

# Coordinated TEM and Nanosims Oxygen Isotope Analysis of Interplanetary Dust Particles Prepared by Focused Ion Beam

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## Summary

The objective of this study is to develop a new method for coordinated TEM and isotopic analysis of Interplanetary Dust Particle (IDP) components. Most bulk and multiphase IDP analyses from literature lack petrographic context [4,5] – we aim to take oxygen isotope analyses of individual mineral grains and components to determine their contributions to the bulk O-isotope trends. Such coordinated TEM/SIMS analyses can help to determine the origin of IDP materials and constrain disk evolution and transport models.

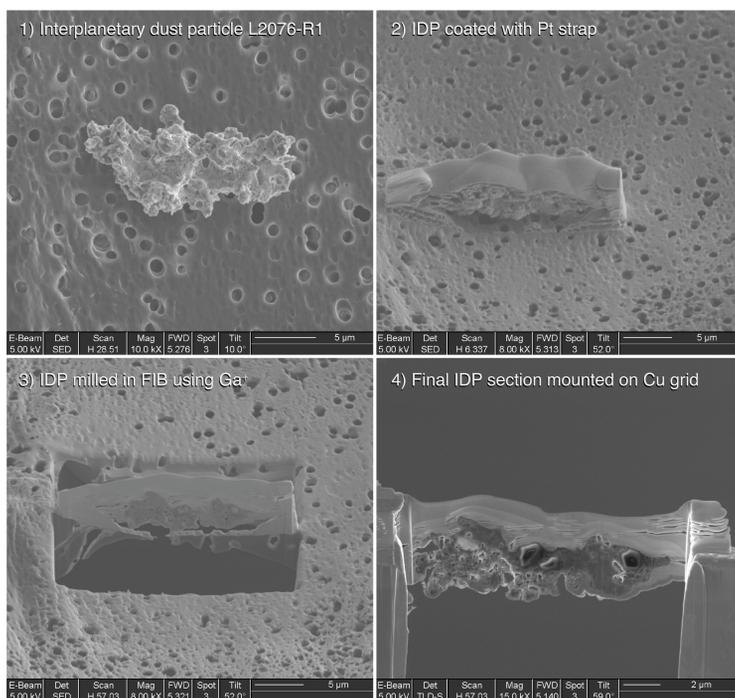


Figure 1: Process of creating the FIB sections of IDPs, showing sample L2076-R1.

## Methods

Four IDPs were prepared for coordinated TEM and NanoSIMS analysis: Three from Cluster 17 (L2071,17), here named Particle 4, Humpty, and Dumpty, and one additional IDP L2076, R1, referred to here as particle R1 (Fig. 1). Humpty and Dumpty are two IDPs in one FIB section (Fig. 2).

### Sample Preparation: FEI Strata 235 Focused Ion Beam (FIB) at NCEM, Lawrence Berkeley National Lab

- Pt strap deposited over the top of each IDP (Fig. 1)
- 30 keV Ga<sup>+</sup> ion beam for milling
- Final sample thickness ~150-250 nm

### Technique advantages:

- Preserves petrographic context of fine-grained IDP material
- Section can be precisely positioned to extract desired regions
- No additional handling or embedding of the particle necessary

### Petrography: Transmission Electron Microscopy (TEM) at NCEM, Lawrence Berkeley National Lab

The petrography and chemical analysis portion of this study was conducted using a variety of TEMs and energy-dispersive X-ray spectroscopy, with methods and results described in [10].

