

Nanometer-scale Analysis of Space-weathered Lunar Regolith by Atom Probe Tomography

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Solar wind implantation and micrometeorite impacts (i.e., space weathering) alter the surfaces of airless bodies throughout the solar system [1]. In lunar regolith, space weathering produces complex microstructures, specifically amorphous rims that coat grains and often contain nanophase and microphase metallic Fe (npFe and mpFe) [2]. FeS inclusions were found on the surface of dust particles returned from asteroid Itokawa [3]. In addition to informing models of space weathering, detailed knowledge of the microstructure, particle size distributions, and number densities of npFe and mpFe is necessary for interpreting and modeling near-infrared reflectance spectra obtained by remote sensing of the lunar surface and other bodies in the solar system [4].

We use atom-probe tomography (APT) [5], with 3D nano-scale spatial resolution and single atom sensitivity, to characterize the composition of the surface layer of a lunar regolith agglutinate from sample 10084, returned by the Apollo 11 mission in 1969. SEM imaging and FIB lift-out techniques enable targeted APT analysis and contextual interpretation of the results [6].

APT analyses of two sample volumes from the surface of the same agglutinate particle reveal distinct compositional layering and a variety of heterogeneous inclusions with nanometer-scale characteristic dimensions. One APT analysis reveals two ~20 nm diameter npFe grains near the surface, label 'A' in Fig. 1. These npFe grains contain a comparatively high Ni concentration (5-6 at.%), similar to kamacite, and thus provide evidence for meteoritic origin. Approximately fifty smaller npFe particles with ~3-5 nm diameter and with a lower Ni concentration are located in a layer at 20-60 nm depth, label 'B' in Fig. 1. Aluminum, Ca, and Mg are depleted near the surface and the relative H concentration is higher in the same region, consistent with a solar-wind altered rim.

A second APT analysis was targeted on a ~0.5 μm diameter mpFe particle that was easily identified by back-scatter contrast in SEM as a region with higher than average atomic number (Z), at a location ~ 5 μm away from the first APT analysis of this same agglutinate grain. This mpFe particle has a lower Ni content ($\text{Ni}/\text{Fe} < 0.01$) but regions enriched in C ($\text{C}/\text{Fe} \sim 0.12$), label 'A' in Fig. 2, enriched in P ($\text{P}/\text{Fe} \sim 0.03$), label 'B' in Fig. 2, and a ~40 nm long ellipsoidal FeS-type inclusion, label 'C' in Fig. 2. The C, P, and S-rich inclusions may also indicate meteoritic contamination.

The evidence for a meteoritic component to npFe and mpFe is an unexpected finding that runs counter to most models describing their formation [7].

