Cometary Particles: Thin Sectioning and Electron Beam Analysis

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Thin sections (500 to 1000 angstroms thick) of individual micrometeorites (5 to 15 micrometers) have been prepared with an ultramicrotome equipped with a diamond knife. Electron microscopic examination of these sections has revealed the internal structures of chondritic micrometeorites, and a subset of highly porous, fragile particles has been identified. Delicate meteoritic materials such as these are characteristic of debris from cometary meteors.

The nature of cometary material is of considerable importance because of the likelihood that comets contain a record of primordial processes that occurred in the outer regions of the solar nebula. Observations of meteors have shown that cometary debris comprises a significant fraction of the extraterrestrial material entering the atmosphere, but there are at present no established chemical, isotopic, or mineralogical criteria for distinguishing it from the abundant asteroidal debris also entering the atmosphere. The only known properties that could be useful for fingerprinting cometary debris are porosity and crushing strength, since studies of the atmospheric fragmentation of cometary meteors have shown that they are predominantly porous, fragile objects (*1*). In fact, cometary particles exhibit a range of strengths; an extreme example is the Draconid meteors from comet Giacobini-Zinner. Draconids fragment in the atmosphere when aerodynamic ram pressure is in the range 1000 to 5000 dyne cm^{-2} (*2*). (A 10-cm cube of Draconid material resting on a flat surface would be crushed by its own weight.)

The porosity of cometary material is presumably related to the loss of volatile ices. All conventional meteorites that have been recovered are rather strong, compact rocks, and these are most likely asteroidal material (*3*). Typical cometary meteoroids, on the other hand, are too fragile to survive atmospheric entry and to be recovered as even small meteorites. This strength selection effect is severe for centimeter-sized objects but is relatively minor for 10-μm particles, which are subjected to ram pressures of only 100 dyne cm^{-2} during atmospheric entry (*2*). Therefore, cometary material is almost certainly represented among unmelted extraterrestrial particles (micrometeorites) that are now routinely collected from the stratosphere (at altitudes of 18 to 20 km) by NASA U-2 aircraft (*4*). Some of these micrometeorites should be identifiable as cometary debris on the basis of their high porosity and fragile structures.

Within the existing micrometeorite collection, there is a group of particles referred to as "chondritic." These are micrometeorites whose compositions agree to within a factor of 2 with those of group CI meteorites (*5*). Chondritic particles are strong candidates for cometary material because they exhibit evidence of primordial grain-forming processes (*6*). Superficially some of them also appear to be porous aggregates of micrometer- and submicrometer-sized grains (Fig. 1), and particle-weighing experiments have suggested that they are low-density objects (*7*). However, in order to determine the true porosity (and strength) of a particle, it is essential to know something about its internal structure. Until now almost all micrometeorites, out of necessity, have been crushed and dispersed before electron-beam analysis, and, as a result, much of the original structure, porosity, and textural relations were destroyed. We report here the application of ultramicrotomy to prepare thin sections of uncrushed particles. These sections have revealed the true structures of chondritic micrometeorites. This group includes a subset of highly porous, fragile particles that may be of cometary origin.

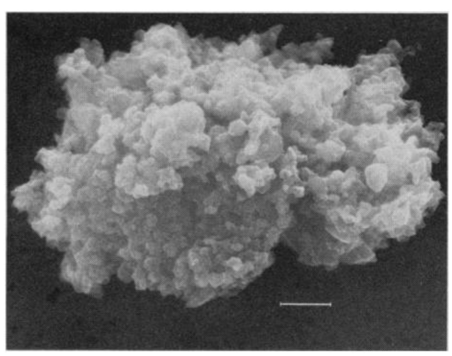

Fig. 1. Scanning electron micrograph of a typical chondritic micrometeorite recovered from the stratosphere. Scale bar, 1 μm.

