ATOM PROBE TOMOGRAPHY OF LUNAR REGOLITH ILMENITE GRAIN SURFACES

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Introduction: The surfaces of airless bodies, such as the moon and asteroids, are subject to space weathering, which alters their mineralogy. Space weathering is predominantly caused by micrometeorite bombardment, irradiation by solar wind, and galactic cosmic rays [1]. Products of space weathering include amorphization resulting in agglutinate glass, submicroscopic or nanophase metallic iron (SMFe and $npFe^0$), re-deposition rims, or vesicles filled with implanted solar wind [2]. Ilmenite from lunar soil ($FeTiO_3$) is particularly well studied as it is retentive to the products of space weathering, including noble gases [3]. Recent studies of space weathering products on lunar samples have used TEM to demonstrate that these noble gases can be present in vesicles and planar defects [4]. In atom probe tomography (APT), atoms are field-evaporated from the surface and detected by a position-sensitive time-of-flight mass spectrometer, allowing both the compositions and the distributions of atoms imaged in 3-D with sub-nm to nm spatial resolution [5]. APT has previously been used to study simulated space weathering products ($npFe^0$ produced by hydrogen implantation) in olivine [6].

Methods: Ilmenite grains in the size range 75-150 µm were extracted from Apollo 17 sample number 71501 at ETH Zurich. The sample was ion-beam-sputter coated with Ni to protect the mineral surface from Ga^+ ion irradiation during focused-ion beam (FIB) based sample preparation. Several APT tips were prepared from this grain using the TESCAN LYRA3 FIB-SEM at the Univ. of Chicago. Ridges of this grain were annularly milled to produce nanotips that were then lifted out [7] and mounted on flat-top Si micro tips by Pt deposition. The nanotips were then sharpened by ion-milling to tip apex radii of ~30 nm. The nanotips were analyzed with a LEAP 4000X Si tomograph at the NUCAPT facility of Northwestern Univ.

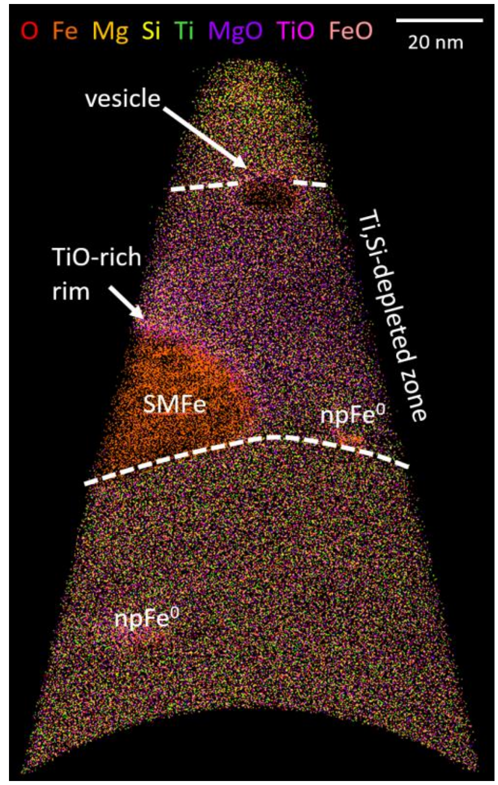

Figure 1: A 3-D APT reconstruction, in cross-section, of Tip C. Atomic or molecular ions are represented by a different color.

Results and Discussion: Three out of four nanotips were successfully analyzed. Two of the nanotips (B and C) sampled the top surface of ilmenite at different depths with space weathering products. Nanotip D sampled the ilmenite at about 50 nm below the surface. Besides Fe, Ti, and O, the major elements of ilmenite, traces of Mg, Si, Al, Ca, Mn, and Cr were detected. These elements are a large component of bulk lunar soil [8]. Nanotip C (Figure 1) contains the most dramatic representation of space weathering products and compositional zoning. Both nanotips contain $npFe^0$; in nanotip B, small Fe clusters (~3 to 10 nm diameter) are present throughout the nanotip, with the particles becoming less frequent towards the bottom. In nanotip C, there is small amount of $npFe^0$ in the very top of the nanotip (not displayed in Figure 1), above the Ti-depleted zone, and near the bottom of the nanotip. The middle of nanotip C is dominated by a large (>30 nm diameter) Fe particle (SMFe) of almost pure Fe, surrounded by a rim of TiO. Nanotip C also contains a void space (~15 nm in diameter), which we interpret as a vesicle. No noble gases were detected in this vesicle, likely because they were simply lost during FIB-milling. Noble gases in extraterrestrial samples present as a product of space weathering have yet to be detected by APT because no one has tried to date.

In summary, we have demonstrated that APT can be successfully used to analyze natural samples to characterize products of space-weathering compositionally and texturally at near-atomic resolution.

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