

COORDINATED TEM AND APT ANALYSES TO UNDERSTAND THE DISTRIBUTION OF SOLAR WIND-SOURCED HYDROGEN AND WATER IN SPACE WEATHERED LUNAR SOILS

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Introduction: Remote sensing observations [1,2], laboratory experiments [3,4], and computational simulations [5] each predict that OH/H₂O (hereafter water) can form in nominally anhydrous minerals on airless bodies due to space weathering. Space weathering is driven by solar wind irradiation and micrometeoroid bombardment and both mechanisms can change the optical, chemical, and microstructural properties of grains on airless surfaces [6]. Microstructural space weathering features include amorphous rims (upper ~100 nm), reduced and/or oxidized nanophase iron (npFe) particles, and vesicles that may form from the coalescence of solar wind implanted H and He [7]. Solar wind H⁺ ions can combine with O to form water species in surface minerals. Electron energy loss spectroscopy (EELS) has previously been used to identify such hydrogen and water species in the rims and vesicles of space weathered interplanetary dust particles and lunar regolith grains [8,9]. Atom probe tomography (APT) has also been used to identify hydrogen and water in the rims and vesicles of space weathered olivine from Itokawa [10] and lunar regolith grains [11,12,9]. However, understanding the abundance of hydrogen and water species in space weathered lunar grains and their relationship with microstructural space weathering features and mineralogy is still at an early stage. Here, we present results of coordinated EELS and APT analyses to understand the distribution of hydrogen and water in relation to microstructural space weathering features and mineralogy.

Methods: In this work we analyzed grains from mature lunar mare soil 79221. Individual grains were transferred to an aluminum stub coated in carbon tape and subsequently sputter coated with Pt. We used scanning electron microscopy (SEM) to identify grains 100-200 µm in diameter that displayed vesicles or bubbles on their surface as evidence of solar wind irradiation. An agglutinate particle, clinopyroxene, olivine, plagioclase, and ilmenite grains were identified using energy dispersive x-ray spectroscopy (EDS). We then prepared electron transparent focused ion beam (FIB) cross sections of each grain with the Helios G4 UX Dual Beam FIB-SEM at Purdue University for analysis in the transmission electron microscope (TEM). Adjacent FIB sections were also collected from each grain to produce nanotips sharpened to have an apex radius of ~30 nm for APT analyses.

High-resolution TEM (HRTEM) and high-angle annular dark field (HAADF) images were collected to characterize the microstructural space weathering features for FIB sections from each grain using the Thermo Scientific Themis Z monochromated and aberration-corrected TEM equipped with a Gatan Quantum 965 EELS detector at Purdue. Low-loss EELS spectra were collected as linescans across individual vesicles and within the amorphous rims of these grains to identify the presence of hydrogen and water via the hydrogen core scattering edge (H-K), energy gap of water (EG), and ionizing threshold of water (IT) features in the low-loss energy region.

APT analyses were performed on tips made from each sample using the CAMECA LEAP 5000XS tomograph at Northwestern University. 3-D reconstructions of elemental information of each tip were analyzed and the presence of H, OH, and H₂O were identified based on their mass-to-charge ratios. We then created depth profiles of each of these species to identify their change in concentration with depth.

Results and Discussion: EELS analyses of an agglutinate grain revealed the presence of the H-K, IT, and EG features in both the space weathered rim and vesicles indicating the presence of hydrogen and water. APT analyses also identified the presence of H, OH, and H₂O in the agglutinate grain and an olivine grain. Depth profiles in both grains reveal an enrichment in all three species in the space weathered rims which exhibit a steady decrease in concentration with depth into the grain. These profiles are similar in shape to the space weathered Itokawa olivine from [11] but differ from the observed shape in immature lunar ilmenite from [12]. The presence of H, OH, and H₂O in vesicles and in space weathered rims provides evidence that solar wind implantation is a viable mechanism to form water on the lunar surface. Further EELS and APT measurements to characterize the relationship between water and microstructural features and comparisons in concentration profiles and abundance across mineralogy are ongoing.

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