NANOSCALE ANALYSES OF VESICLES IN SPACE-WEATHERED LUNAR SOIL SILICATES AND ILMENITE. J. Greer[1,2,*], A. M. Kling[3], D. Isheim[4], D. N. Seidman[4], M. S. Thompson[3], P. R. Heck[1,2], [1]Department of the Geophysical Sciences, University of Chicago, Chicago IL USA; [2]Robert A. Pritzker Center for Meteoritics and Polar Studies, Negaunee Integrative Research Center, Field Museum, Chicago IL USA; [3]Department of Earth, Atmospheric, and Planetary Sciences, Purdue University, West Lafayette IN USA; [4]Northwestern University Center for Atom-Probe Tomography (NUCAPT), Department of Material Science & Engineering, Northwestern University, Evanston IL USA; *jgreer@fieldmuseum.org

Introduction: Space weathering is the alteration of the outer surfaces of grains on airless bodies when irradiated by cosmic rays, the solar wind, electromagnetic radiation, and impacts from micrometeorites. Vesicles are one of the features that can form when a mineral grain is exposed to the space weathering environment, and it has been suggested that the solar wind is particularly important in the development of these features, implanting H and He ions that concentrate in these void spaces [1,2]. Atom probe tomography (APT) is a technique that field-evaporates atoms on a sample nanotip and uses time-of-flight and a position-sensitive detector to recreate the sample in 3D, and is an ideal method to investigate the outermost ~100s of nanometers of the surfaces of space weathered samples (e.g., [3,4]). The exposure to solar wind may be particularly important as this irradiation leads to the formation of vesicles. Samples exposed for a long time to space weathering are termed mature and contain a higher density of vesicles. Here we present APT analyses of mature lunar soil in contrast to our previously published data on submature soil 7150 [3], with a specific emphasis on the structure and composition of the vesicles.

Methods: Apollo 17 sample Soil 79221 is classified as mature and has previously been used to study space weathering on the moon, e.g., [6]. Using a FIB-SEM microscope at the University of Chicago, a 1 µm × 10 µm area of interest was coated with Pt to protect it from damage by the FIB, and a lamella was lifted out of 4 target grains with compositions identified as clinopyroxene, plagioclase, olivine, and ilmenite. These lift-outs were performed close to the location of lamellas extracted for TEM to allow for correlative analysis [7], but far enough away to avoid damage from previous FIB sample preparation. Out of each lamella, 4 to 5 nanotips were prepared using established APT sample preparation methods [8] and analyzed with a CAMECA LEAP 5000XS at the NUCAPT facility of Northwestern University. The detection efficiency of this instrument is 80%. Reconstructions were done with IVAS.

Results: A total of 14 nanotips were analyzed for their space weathering features, with 8 providing useful data. From the four different materials analyzed for this project, only the ilmenite and olivine nanotips contained vesicles and also remained stable enough to capture the vesiculated texture before failure. If the vesicle comprised a significant volume of the nanotip, the nanotips fractured before an analysis reached the lower half of the vesicle.

Clinopyroxene: There were no vesicles represented in the volumes of decreased density in the two clinopyroxene nanotips. Instead, these nanotips are dominated by Fe-rich structures, including whisps of increased oxidation and a microphase Fe particle. These Fe and FeO-rich features are depleted in H [7], and in the two nanotips that we analyzed, do not correspond to any vesicles or void-like features.

Plagioclase: Nanotip failure usually occurred when the sample transitioned from the Pt cap to the plagioclase target grain. A small volume of plagioclase was analyzed, but the volume is too small to identify vesicles that can easily be distinguished from the surrounding matrix. In general, plagioclase was a difficult material to analyze and requires more optimization for APT analysis.

Olivine: This particular grain is Mg rich. Several nanotips preserved the outermost surface of the grain (Fig. 1), but lack nanophase Fe particles. Other nanotips had zones of decreased density that could be interpreted as vesicles. These zones were enriched in H species relative to the other elements present in olivine.