In order to produce thin sections, it is necessary that each micrometeorite be mounted in a configuration suitable for ultramicrotomy (*8*). The particle is initially mounted in a low-viscosity epoxy, which, upon curing, forms a translucent bullet-shaped mount (~7 mm by 18 mm). The micrometeorite is embedded toward the tapered extremity of the mount, and several carbon fibers (~100 μm by 10 μm in diameter) are arranged symmetrically about the particle to highlight its position. A diamond knife is used to cut sections (500 to 1000 Å thick) from the epoxy mount; these sections are then floated away from the diamond edge (*9*). When viewed under a stereobinocular microscope, sections of interest contain a small black speck (the sectioned particle) symmetrically surrounded by dark lines (the sectioned carbon fibers). These sections are then transferred onto thin carbon (<200 Å) support films for characterization by analytical scanning transmission electron microscopy.

Of the ten micrometeorites that we sectioned by ultramicrotomy, all exhibited one or other of two distinct textural types in thin section. These two textural classes differ fundamentally in porosity and mineralogy. Five micrometeorites are highly porous aggregates of anhydrous phases, and the other five are compact assemblages of hydrated silicates. The hydrated type (Fig. 2a) is composed of poorly crystalline layer-lattice phyllosilicates that are rich in iron and magnesium. Using high-resolution lattice fringe imaging and x-ray (*10*) and electron diffraction, we found that in some particles the phyllosilicate basal spacing is 7.3 Å and in others it is 10 to 12 Å. The 7.3-Å spacing is consistent with serpentine-septachlorite minerals (*11*), whereas a 10- to 12-Å spacing is characteristic of both smectites and mica (*12*). Other mineral species, notably iron sulfides, are present only as minor constituents, and olivine and pyroxenes are usually rare. The exterior form of hydrous particles may vary from granular (Fig. 1) to smooth, but all the samples of this type that we sectioned have low-porosity interiors (Fig. 2a). We have not yet conducted detailed studies of sections of this particle type except to note distinct differences from its anhydrous counterpart and its mineralogical similarity to CI/CM meteorites.

Anhydrous chondritic particles exhibited both granular (Fig. 1) and fluffy exteriors, and all that were sectioned were found to be porous aggregates. These aggregates are complex mixtures of mineral grains and carbonaceous material (Fig. 2b). The mineral grains ranged in size from several micrometers to 100 Å. Magnesium-rich olivine and pyroxene and iron sulfides were the dominant species, together with lesser amounts of magnetite, iron-nickel alloy

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(kamacite), and iron-nickel carbides, although the relative abundances of mineral species varied widely from one micrometeorite to another. Glass was also a minor but important component of the anhydrous particles (Fig. 3a). No evidence for the presence of hydrated phases was found within this type of particle, and in all cases high porosity (>50 percent) persisted throughout the entire volume of the particles.

Textural relations (Fig. 2b) were indigenous to the parent micrometeorite and were largely undisturbed by the sectioning procedure. This has been confirmed by the fact that several times we noted the preservation of the orientation of surface grains that had been marked with a sputtered palladium film before ultramicrotomy (*13*). The internal structure of porous particles is a delicate assemblage of micrometer- and submicrometer-sized components. These components include (i) single mineral grains, (ii) clumps of grains (<1000 Å in diameter) in a carbonaceous matrix ("tar balls"), and (iii) carbon (Fig. 2b). The relative abundances of these three species may vary widely from one anhydrous micrometeorite to another. The components are often aggregated in approximately micrometer-sized clusters, which in the plane of section are not in direct contact with adjacent clusters (Fig. 2b). Most particles are highly heterogeneous and appear to be simply mechanical mixtures of the various components. Particle formation appears to have proceeded via at least two aggregation events. The first was the coagulation of "tar balls" composed of extremely small (0.01- to 0.1-μm) grains in carbon. The second event was the aggregation of roughly 0.1-μm clumps ("tar balls," carbon, and mineral grains) to form the complete particle. If the particles are from comets, then the open pore spaces presumably would have been filled with ices or other volatile components during the time of residence in the cometary body. Variation in the original dust-to-ice ratio would have led to a wide range of bulk densities even for particles as small as 10 μm. Whatever the origin of these particles, they must be very primitive because they were not subjected to the compaction process that produced the low porosity (10 to 15 percent by volume void space) observed in conventional chondritic meteorites (*3*).

