

# Apollo 11 Agglutinate Investigated by Atom-probe Tomography

Josiah B. Lewis (1), Jeffrey J. Gillis-Davis (2), Dieter Isheim (3), Bradley L. Jolliff (4), David N. Seidman (3), and Ryan C. Ogliore (1).

(1) Laboratory for Space Sciences, and Department of Physics, Washington University in St. Louis, Missouri, USA, (2) University of Hawai'i at Mānoa, Hawai'i Institute of Geophysics and Planetology, Hawai'i, USA, (3) Northwestern University Center for Atom-Probe Tomography, and Department of Materials Science and Engineering, Northwestern University, Illinois, USA, (4) Department of Earth and Planetary Science, Washington University in St. Louis, Missouri, USA. (jlewis@physics.wustl.edu)

#### Abstract

We use atom-probe tomography (APT) to analyze nanophase iron  $(npFe^0)$  grains and surrounding glass matrix in lunar agglutinates in 3D with subnanometer spatial resolution. The APT reconstructions allow us to investigate the relationship between the  $npFe^0$  and surrounding agglutinate glass or amorphous rims in terms of local chemical composition. We use this information to discuss space-weathering processes that lead to their formation.

# 1. Introduction

Space weathering is a complex set of processes that affects the surfaces of airless bodies. One of its principal effects is the formation of nanophase metallic iron particles (npFe<sup>0</sup>) [3]. 2D methods (e.g., TEM) have advanced our understanding of the formation of npFe<sup>0</sup> over the past 25 years. Regardless, there is still much that is unknown about npFe<sup>0</sup>, such as mechanisms of formation (e.g., in situ reduction [4], immiscible melt droplets forming from vapor [3], entrapment of meteoritic Fe, or Ostwald ripening [2]). The size distribution of npFe<sup>0</sup> spheres also lacks constraints: npFe<sup>0</sup> found in agglutinates have been reported as (1) similar in size to those found in amorphous rims [5]; (2) at least twice as large as those found in amorphous rims, [4]; or (3) over 30 times larger [1].

# 2. Methods

Using focused ion-beam liftout and milling, we prepared two <50 nm diameter APT nanotips, extracted  $\sim 6 \mu m$  apart from the surface of an Apollo 11 agglutinate (10084). We analyzed these samples using a CAMECA LEAP 5000X Si atom-probe tomograph, a time-of-flight mass spectrometric

instrument with an element-independent 80% detection efficiency. Several million atoms were collected from each nanotip, resulting in a reconstructed volume of  $70 \times 70 \times 70$  nm<sup>3</sup> from the first nanotip and  $100 \times 100 \times 115$  nm<sup>3</sup> from the second nanotip. The data were corrected for background and relatively minor effects from isobaric overlaps.

## 3. Initial Results and Discussion

The first nanotip contained two ~20 nm diameter npFe<sup>0</sup> grains within 10 nm of the surface, and a number of smaller npFe<sup>0</sup> located more than 25 nm deep in the agglutinate. The two npFe<sup>0</sup> grains contain sufficient Ni to be kamacite (Ni/Fe  $\approx 0.06$  and 0.05), which is evidence for meteoritic origin. The Ni concentration is lower in the small npFe<sup>0</sup> more than 25 nm deep (Ni/Fe < 0.05). The concentration of Al, Ca, and Mg relative to Si and O in the upper surface of the agglutinate (~5–25 nm), where the larger Fe grains are located, is (Al+Ca+Mg)/(Si+O) < 0.25. But the ratio is higher deeper in the sample: (Al+Ca+Mg)/(Si+O) > 0.4. Hydrogen is enriched in the same region. These compositional trends appear consistent with a solar wind-altered rim.

The second nanotip was composed almost entirely of a half-µm Fe grain. This grain has a low Ni content (Ni/Fe < 0.01), and regions enriched in C (C/Fe $\approx$ 0.12 in these regions) and P (P/Fe $\approx$ 0.03). The P- and Cenriched regions in the large Fe grain may be the result of incomplete intermixing of P- and C-rich grains within the Fe during a partial melting event. Fe grains with concentrations of Ni and S this low are typically <10 nm and formed by vapor deposition from impacts/solar wind sputtering, but this Fe grain is too large to be vapor-formed. Hence it could be formed by Ostwald ripening, but further analyses are needed before we can confirm this origin. A 40 nm long, smooth Fe-S subgrain (S/Fe $\approx$ 0.4) is located inside the Fe,  $\sim 80$  nm beneath the surface (lower gold shape in Fig. 1). The Fe-S grain has Ni/Fe $\approx 0.02$ . This Fe-S could be a lunar mineral in a reaction relationship with the surrounding Fe, that was incorporated into the still-liquid agglutinate.

### 4. Summary and Conclusions

We have observed two Fe-Ni grains that we suggest are meteoroid metal, and an Fe-S subgrain that we suggest is lunar in origin. The Fe-S grain is embedded in a large Fe grain that may have formed by Ostwald ripening. We also see that Al, Ca, and Mg concentrations are depleted near the surface and enriched deeper in the silicate. The depletion may result from solar wind sputtering. We will continue this work by further peak corrections, quantifying uncertainty, and analyzing trace elemental concentrations, such as Cl, Co, Cr, Cu, H, Na, and Ti.

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Figure 1: Rotation series of the APT data reconstruction for the second nanotip. Isoconcentration surfaces surround regions with more than: 10 at. % C (gray); 20 at. % S (gold); and 2 at. % P (purple). The green dots are a small fraction of the detected Fe atoms and represent the Fe distribution. Annotation "A" marks the Fe-S grain. "B" marks the largest of the P-enriched areas.