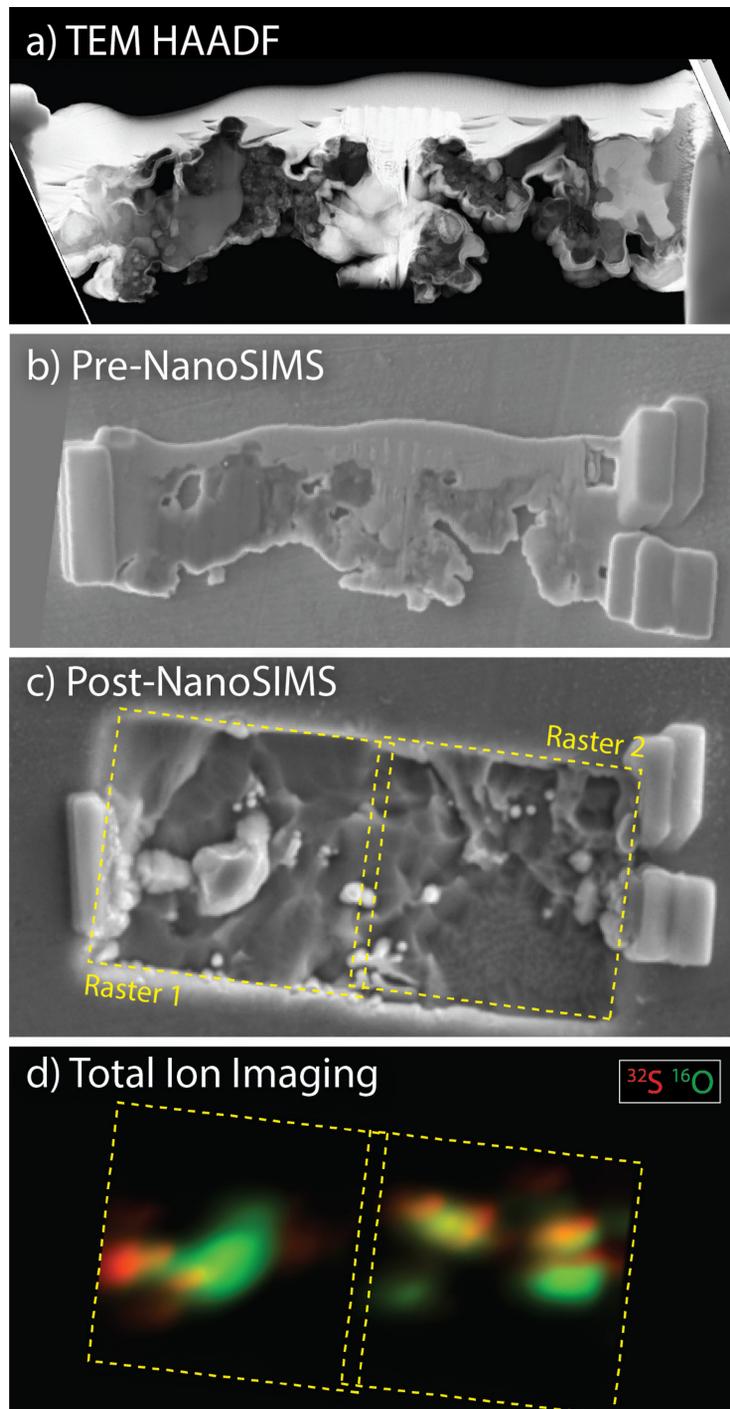


Figure 2: Analysis of IDPs Humpty and Dumpty. a) TEM High-Angle Annular Dark-Field image (HAADF) showing both coarse and fine grained materials in the two IDPs. b) Secondary electron image of the FIB section after re-mounting onto a flat conductive steel surface. c) Secondary electron image of the same section after NanoSIMS analysis. d) Total ion image from NanoSIMS analysis, depicting <sup>32</sup>S in red, <sup>16</sup>O in green.

### Oxygen Isotopes: NanoSIMS 50L at Academia Sinica

After TEM analysis, the IDP sections were affixed to a 1-cm polished stainless steel disc using Pt deposition in the FIB (Fig. 2b). The disc was coated with 20 nm of Au for conductivity

- Imaging mode: 5 x 5 μm raster, 64 x 64 pixels, 3000 μs/px dwell
- Isotopes measured: <sup>16</sup>O, <sup>17</sup>O, <sup>18</sup>O, <sup>28</sup>Si, <sup>32</sup>S, <sup>24</sup>Mg<sup>16</sup>O
- 3 pA current, MRP ~ 7600
- Standards: San Carlos olivine prepared in by same FIB method

## Data Analysis

Preliminary results were processed using L'Image NanoSIMS software (Fig. 2d). However, due to the complex, fine-scale heterogeneous structure of the IDPs, we needed to define regions of interest (ROI) for phases that fade in and out throughout the analysis cycles, essentially creating 3-dimensional ROIs in the X-Y-cycle datacube. To address this, we developed a python package that visualizes, and calculates ratios for 3D ROIs.

