

FIB-TEM STUDIES OF A GIANT CLUSTER INTERPLANETARY DUST PARTICLE. T. K. Croat¹, D.E. Brownlee², D.J. Joswiak², and R.C. Ogliore¹, ¹ Laboratory for Space Sciences and Department of Physics, Washington University, St. Louis, MO 63130, USA, ² Dept. of Astronomy, University of Washington, Seattle, WA 98195 (tkc@wustl.edu)

Introduction: While many microstructural studies have been performed on interplanetary dust particles (IDPs), most have employed ultramicrotomy to obtain serial sections that succeed in preserving a significant fraction of the material for TEM observation [1]. However, components within the IDPs can be detached and displaced during ultramicrotomy, particularly in the more friable chondritic porous (CP)-IDPs, which to some degree disrupts the observed microstructure [2]. Although it destroys more sample, the focused ion beam (FIB) technique more accurately preserves mineral associations, void structures and other microstructural features. This technique also allows for direct targeting of specific grains of interest, such as enstatite whiskers. Here we present TEM microstructural studies of five FIB sections taken from the fragments of a giant cluster IDP. Continued mineralogical studies of this type are important to clarify the relationship between CP-IDPs and the Stardust Wild 2 samples [3].

Methods: Five FIB sections were made from fragments of a giant cluster IDP using a FEI Quant 3D FIB. A total cross-sectional area of $11.2 \mu\text{m}^2$ was examined from all five sections using JEOL 2000FX and 2100F TEMs, and the degree of crystallinity, crystal structure using electron diffraction, and chemical composition were all assessed. TEM-EDX quantitative analyses were made using various stoichiometric oxide and USMN standards and Cliff-Lorimer techniques.

Results and Discussion: Fragments from the giant cluster particle show the friable microstructure typical of chondritic porous IDPs (Fig. 1). Regions often containing multiple grain fragments were chosen for FIB to maximize the amount of material contained in each section (Fig. 2). The five FIB sections consisted primarily of silicate glass, with a crystalline volume fraction of ~6%, although this estimate does not include <10 nm nanocrystals due to lower detection probability. Iron nickel sulfide grains with mean diameters of 100 to 350 nm are the most common crystallites, and these occur in both low ($\text{Fe}_{48}\text{Ni}_{2}\text{S}_{50}$) and high ($\text{Ni}_{29}\text{Fe}_{23}\text{S}_{48}$) Ni variants. Four MgFe-rich silicate crystals (30-170 nm diameters) were also present and these showed a wide range of Mg/Fe ratios. Four MgFe-rich silicate crystals (30-170 nm diameter) were also present and these showed a wide range of Mg/Fe ratios. The TEM-EDX compositions of the individual silicate and sulfide crystals along with the wide area averaged compositions of 300-700 nm clumps of

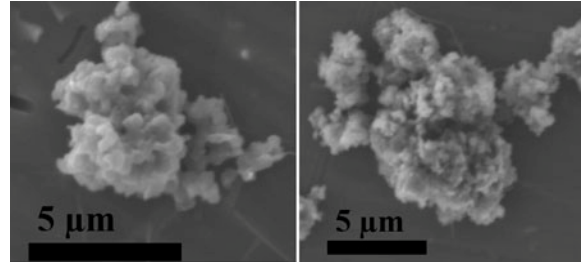


Fig. 1. Secondary electron images of fragments from the giant cluster IDP with surface morphology typical of CP-IDPs.

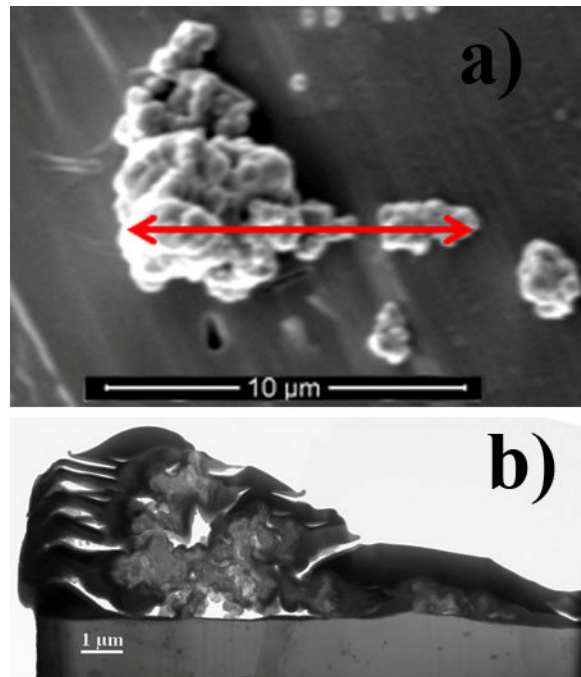


Fig. 2. A) Secondary electron image of planned FIB traverse (red arrow) and b) bright-field TEM image of the resulting FIB section containing multiple fragments from the giant cluster particle.

glassy material are plotted in Mg-Fe-Si and Mg-Fe-S ternary diagrams (Fig. 3). The compositions of the smaller MgFe silicates are somewhat uncertain due to background contributions from surrounding glass regions. However, further diffraction and EDX data will be collected to conclusively identify these grains (likely either olivine or pyroxene). The friable