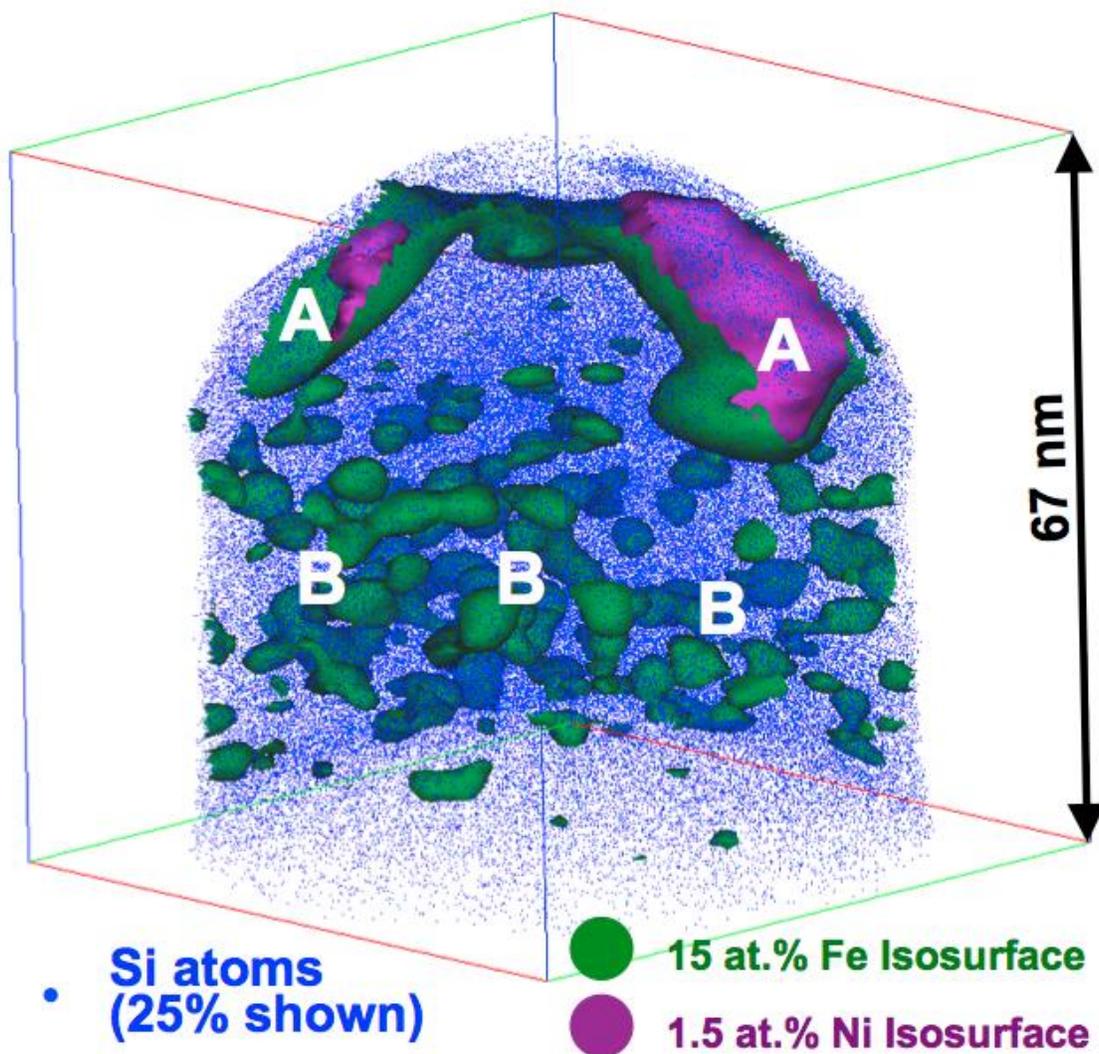


Figure 1. 3D atom-by-atom APT reconstruction of a volume from the surface layer of a lunar agglutinate particle. Two nanophase Fe particles (label 'A'), delineated by a 15 at.% Fe isoconcentration surface, are present in a layer ~7-22 nm from the surface (top). These larger npFe particles contain up to ~5-6 at.% Ni, shown by a 1.5 at.% Ni isoconcentration surface. Approximately 50 smaller npFe 3-5 nm in diameter (label 'B') are located in a layer at 20-60 nm depth from the surface.

