

ULTRASTRUCTURE OF COMETARY MAGNETITE CAPTURED IN AEROGEL. B. T. De Gregorio¹ and R. M. Stroud¹, ¹U.S. Naval Research Laboratory (4555 Overlook Ave. SW, Washington, DC 20375; bradley.degregorio@nrl.navy.mil).

Introduction: Magnetite is a common secondary mineral in many planetary materials. In chondritic meteorites, it is almost exclusively the product of low temperature aqueous processing of asteroids. These grains are found in various euhedral crystal habits, including octahedral, framboids, and plaquettes [1]. Magnetite can also form by oxidation of Fe,Ni-metal grains [2], either on a parent body or within the solar nebula. The latter process explains how magnetite grains form on the outer surface of chondritic interplanetary dust particles (IDPs) during atmospheric entry heating, even though the bulk of the particles contain a relatively pristine collection of chondritic material [3]. However, the origin of magnetite observed in cometary dust impacts into silica aerogel collected from comet 81P/Wild 2 by the NASA Stardust spacecraft remains enigmatic. Some examples are associated with Fe-metal or Fe-sulfide [4,5], implying secondary alteration, but these observations were not capable of determining whether aqueous alteration or thermal oxidation were responsible. Other observations indicate the magnetite is isolated from other minerals [6-9], or is in close association with carbonaceous matter [7-10], suggesting alternative mechanisms may be in play. Also, there is always the possibility that these grains are terrestrial contaminants trapped during aerogel synthesis. Here we describe one of these isolated magnetite grains extracted from Stardust capture track 196, and the possible origins implied by its ultrastructure.

Methods: The aerogel wafer C2098,6,196,0,0 contains the entrance and most of the stylus of type A track 196, as well as several terminal particles. The wafer was further sub-sectioned with two glass needles held in micromanipulators. A 3 μm terminal particle, TP7, was extracted from the end of a short offshoot track, embedded in molten sulfur, and ultramicrotomed. Various sections were produced: 70 nm sections placed on carbon-supported 200 mesh “thin bar” transmission electron microscopy (TEM) grids for TEM characterization, 90 nm sections placed on SiO₂-supported “thin bar” TEM grids for future, synchrotron-based, X-ray absorption measurements, and 150 nm sections placed on 5 mm silicon chips for future isotopic analysis by secondary ion mass spectrometry.

TEM characterization of sections was performed using the JEOL 2200FS instrument at the U.S. Naval Research Laboratory (NRL). This TEM is equipped with a 65 mm² Oxford X-Max silicon drift detector energy dispersive spectrometer (EDS) for sensitive

determination of elemental composition. In addition, assessment of possible variations in local iron oxidation state will be made with the Nion UltraSTEM instrument, also at NRL.

Results: TP7 is composed of multiple euhedral to anhedral fragments of crystalline Fe-oxide, comprising a single coherent particle (Figure 1). EDS data do not show the presence of any elements other than iron and oxygen within the particle. High resolution imaging of the crystal lattice reveals significant shearing and deformation features (Figure 2a). Consequently, most of the fragments produce poor electron diffraction patterns. However, fragments with the best structural order produce diffraction patterns consistent with magnetite (Figure 2b). Despite their close association, the crystal orientation of individual fragments can vary significantly within a given ultramicrotome section, indicating widespread reorientation of fragmented material.

Discussion: Ultramicrotomy often produces a typical fracture pattern in brittle materials, known as “chatter”, characterized by a relatively parallel, scalloped fracture pattern and lens-shaped fragments. Extreme examples of chatter can cause rotation of indi-

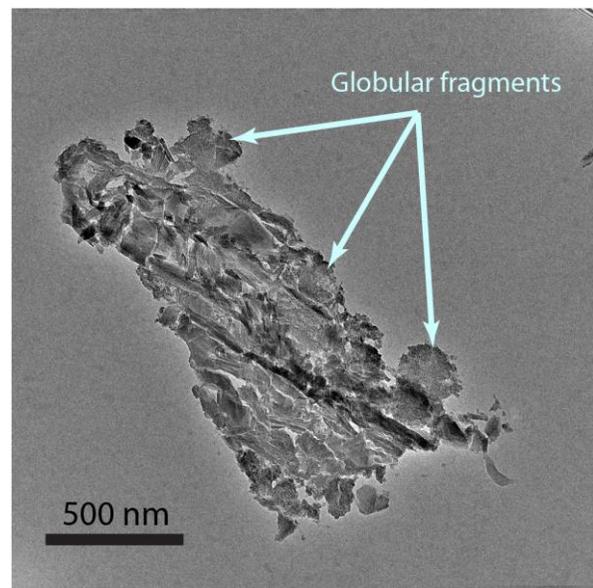


Figure 1. Bright-field TEM image of an ultramicrotome section of a magnetite terminal particle, TP7, from Stardust track 196. The particle shows extreme fragmentation and rotation of fragments. Several fragments have anhedral, “globular” shapes.

dual fragments, but does not cause plastic deformation of the lattice. In contrast, the fragmentation process for TP7 appears to have caused significant crystallographic distortion of lattice planes, and the deformation features observed by high-resolution TEM (Figure 2a) suggest shock processing may be responsible.