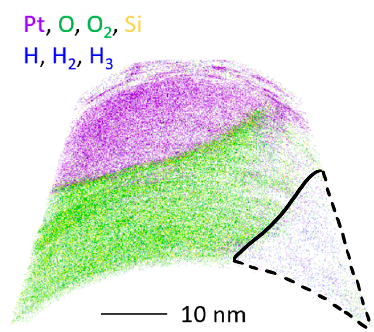

Fig. 1: A cross-section through an APT reconstruction of an olivine grain from lunar soil 79221, with individual ions shown as dots (capping Pt in purple, O and O_2 in green, Si in yellow, and H species in blue). In the bottom right of the cross-section, a vesicle is present (black outline). This is represented by a decreased volume concentration of O and Si species and an increased concentration of H species.

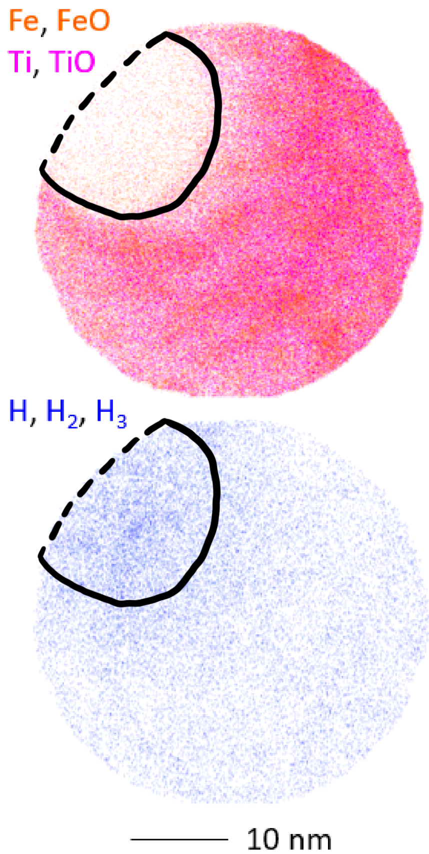

Fig. 2: A cross-section of an APT reconstruction of a lunar ilmenite soil grain, with individual ions shown as dots (Fe and FeO in orange, Ti and TiO in magenta, and H species in blue). In the upper left of the cross-section, a vesicle is present (outlined in black). This is represented by a decreased volume concentration of Fe and Ti species (top) and an increased concentration of H species (bottom).

Ilmenite: The ilmenite nanotips contained large vesicles taking up a significant volume of the nanotips. Ilmenite typically exhibits a heterogenous distribution of Fe and Ti species. A small burst of ions at mass-to-charge-state ratio 3, interpreted as H_3, was detected at the top of the void.

Discussion: Each nanotip presents a unique composition and structure, even though they are only separated by 1 µm on the grain's surface, consistent with what we described in [3]. Our previous APT study [3] identified a void structure in a lunar ilmenite grain from Apollo 17 submature sample Soil 71501 with a possible ^{4}He detection (3±2 atoms, 1σ, above background). The previously detected vesicle was similar in size (~8 nm × 14 nm) to the ilmenite nanotip presented in Fig. 2 (~11 nm × 16 nm). This sample (Fig. 2) also included a possible He signal. The burst at the top of the vesicle included an increased detection of H_2 and H_3 and ^{4}He (7±3, 1σ, above background; ^{3}He is too low in abundance to be detected here). Like the nanotip analyzed in [3], this sample is missing at least 3 orders of magnitude of He atoms when compared to bulk analyses [9]. Although this is significantly less than the expected He content for the vesicle if He primarily resides in vesicles, the peak in the mass spectrum is above the background for an equivalent sized region of interest measured anywhere else in the nanotip, which ranges from 0 to 2 counts. This indicates the opening and degassing of the vesicle as the top of the nanotip eroded was during analysis. The increased concentration of H and He species detected at the top of these vesicles suggests that the gaseous contents largely escaped into the vacuum without being analyzed, though some of these atoms or molecules were ionized and detected, resulting in the increased detection.

The detection of He in vesicles in space weathered samples using APT remains a challenge as closed vesiculated textures in space weathered samples have non-ideal geometries. The topography of the nanotip is not symmetrical and hemispherical due to the presence of these vesicles, which may change the local electric field in ways that lead to nanotip failure. Vesicles that are open to the nanotip's surface and not fully infilled with capping Pt may introduce an instability that leads to nanotip failure during analysis.

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