### Custom Python Package

- Reads data, performs deadtime & QSA corrections
- Calculation of ratios, uncertainties, delta values
- Outputs NanoSIMS raster data to Paraview visualization software
- Selection of 3D ROI in Paraview (e.g., Fig. 3)
- Additional data reduction using python programmable filters

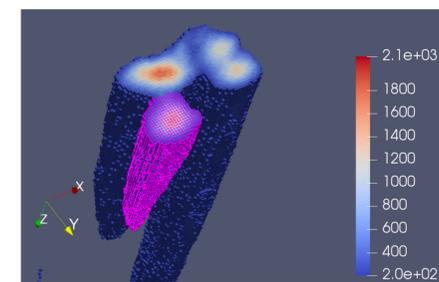


Figure 3: Three-dimensional rendering of NanoSIMS IDP isotope data. The X-Y plane is the raster area, and Z is the analysis cycle. Color bar represents counts of <sup>16</sup>O, with a threshold value of 200 counts/px to remove background and noise. The pink depicts a selected area for component analysis.

## Results and Discussion

- Unequilibrated material of diverse O-isotopic composition
- Coarse-grained silicates have <sup>17,18</sup>O-enriched compositions relative to corresponding matrix (Fig. 4)
- Fine-grained matrix composition may reflect a mixture of material inherited from parent molecular cloud, or diverse sampling of inner Solar System reservoirs [e.g., 6]
- Low counting statistics due to thin samples
- Mass-dependent fractionation trend in some analyses: analytical artifact due to variations in sample topography?

### Future Development

We are working on improving this method by using thicker samples (up to ~300-500 nm) to increase precision, and to reduce causes of mass-dependent fractionation due to thin samples and variable topography.

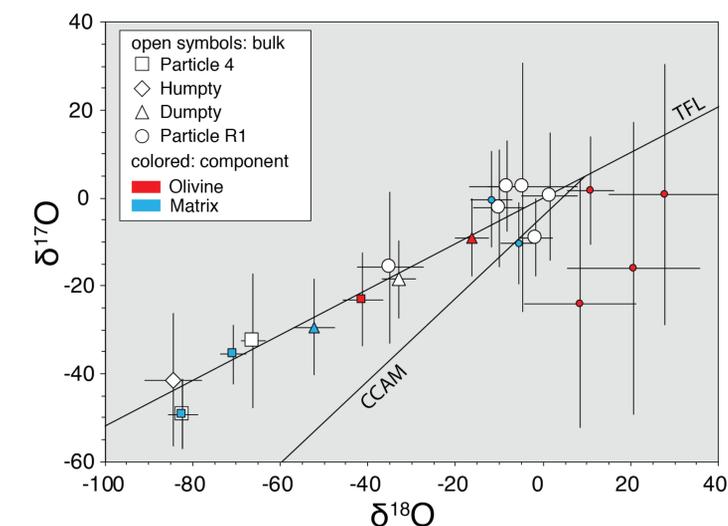


Figure 4: Oxygen isotope result for the four IDPs in this study. Bulk analyses are given in open symbols, while components are color coded in red (silicate) and blue (fine-grained material).

References: [1] Nesvorný D. et al. (2010) *Astrophys. J.* 713, 816-836. [2] McKeegan K. D. et al. (2006) *Science* 314, 1724-1728. [3] Nakamura T. et al. (2008) *Science* 321, 1664-1667. [4] Aleon J. et al., (2009) *GCA* 73, 4558-4575. [5] Starkey N. A. et al. (2014) *GCA* 142, 115-131. [6] Oglione R. C. et al. (2015) *GCA* 166, 74-91. [7] Defouilly C. et al. (2017) *EPSL* 465, 145-154. [8] Traxlmayr U. et al. (1984) *Int. J. Mass Spec. Ion Proc.* 61, 261-276. [9] Hillion F. et al. (2008) *GCA* 72, A377. [10] Gainsforth Z. et al. (2017) *LPS XLVIII*, 1642. [11] Caplan C. E. et al. (2015) *MetSoc* 78, 5333.

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