The ability to produce thin sections of anhydrous particles has also made possible novel observations regarding the implantation of solar flare tracks in micrometeorites. Difficulties encountered earlier (*14*) when imaging tracks in unsectioned particles have largely been overcome, since tracks are easier to image in uniformly thick grains because of reduced artifacts. It is also possible to observe track density gradients in thin section; in one micrometeorite an olivine grain at the edge of a section exhibited a track density of 2×10^{11} cm^{-2}, whereas the observed density within an olivine located toward the center of the thin section (some 5 μm from the outer edge) was on the order of 5×10^{10} cm^{-2} (*15*). Some olivines and pyroxenes also have a distinct rim (~500 Å thick) along that portion of their edges that formed the outer surface of a parent micrometeorite (Fig. 3b). Similar rims seen on lunar dust grains have been attributed to solar wind irradiation (*16*).

All these track implantation phenomena confirm an important assumption made about micrometeorites, that before atmospheric entry they were exposed to the interplanetary medium as discrete small bodies rather than as pieces of much larger meteoroids. The observed track densities are consistent with exposure to contemporary solar cosmic rays for about 10^4 years (*14*). Future work on the sections may reveal whether the individual grains inside a single particle experienced significant exposure to solar or galactic cosmic rays before formation of the aggregates.

Thin sections provide an unambiguous and reliable method for classifying stratospheric micrometeorites with chondritic compositions. In the past all chondritic mi-

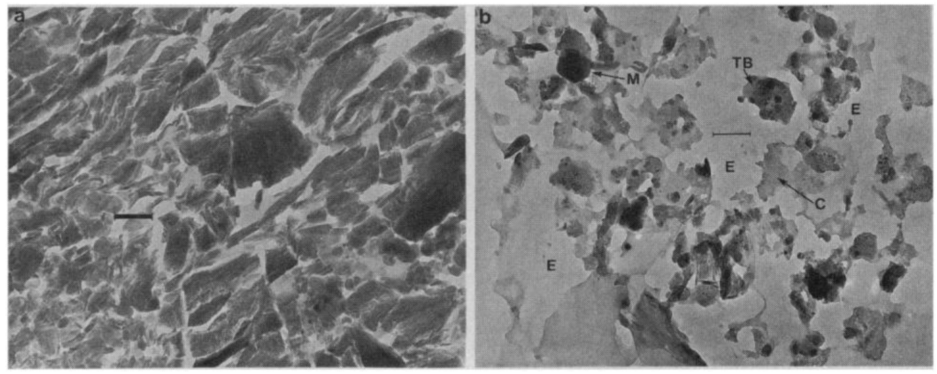

Fig. 2. High-magnification, bright-field electron micrographs of sectioned micrometeorites. (a) Hydrous type. Note the dominance of just one phase (phyllosilicate) and the low porosity. Some of this porosity may be an artifact of thin sectioning (*13*). (b) Anhydrous type. The three major components are carbon (C), single mineral grains (M), and "tar balls" (TB). The relative proportions of these components may vary widely; for example, this micrometeorite contains more carbon than other anhydrous micrometeorites. Epoxy-filled areas (labeled E) represent void spaces indigenous to the parent micrometeorite. Scale bar, 0.2 μm.