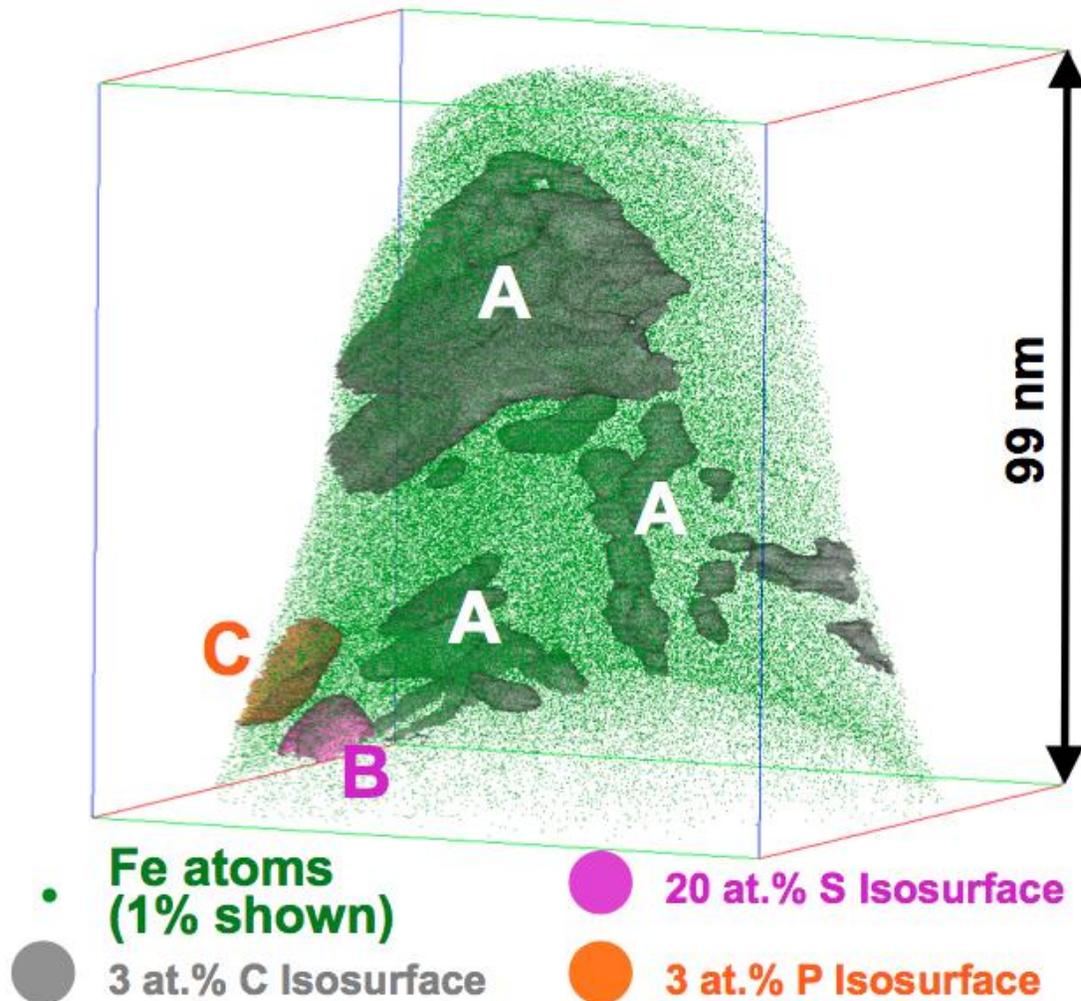


Figure 2. APT reconstruction of a volume in a microphase Fe particle at the surface of the same lunar agglutinate particle but about 5 μm away from the volume displayed in Fig. 1. This mpFe particle contains regions rich in carbon (label ‘A’), delineated by a 3 at.% C isoconcentration surface. Additionally, a region enriched in P (label ‘B’, 3 at.% P isoconcentration surface), and a region rich in S (label ‘C’, 20 at.% S isoconcentration surface), possibly a FeS-type inclusion, are detected in this mpFe grain.

References

- [1] J Bibring et al., *Science* **175** (1972), pp. 753-755.
- [2] LP Keller, DS McKay, *Science* **261** (1993), pp. 1305-1307.
- [3] T Noguchi et al., *Meteoritics & Planetary Scienc.* **49** (2014), pp. 188-214.
- [4] B Hapke, *Journal of Geophysical Research: Planets* 106(E5) (2001), pp. 10039–10073.
- [5] DJ Larson et al., *Local Electrode Atom Probe Tomography: A User's Guide*, Springer (2013).
- [6] JB Lewis et al., *Apollo 11 Agglutinate Investigated by Atom-probe Tomography*. EPSC-DPS Joint Meeting 2019. 13, EPSC-DPS2019-75-2.
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