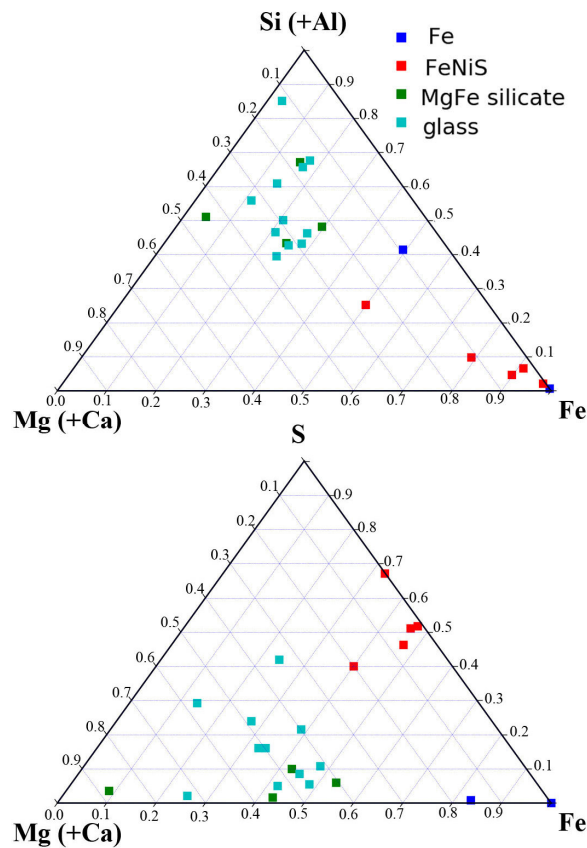


Fig. 3 Ternary plot of TEM-EDX compositions of crystallites along with wide-area averaged compositions of 300-700 nm diameter glass regions. Minor Ca (0-2.4 at. %) and Al (0-8 at. %) content are added to the Mg and Si respectively.

microstructure, unequilibrated compositions and the presence of anhydrous minerals are all consistent with a cometary origin for this CP-IDP [4].

Some GEMS-like objects (Fig 4) are found within the FIB sections, with apparent diameters of 250-600 nm. The average composition of the entire GEMS-like region in Fig 4 is $(\text{Si}_{33}\text{Fe}_{21}\text{Mg}_{20}\text{S}_3\text{Ni}_1\text{Ca}_1)\text{O}_{31}$ and the region contain nanocrystalline metal grains (Fig 3b.). The size, bulk composition and types of internal phases are all similar to those reported for GEMS grains [1].

The FIB technique allows one to make a fairly accurate estimate of the particle's porosity from cross-sections without the possibility of mechanical damage to low density particles from ultramicrotomy. Using similar techniques, we estimate a porosity of 5% in agreement with the porosity of [5]. However, void

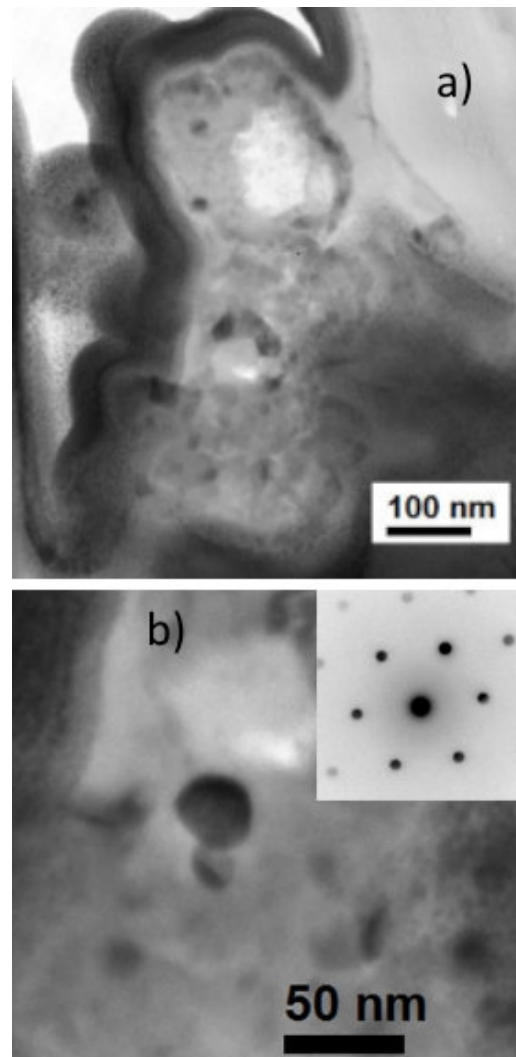


Fig. 4 Bright-field image of a) GEMS-like region in giant cluster IDP and b) close-up of small included kamacite crystal (with inset 111 zone diffraction pattern).

spaces smaller than the FIB section thickness are not identified in these cross-section, and infill by Pt/Ga during FIB can also induce errors [2]. Porosity estimates using other techniques such as x-ray nanotomography [6] can be considerably higher.

References: [1] Bradley J.P. (2004) in *Treatise on Geochemistry* vol. 1, New York, Elsevier. [2] Gainsforth Z. et al. (2017) *LPSC 48*, #1642 [3] Westphal et al. (2017) *Met. Planet. Sci.* 52, 1859-1898. [4] Joswiak et al. (2017) *Met. Planet. Sci.* 52, 1612-1648. [5] Dahl et al. (1995) *LPSC 26*, #307. [6] Hu Z.W and Winarski R. (2013) *LPSC 44*, #2521