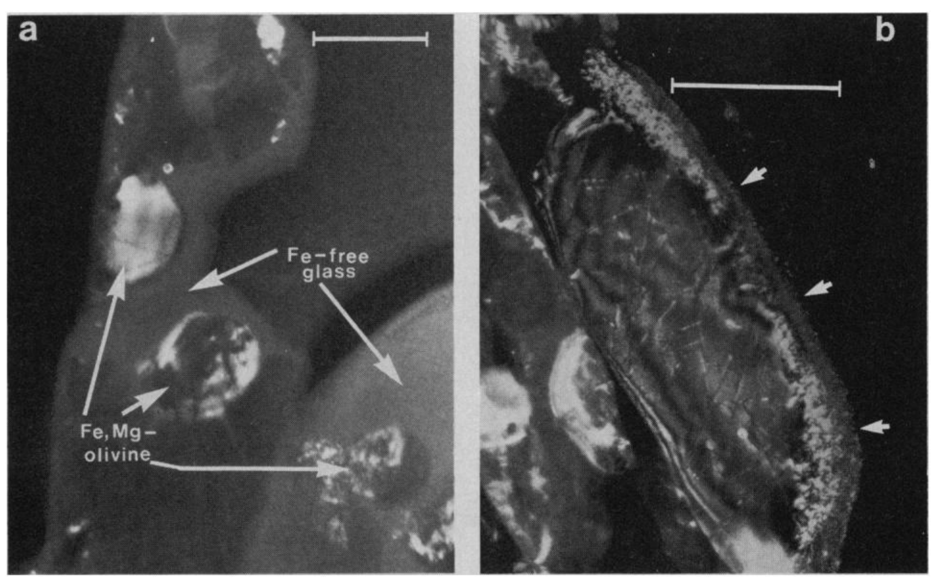

Fig. 3. Petrographic features of sectioned micrometeorites. (a) Dark-field electron micrograph of olivine crystals embedded in silicate glass. Note the presence of solar flare nuclear tracks in the uppermost crystal. (b) A track-rich olivine grain within a sectioned meteorite. The arrows indicate an outer surface of the olivine that was exposed directly to interplanetary space. Note the presence of an outer rim (arrowed) that probably resulted from irradiation by low-energy solar wind ions. Fragmentation of part of this olivine grain (left side of micrograph) is an artifact of the sectioning procedure (*13*). Scale bar, 0.1 μm.

crometeorites have been referred to collectively as chondritic aggregates, with further division into subgroups based either on particle appearance (*17*) or on the presence of characteristic infrared transmission absorption bands (*18*). Observations of morphology and surface microstructures made it possible to categorize the particles as CP (chondritic porous), CF (chondritic filled), or CS (chondritic smooth) (*17*). The major drawback with this type of scheme, however, is that not all micrometeorites fall clearly into a particular category. Although many micrometeorites with highly porous surface structures (CP's) are the anhydrous variety and most with smooth surfaces (CS) are hydrated, there have been numerous exceptions. For example, micrometeorites exhibiting highly porous surface structures have, upon crushing or sectioning, turned out to be solid and dominated by hydrated silicates. Using infrared spectral transmission measurements, Sandford and Walker (*18*) found that most chondritic micrometeorites fall into one of two categories: those whose mineralogy is dominated by anhydrous silicates (olivines and pyroxenes) and those dominated by hydrated (layer-lattice) silicates. Examination of microtomed sections confirms the validity of the infrared classification scheme, since chondritic particles do indeed fall clearly into hydrous and anhydrous subgroups. Although both types of particle can exhibit almost identical bulk compositions and outward appearances (Fig. 1), they are distinct from one another in thin section. Only the anhydrous variety exhibits high porosity typical of cometary meteoroids, although a lack of porosity does not necessarily preclude the possibility of a cometary origin for the hydrated particles (*1*).

