

PETROGRAPHY OF FOUR CP-IDPS. Z. Gainsforth^{1,†}, C. E. Jilly-Rehak¹, A. L. Butterworth¹, A. J. Westphal¹, ¹Space Sciences Laboratory, University of California, Berkeley, CA 94720, [†]e-mail: zackg@ssl.berkeley.edu.

Introduction: Focused ion beam (FIB) has proven useful for petrographic studies of meteorite matrices[1]. Here we present petrographic studies by Transmission Electron Microscopy (TEM) of four particles FIBed from a giant cluster chondritic-porous interplanetary dust particle (CP-IDP). In contrast with ultramicrotomy, FIB preparation preserves petrographic relationships.

Experimental: We studied cluster IDP L2071,17 at the National Center for Electron Microscopy at the Lawrence Berkeley National Laboratory. IDP fragments were FIBed directly from a nucleopore filter with an FEI Strata 235 Dual-Beam, using Ga⁺ at 30 keV for primary milling and 6 keV for cleaning. Pt deposition was used to protect the fine-grained material from the ion beam as much as possible, which resulted in some infill of the pores. We used an FEI Titan TEM with beam voltages between 80-200 keV, and a 0.6 sr EDS detector for imaging, electron diffraction and EDS analysis. We used a Zeiss Libra 200MC TEM operating at 200 keV and with an in-column Omega energy filter for imaging and diffraction.

Terminology: *Equilibrated Aggregates (EAs)* are groups of nanophase minerals with melt textures including triple junctions, and planar interfaces [2]. *Unequilibrated Aggregates (UAs)* are nanophase groups of minerals that do not show any evidence of melt textures [2]. GEMS are sometimes considered a type of UA. The definition of *Matrix Aggregates (MAs)* is dependent on various models for IDP formation as discussed in Rietmeijer *et al.*, (1994) [3]. Here we define MA as a collection of UAs and/or EAs in a carbonaceous matrix.

Observations: *Humpty and Dumpty* are separate IDPs that were < 1 μm apart on the nucleopore filter. We fit them into one FIB section. Humpty contains three MAs. One contains a 3 μm sulfide EA with varying amounts of Ni, no metal, olivine, high- and low-Ca pyroxene and some UAs. The other two MAs contain UAs including GEMS, silicates, and Fe-S-O spheroids. The spheroids have a hydrated appearance, and are embedded in material that appears pristine and unhydrated. Dumpty has one MA with a 3 μm silicate EA, and a region of UAs. Because the FIB Pt did not penetrate the MAs in Humpty or Dumpty, we conclude that the porosity of the MAs is low.

Metropolis contains three MAs. One has a large amorphous SiO₂ grain decorated with UAs and carbon. Another MA (Figure 1) is considerably more porous than the MAs in Humpty and Dumpty with holes lined with Pt from the FIB sample preparation. It contains 14 EAs with silicate branch-like structures each terminated with a sulfide, often hemispherical, indicating that the sulfides grew on to the branches after the branches formed. Some of the sulfides and underlying silicates have epitaxial crystal orientations, indicating a close relationship during growth and not simple aggregation of pre-formed silicates and sulfides. GEMS are attached to the EAs, and GEMS also show sulfides apparently grown onto the surface — the sulfides lie flat against the surface of the GEMS, yet have various morphologies on the external face. The GEMS

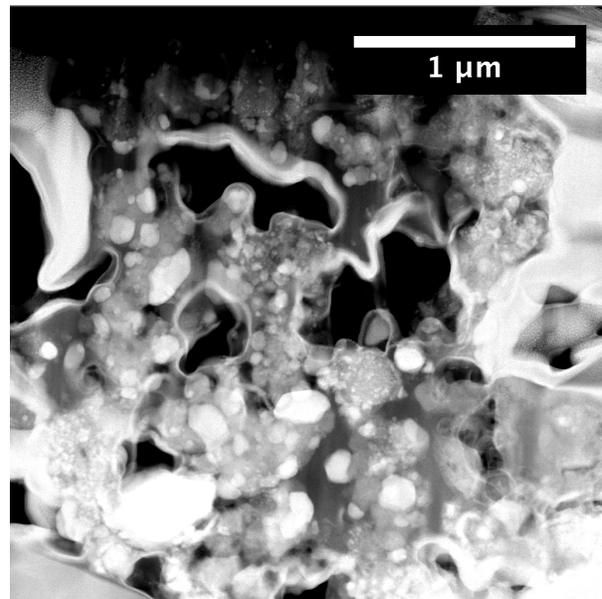


Figure 1: TEM HAADF of a matrix aggregate in Metropolis.

contain Ni-rich metals, but no internal sulfides. Except for a single sulfide, each EA has the same bulk composition as the others. All measured elements of each EA are within 40% of chondritic (normalized to Mg) except O and Si (the sample was mounted on an Si frame).

Nessie contains an MA composed of a 4 μm EA coated with 500 nm of fine-grained crystals and carbon (Figure 2). On either side of the MA are two more MAs made of UAs and carbon. *Nessie* also contained Fe-O-S hydrous-looking spheroids similar to those found in Humpty and Dumpty.

