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Developing a Methodology to Analyze Volatiles in Frozen Lunar Samples in Preparation for Artemis Sample Return.

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The widespread presence of water on the lunar surface was revealed by remote sensing observations [1] and confirmed by the analysis of returned Apollo samples [2]. This water has been proposed to originate on the lunar surface through several mechanisms, including via solar wind implantation through space weathering processes. However, our understanding of the life cycle of water and other volatiles on the Moon is still at an early stage. With the NASA Artemis missions planning to return volatile-rich, frozen samples from the lunar South Pole, developing techniques to characterize their volatile content will be crucial for building a model for the life cycle of these volatile species on the surface of the Moon. To develop this analytical pipeline, we prepared a spaced weathered ilmenite grain from Apollo 17 mature soil 79221 for analysis using a cryogenic focused ion beam scanning electron microscope (FIB-SEM). We collected high resolution images and conducted electron energy loss spectroscopy (EELS) on the ilmenite FIB section using a cryogenic transmission electron microscope (TEM). We observed nanophase iron particles and microstructural defects like vesicles within the space weathered rim of the grain. We acquired EELS spectra from within the vesicles to identify the potential presence of water and hydrogen. We positively identified an H K-edge at ~13 eV in cryogenic EELS spectra collected from within the vesicles. Going forward, we would like to further enhance this end-to-end cryogenic pipeline by transferring the sample from the FIB to the TEM in a cold glovebox. Ultimately, we will utilize this analytical methodology on experimentally irradiated analog mineral phases (olivine, pyroxene, ilmenite, and plagioclase) under cryogenic conditions and gradually warm them to ambient temperatures in 50-degree increments and reanalyze at each stage. The irradiation and analysis of plagioclase is underway, and we will present the results at the meeting. This will allow us to understand the migration or loss of volatiles under varying thermal conditions that might be expected on the lunar surface.

References: [1] Pieters, C.M. et al. (2009) *Science,* 326*,* 568-572. [2] Liu, Y. et al (2012) *Nature Geoscience,* 5*,* 779-782.