Electron microscopic characterization of ultramicrotomed thin sections is a useful means of determining the true nature of small extraterrestrial particles. It is probably the ultimate method for studying fine-grained meteoritic materials that are mineralogically heterogeneous at the submicrometer level. In principle, it is possible to section a single 10-μm particle into 200 slices with almost complete preservation of internal microstructure in three dimensions. The present application has revealed a subset of chondritic particles that are without doubt the most porous meteoritic materials known. These aggregates of anhydrous silicates, sulfides, carbides, glass, and other phases are the only materials sufficiently fragile and porous to be considered similar to the typical cometary particles observed as meteors in the night sky. The structure of these particles closely matches Verniani's (1, p. 258) description of cometary meteors as "porous crumbly objects made of loosely conglomerate, spongelike material." Material similar to the sectioned anhydrous particles may be encountered as the spacecraft approaches comet Halley.

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9. Successful preparation of thin sections depends on the choice of embedding media, the properties of the micrometeorites themselves, and user experience with ultramicrotomy. Usually it is desirable to choose an embedding medium whose hardness closely approximates that of the material being sectioned. We use Embed 812, a low-viscosity epoxy that penetrates the micrometeorites well and forms a hard mount when cured. The carbon fibers we use are taken from commercially available graphite yarn. Diamond cutting edges and the ultramicrotome itself are standard sectioning equipment.
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13. Problems can arise with large (>2 μm) olivine and pyroxene grains (commonly found in anhydrous micrometeorites), which tend to fragment during sectioning (see Fig. 3b). This phenomenon, which is referred to as "chatter" (*8*), is often encountered during ultramicrotomy of materials that are very hard or that lack cleavage. Problems can also arise with materials that are very soft and that exhibit good cleavage. For example, hydrated (phyllosilicate) micrometeorites (Fig. 2a) respond well to diamond knife sectioning but the phyllosilicates themselves tend to cleave spontaneously in thin section. Therefore, some of the porosity observed in Fig. 2a may be an artifact of sample preparation. Despite these potential complications, examination of thin sections cut sequentially from micrometeorites suggests that the positions of most grains are relatively undisturbed by the sectioning procedure.
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15. When comparing measured track densities in unsectioned grains, one must make a grain volume correction, since larger grains may appear to contain higher track densities (per equivalent unit area) than smaller grains [see P. Fraundorf, G. J. Flynn, T. Shirck, R. M. Walker, *Proc. Lunar Planet Sci. Conf.* **11**, 1235 (1980)]. However, with thin sections all constituent grains are the same thickness, and therefore track density differences observed in olivines probably reflect actual differences in solar flare exposure.
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A Carbonate-Rich, Hydrated, Interplanetary Dust Particle: Possible Residue from Protostellar Clouds

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Transmission electron microscopy of a hydrated interplanetary dust particle (IDP) indicates that it contains abundant magnesium-iron carbonates, primarily breunnerite and magnesian siderite. This IDP displays a strong absorption band at 6.8 micrometers in its infrared spectrum, similar to that in certain protostellar spectra. The carbonates probably account for the 6.8-micrometer band in the IDP spectrum, suggesting that carbonate also may occur in interstellar dust and be the source of the controversial 6.8-micrometer feature from the protostellar spectra.

INTERSTELLAR DUST AND INTERPLANETARY dust particles are two types of fine-grained extraterrestrial materials whose character and origin are subjects of current interest and controversy. Interstellar dust contains relatively few distinctive spectral features that permit detailed characterization (*1*). Interplanetary dust particles (IDP's), a relatively recently discovered class of primitive solar-system materials, contain a limited range of minerals (*2*). There is an intriguing correspondence between the infrared (IR) spectra of the so-called hydrated IDP's and those of protostars like W33 A (*3, 4*), suggesting that at least some of the two types of materials may be similar mineralogically and that the hydrated IDP's may contain residual material from protostellar clouds.

One of the most prominent features in the spectra of both W33 A and hydrated IDP's is a strong absorption band at 6.8 μm. This band, when it occurs in interstellar spectra, is generally ascribed to hydrocarbons (*4, 5*),

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