinning produces surface relief (minerals and other solids exhibit a range of sputtering rates), there are great advantages to be gained from minimizing the amount of material to be removed by this process. PUTS would be ideal starting material, therefore, and they would have the additional merit that the comparison of light-optical and electron-optical observations would be greatly facilitated. Acknowledging this fact, but accepting that PUTS, as prepared by Fredriksson *et al.* (1978), cannot be removed from the supporting glass slide for ion-thinning, a technique has been evolved for the preparation of demountable thin sections much less than 30 microns thick. For meteorites, which have been the materials of chief concern, the final thicknesses are typically 5–8 microns and the sections are hereafter referred to as DETS (demountable extra-thin sections). With very dense, strong aggregates (ceramic oxides, for example), DETS can probably be reduced to slightly less than 5 microns in thickness but the limitation is, in general, more a function of lack of rigidity of soluble mounting media than of the specimen material.

*Method.* Stages in the preparation of DETS from C2M meteorites, the specimens being entirely hand-held and without using rotating laps, are as follows:

(i) A thin-bladed diamond saw is used to cut ~0.5-mm sections from an epoxy-encapsulated fragment.

(ii) One side of a cut section is lightly abraded on grade 0 polishing paper (dry) to ensure flatness. This surface is then thinly coated with a low-viscosity epoxy resin (Ciba Araldite formulations MY750/HY905, AY18/HZ18, two-tube mix warmed to ~70 °C, and rapid-setting Devcon '2 ton' clear cement have been used) and subjected to vacuum to ensure the removal of bubbles and some impregnation.

(iii) The coated surface is then restored to flatness on grade 0 paper (dry) and then further abraded to remove the bulk of the epoxy cement. Small sections can be worked without mounting but larger ones are bonded to a ground 1-inch-diameter glass disc with Crystalbond.

(iv) The same surface is polished sequentially on 10-, 3-, and ¼-micron diamond compound, diluted with white spirit, on fine-quality brown wrapping (craft) paper (with acknowledgements to the late Grover Moreland, private communication, and Moreland and Johnson, 1977).

(v) The singly polished section, which should show very little relief or plucking, is detached from the glass disc by warming it to 140 °C and the polished face is rebonded (excluding all bubbles and surplus mounting medium) to a new disc with 'Crystalbond).

(vi) Steps (ii) to (iv) are applied to the second cut surface until a section of suitable thickness is obtained. This normally entails grinding on grade 0 paper until the section has a thickness of about 15 microns, and more than one application of low-viscosity epoxy resin, with vacuum impregnation, may be needed *en route*.

(vii) Detailed optical work on the DETS is possible at this stage and it may be advantageous to do this before proceeding to the next stage.

(viii) TEM 3-mm-diameter grids are cemented to selected parts of the DETS with Araldite (household type) two-pack epoxy. Coarse-mesh thick copper grids are usually best as they offer maximum support compatible with minimum coverage of chosen area (e.g. Pelco 7GC HEX, with seven continuous holes and a wide rim). A thin coat of cement is applied to the grids, which are positioned face down on the DETS with the aid of a

low-power stereomicroscope. Setting occurs overnight, thereby fixing the DETS to the grids.

(ix) The assemblage is warmed to 140 °C and the DETS, with the grids serving to strengthen it, is slid off the glass disc. Unsupported parts will almost certainly disintegrate at this juncture but the grid-supported areas will remain largely intact. These are washed in acetone for about 1 minute to remove any trace of Crystalbond, but this time is insufficient to cause softening or swelling of the epoxy cement.

(x) The mounted DETS can now be separated and ion-thinned in the normal way.

(Note that at various of the above stages, where absolute freedom from adherent abrasive materials is necessary, the specimen surface should be cleaned with a jet of clean, dry, compressed gas.)

