doi:10.1017/S1431927621008151
Microsc. Microanal. 27 (Suppl 1), 2021

Coordinated analysis of space weathering characteristics in lunar samples to understand water distribution on the Moon

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Background

Space weathering processes affect the outermost rims (100 nm) of grains on the surfaces of airless planetary bodies, such as the Moon. Driven by solar wind irradiation and micrometeorite bombardment, space weathering alters the optical, chemical, and microstructural properties of these grains [1]. Microstructural changes in space weathered samples include vapor and melt deposits formed by micrometeorite impacts and amorphous rims formed by solar wind irradiation. These rims may also contain nanoparticles, predominantly composed of reduced Fe metal, called nanophase iron (npFe), and vesicles that may be formed by the coalescence of H and He implanted by the solar wind [2,3]. Previous work has shown that space weathered rims of interplanetary dust particles (IDPs) can also contain vesicles and amorphous zones formed by solar wind irradiation. Valence electron energy loss spectroscopy (VEELS) measurements of these IDPs revealed that the vesicles and amorphous rims produced by solar wind irradiation contain water in both the H2O and OH molecular forms [4]. Further, studies that have simulated solar wind implantation into the lunar regolith have predicted that this irradiation forms crystal defects within the implantation layer and that hydrogen gets trapped in these sites to then react and form water in either the H2O or OH form [5,6]. However, the precise nature of the relationship between space weathering and the formation and retention of water within space weathered rims is still unknown. We present preliminary analyses of the space weathered rims of lunar soil particles with plans to conduct a series of coordinated analyses of including atom probe tomography (APT) and transmission electron microscopy (TEM) analyses to better characterize the relationship between water and space weathering characteristics.

Methods

Sample 79221 is a mature lunar mare sample collected from the Apollo 17 mission. Maturity is an indicator of how long the grains were exposed to the space environment on the lunar surface – and is therefore a proxy for the degree of space weathering that the sample has undergone. Individual lunar regolith grains of lunar sample 79221 were first mounted onto carbon tape on a scanning electron microscopy (SEM) stub and sputter coated with Pt. For preliminary characterization, the sample was imaged in a Hitachi TM 4000 Plus benchtop SEM at Purdue University equipped with backscatter (BSE) and secondary (SE) detectors and an Oxford 30 mm2 energy-dispersive X-ray spectroscopy (EDX) detector. Further analyses were performed using the FEI Nova NanoSEM equipped with an Oxford INCA Energy 250 EDX detector at Purdue. We targeted grains 100-200 µm in diameter that displayed vesicles or bubbles on the surface (Fig. 1). These microstructural features may indicate the grain has experienced solar wind irradiation. Once identified, focused ion beam (FIB) sections were extracted from the grain and thinned to < 100nm using the Helios G4 UX Dual Beam scanning electron microscope at Purdue. A protective Pt cap of ~ 10 µm × 1.5 µm × 1 µm was first deposited by an electron beam (to protect the surface of the grain) and then an ion beam before trenching. Trenches were milled to a depth of 10 µm into the sample to obtain both rim and material (~100 nm thick) and material from the grain interior.

Once the FIB section was extracted and thinned, we began characterizing the microstructural features of the lunar soil rim in the 200 keV Tecnai T20 transmission electron microscope at Purdue University to investigate the microstructural and chemical characteristics of the grain.

Results

We have acquired high-resolution TEM (HRTEM) images of the rim to characterize evidence of space weathering (Fig. 2). These images show the presence of an amorphous rim of uniform thickness (~60 nm) extending the entire length of the FIB section. The thickness of this amorphous rim is similar to the penetration depth expected for H+ and He+ ions from the solar wind [7]. Distributed throughout this amorphous rim are nanoparticles measuring between 5 and 15 nm in diameter and the grain underlying this rim is crystalline. We are currently performing EDX mapping to understand the composition of this rim and its relationship to the grain interior.

After we complete the initial characterization of this grain, we will transfer the lunar samples to the University of Chicago where APT tips will be prepared with the TESCAN LYRA3 FIB. The APT tip will be extracted from the region of the grain adjacent to the location of the TEM FIB liftout to ensure a likelihood of continuity between the microstructure of the two regions to enable correlation of TEM and APT measurements. APT analyses of the tips will be performed using the LEAP 5000X Si tomograph at Northwestern University.

Future TEM analyses include electron energy loss spectroscopy (EELS) measurements as linescans across individual npFe nanoparticles to determine their oxidation states following the methodology of [8]. These EELS spectra will then be compared to standards of Fe0, Fe2+, and Fe3+ to determine the contribution of each oxidation state to the nanoparticle composition. We will also attempt to measure H, He, and water signatures by collecting VEELS spectra of vesicles and amorphous regions adjacent to other space weathering features following the methodology of [4].

Acknowledgements

This work is supported by NASA Solar System Workings grant 80NSSC20K0863 (MST).

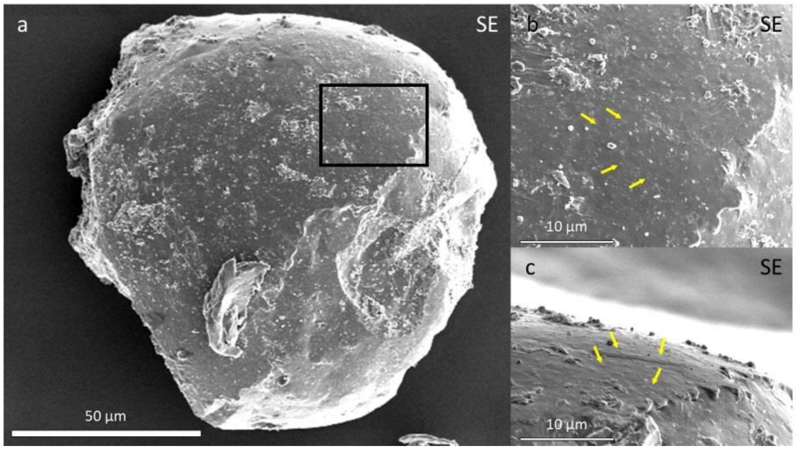

Figure 1. Secondary electron (SE) images of a grain from lunar sample 79221. a) Image of the entire grain of interest. b) Inset region of the grain where FIB section was extracted. Yellow arrows indicate hummocks on the surface which may be surface expressions of vesicles within the rim. c) An oblique view of the same region as b. Yellow arrows again indicate hummocks on the surface which may be surface expressions of vesicles within the rim. A protective Pt cap has been electron-beam deposited in the target area for FIB extraction to protect from beam damage.

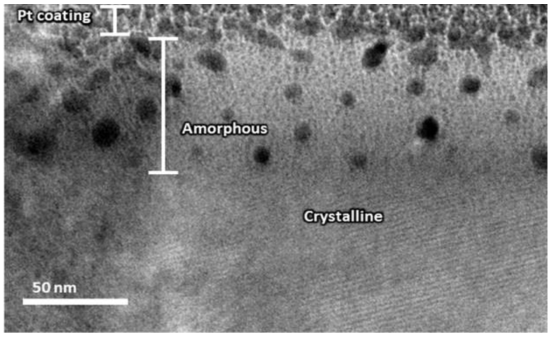

Figure 2. HRTEM of a lunar soils grain showing a crystalline interior and an amorphous rim ~60 nm thick containing nanoparticles.

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