Particle 4 has several densely packed MAs. One is composed of a 4 μm jagged olivine/pyroxene EA with embayments filled by UAs of varying composition. There are also MAs composed of UAs similar to those seen in Humpty and Dumpty, Metropolis and *Nessie* except that the MAs are separated by SiO₂ veins. We suspect the SiO₂ could be oxidized silicone oil, and plan to examine it further with XANES or EELS to verify this suspicion[4]. Because the FIB section for particle 4 is thicker than in Metropolis, there is some 3D overlap of MAs and it is not clear how to define their outlines, nor the outlines of their finer components. Nevertheless, we could see that MAs were chemically diverse. They included an unusual 400 nm wide carbon triangle, whose original shape was probably tetrahedral or prismatic prior to FIBing. We also found an example of sulfide growing conformally around a silicate. Finally, there are two sulfides which are surrounded with a conformal 10nm C coat suggesting they were resident

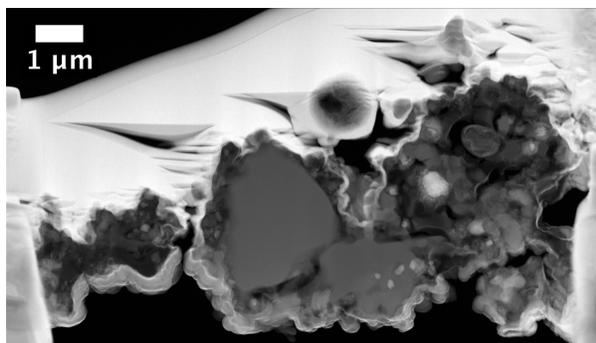


Figure 2: TEM HAADF of Nessie.

in the nebula without accompanying phases, yet now they are fused into an MA with other material.

Porosity: Determination of porosity (ϕ) from BF or HAADF images was unreliable because of infill by Pt and Ga in the FIB. However, with EDS maps we could correctly draw outlines and view pores in the liftout. We did this by adding the counts from major rock-forming elements (including C) and subtracting the Ga and Pt counts. This produced an image with positive pixels showing rocky material and negative pixels showing deposition material. We drew a convex hull around the rocky portion and used the fraction of positive pixels to compute the porosity. The results are summarized in Table 1 and show that these CP-IDPs have a porosity between 25-45%.

IDP	ϕ	A_C	A_S	A_{Si}	Σ	ρ
		% of area				g/cm^3
Humpty	45	26	27	43	96	1.8
Dumpty	40	34	19	45	98	1.8
Metropolis	40					
Nessie	40	5	8	78	91	1.9
Particle 4	25					
Particle 4 ¹	5					

Table 1: Porosity (ϕ) and modal abundance for carbonaceous (A_C), sulfide (A_S) and silicate (A_{Si}) phases and the sum of the three (Σ) for each of the FIBed IDPs. All porosities are rounded to the nearest 5%. ¹Computed from imaging – wrong result for comparison only. ρ is computed from A_C , A_S , A_{Si} and ϕ .

In addition to making the IDP outline obvious, EDS maps also allowed us to distinguish between extraterrestrial carbon and carbon deposited during the Pt deposition which contains some Pt and Ga. For three of the IDPs we computed the modal abundance of material which was carbonaceous (A_C), sulfide (A_S), or silicate (A_{Si}), see Table 1. In principle $A_C + A_S + A_{Si} = 100\%$, but some pixels could not be correctly sorted due to noise or bias in choosing the thresholds. By assuming densities of $\rho_C = 2 \text{ g/cm}^3$ for the carbonaceous material, $\rho_S = 4.5 \text{ g/cm}^3$ for sulfide, $\rho_{Si} = 3.2 \text{ g/cm}^3$ for silicate, we estimated the density of these CP-IDPs to be ≈ 2

g/cm^3 . This density is in agreement with measurements from the Rosetta mission using the GIADA instrument that found dust grains ejected from Comet 67P/Churyumov-Gerasimenko had densities between 1-3 g/cm^3 [5], but is considerably larger than measurements of the C-G nucleus density of 0.5 g/cm^3 [6] measured independently by radio science on Rosetta. This estimate is also somewhat higher than 0.7-1.7 g/cm^3 previously measured from TEM sections [7].

Formation Sequence: The overall formation sequence we saw in these IDPs starts with UAs and EAs agglomerating into compact clusters with low porosity and held together with carbonaceous material (MAs). Several MAs are then loosely bound together into an IDP. In most cases, the majority of the pore space in the IDP resides between the MAs, not within.

Studies using microtomed sections frequently find dispersed UAs or EAs and fragments of carbonaceous material. The petrographic relations are often not clear though there is a tendency for some IDPs to have multiple objects of a similar family. For example, Gainsforth (2017) [8] observed sulfidation that affected multiple GEMS and other objects in an IDP yet looked independent from other material. However, almost all material in these FIBed IDPs is bound into MAs, and each MA is less diverse than the IDP as a whole. We propose that many IDP components are part of an MA, but microtoming MAs produces fragments that appear separated and mask the petrographic relationships.

We suggest that the MAs in our IDPs sample different histories and are not necessarily related to other MAs even though they are found in the same IDP. For example, the MA in Metropolis with the chemically homogenous EAs could sample a region of condensation material that was sulfidized and then aggregated, while the MA containing the large amorphous SiO_2 grain is probably not condensation material. If Metropolis had been studied in microtomed sections, SiO_2 fragments may have been mixed with the chondritic EAs, the tree-branch shapes of the EAs could have been destroyed so the sulfides and silicates would have appeared coeval instead of sequential, or worse, they may have appeared entirely dis-related. The GEMS could also have been separated from the EAs making them appear independent, and other chance juxtapositions may have lead to incorrect conclusions.

Conclusions: FIB lift-out sample preparation enables mesoscale petrographic studies that have not been possible with samples prepared by ultramicrotomy. CP-IDPs are made mostly of MAs that are less diverse than the IDP as a whole; therefore, this technique may provide improved insights into early nebular processes.

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