*Results.* The advantages of the technique are not easy to illustrate briefly because they lie mainly in the improved correlation which can be consistently obtained between optical and TEM observations (as compared with use of ion-thinned 'thick' sections) and in the production of large electron-transparent areas, embracing interphase regions. Fig. 1*a* shows a transmission optical micrograph of a calcite grain (actually corresponding to a composition $Ca_{0.93}Fe_{0.07}CO_3$) in a DETS from the Murchison (C2M) meteorite, together with matrix phyllosilicate materials. The interference colour of the calcite (3rd-order blue/green) showed that the grain and its surrounding were approximately 7–8 $\mu$m thick. This DETS was subsequently ion-thinned and fig. 1*b* shows the internal structure of the same calcite grain, imaged at 1 MeV. The grain is multiply twinned on $\{01\bar{1}8\}$, the most common twin plane for calcite; some parts of it also contain numerous small ($\sim$0.1 $\mu$m diameter) rounded voids.

As an illustration of the large uniformly thin unbroken areas which can be produced from DETS, fig. 2 shows a fairly low-magnification HVEM image of Fe, Mg mixed phyllosilicates in an ion-thinned DETS from the Cold Bokkeveld (C2M) meteorite. The field shows a region where

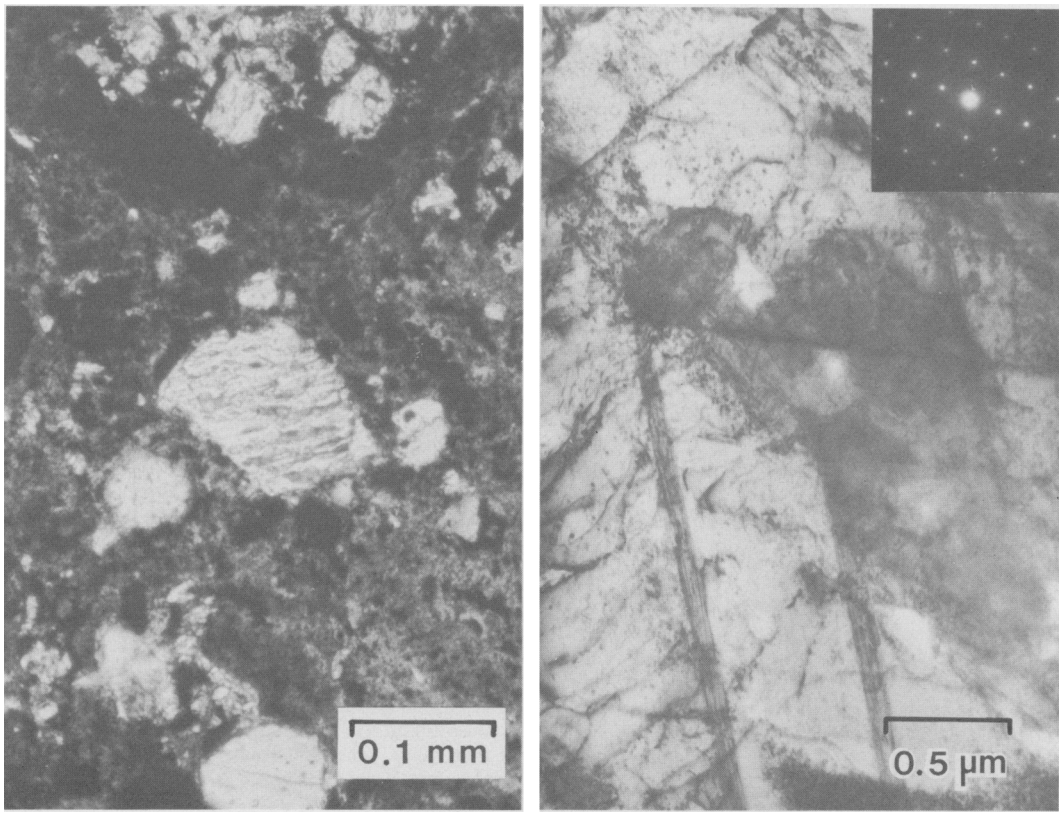


FIG. 1. (*a*), left: transmission optical micrograph (polarized light) of demounted extra-thin section of the Murchison meteorite, showing central calcite grain (twinned) of thickness 7–8 $\mu$m. (*b*), right: transmission electron micrograph (1 MV) showing twins, dislocations, and small dislocation loops within the calcite grain illustrated in *a*, together with zone axis selected area diffraction pattern.