Shock experiments show that μm -sized magnetite grains fragment readily and develop shear banding within their crystal structure [11]. At 20 GPa, globular nanograins were observed along shear surfaces, presumably due to incipient amorphization and/or melting [11]. At 30 GPa, twin lamellae form [11]. Stardust particle TP7 contains both extensive microshear banding (Figure 2a) and globular grains (Figure 1), although the globular grains are crystalline rather than amorphous. These observations would imply a local shock of 10-20 GPa during particle capture in the aerogel collector, or, if the globular fragments are due to ablation of material rather than shock, a lower shock pressure of 5-10 GPa. Given the low density aerogel flown on the Stardust mission, impact pressures of up to 8 GPa may be possible [12]. This would also imply that the original impactor could have been a single euhedral grain of multi-domain magnetite that was fractured and deformed during the intense process of sample capture. Since no melt features were observed, capture heating would not have exceeded 1590 °C.

Since the main structural and morphological features of the magnetite in TP7 can be explained by capture processes, we need only consider typical formation mechanisms—namely aqueous alteration or metal oxidation. Magnetite produced in oxidation experiments of chondrite-like metal typically incorporates all metallic elements present in the precursor metal, including Ni, Cr, and Co [12]. Conversely, magnetite produced during aqueous alteration tends to have nearly ideal compositions (> 95% Fe + O). Since only Fe and O are detected in TP7 by EDS measurements, the pre-impact magnetite grain likely originated during aqueous processing on some parent body.

The parent body on which this aqueous processing took place is unclear. Comets are generally too cold to contain significant amounts of liquid water, but transient formation of brines or hydrocarbon fluids could be possible in the subsurface [13]. The relatively late formation age implied for comet Wild 2 [14] also opens the door for accretion of processed asteroidal material, although it is unclear how particle transport out to the Kuiper Belt would be possible once the majority of gas and dust was ejected from the solar nebula.

References: [1] Hua X. & Buseck P. (1998) *M&PS*, 33, A215-A220. [2] Hong Y. & Fegley B. (1998), *M&PS*, 33, 1101-1112. [3] Germani M. et al. (1990) *EPSL*, 101, 162-179. [4] Bridges J. et al. (2010)

M&PS, 45, 55-72. [5] Bridges J. et al. (2011) *LPSC XLII*, 2692. [6] Stodolna J. et al. (2010) *LPSC XLI*, 1657. [7] Hicks L. et al. (2014) *LPSC XLV*, 2051. [8] Price M. et al. (2014) *LPSC XLV*, 1252. [9] Price M. et al. (2015) *LPSC XLVI*, 2000. [10] De Gregorio B. et al. (2015) *LPSC XLVI*, 2625. [11] Reznik B. et al. (2016) *Geochemistry Geophysics Geosystems*, 17, 2374-2393. [12] Dominguez G. et al. (2004) *Icarus*, 172, 613-624. [12] Laurretta D. & Schmidt B. (2009) *Oxidation of Metals*, 71, 219-235. [13] Miles R. & Faillace G. (2012) *Icarus*, 219, 567-595. [14] Matzel J. et al. (2010) *Science*, 328, 483-486.

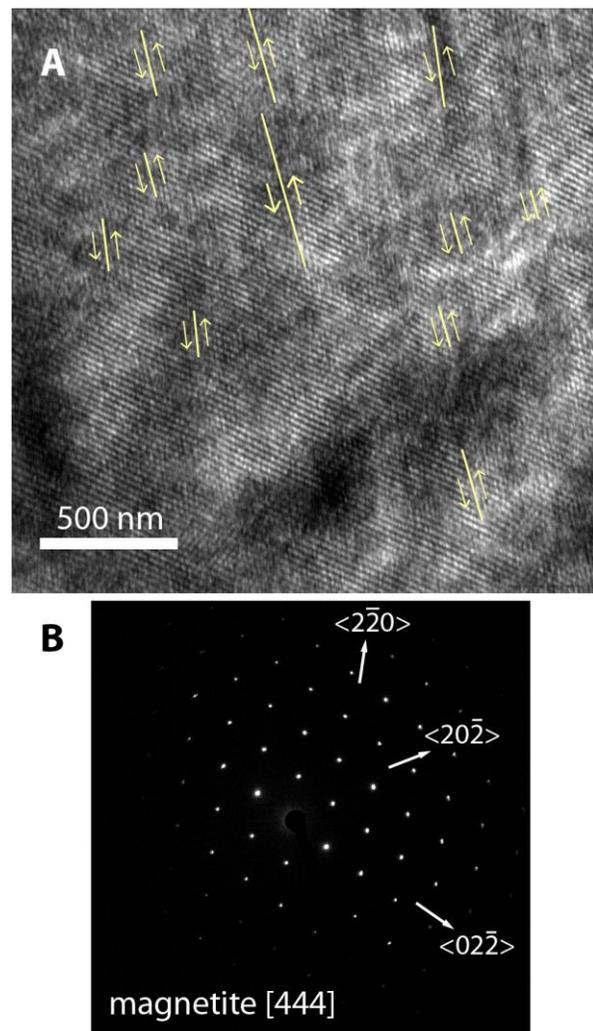


Figure 2. (A) High-resolution TEM image of a magnetite fragment, showing 3.0 Å lattice spacings. Even though this is one of the more crystalline areas, the structure is significantly deformed, causing lattice planes to appear wavy. In addition, shear zones are prevalent (yellow lines). (B) Electron diffraction pattern from a fragment of TP7, showing magnetite lattice reflections.