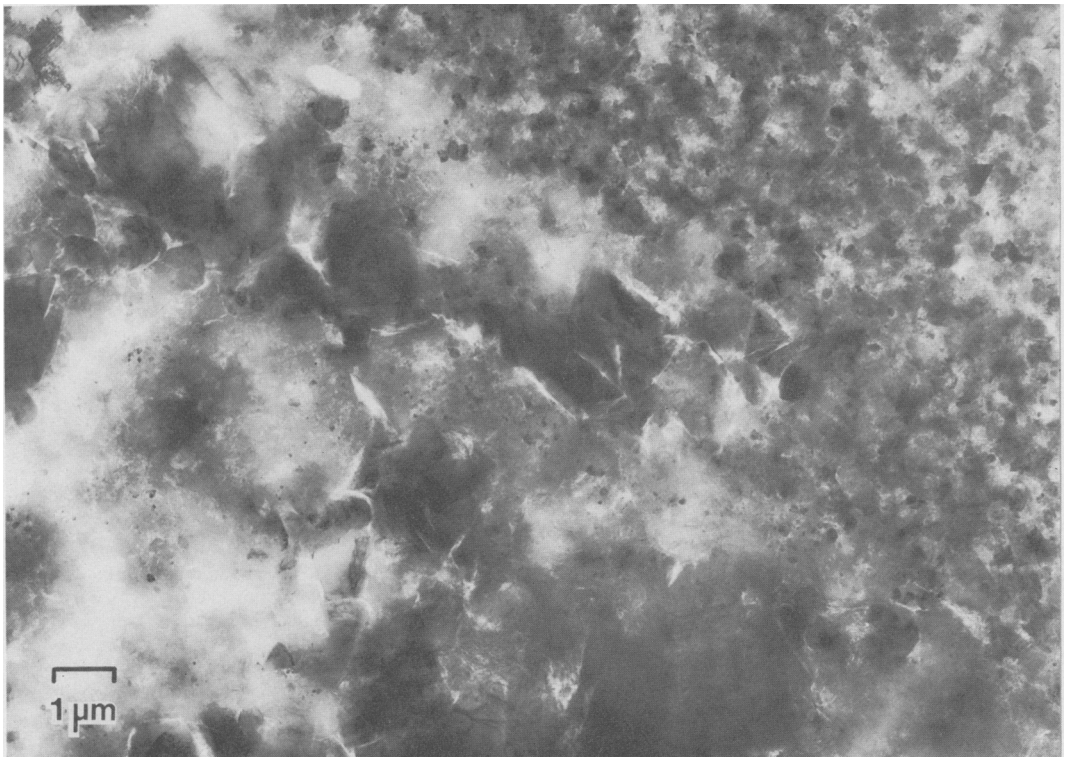


FIG. 2. Transmission electron micrograph (1 MV) of a region with variable grain size and mineralogy in the phyllosilicate matrix of the Cold Bokkeveld meteorite, initially prepared as a demountable extra-thin ($\sim 7$ $\mu$m) section.

the grain size and mineralogy changes significantly, which customarily leads to difficulties in obtaining good quality (i.e. fairly uniformly electron-transparent) ion-thinned specimens from sections of thickness $\gtrsim 30$ $\mu$m.

It should be apparent from these illustrations that the technique for making DETS could have considerable applications in other fields where the close correlation of light- and electron-optical methods would be advantageous, especially where grain sizes approach the limits of optical resolution and the mineralogy is complex.

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