SIMULATING MICROMETEOROID BOMBARDMENT OF SULFIDES VIA IN SITU HEATING IN THE TRANSMISSION ELECTRON MICROSCOPE.

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Introduction: The surfaces of airless bodies continually experience hypervelocity dust impacts from micrometeoroids and irradiation via energetic ions from the solar wind. Together these processes are known as *space weathering*, and they affect the microstructural, chemical, and spectral characteristics of regolith on airless surfaces [1,2]. Our studies of space weathering have long focused on how these processes affect silicate minerals which comprise the bulk of returned samples from the Moon. Analyses of lunar soils grains in the transmission electron microscope (TEM) revealed nanoscale space weathering products including amorphous rims, vesicles, and nanophase Fe particles (npFe) [3]. However, more recent sample return missions have targeted asteroidal surfaces (e.g., Hayabusa, Hayabusa2, and OSIRIS-REx) and returned materials with more diverse compositions, including sulfide minerals. Sulfides are thought to play a critical and active role in asteroidal surface processes as samples from the Hayabusa and Hayabusa2 missions revealed the presence of npFeS, and npFeNiS particles in space weathered grain rims [4,5]. In addition, unique microstructural features described as Fe whiskers have been identified in samples from Itokawa and the Moon [6,7]. Hypothesized to form via solar wind irradiation and implantation of H^+, these whiskers are depleted in S and have a filamentous structure that protrudes from the surface of sulfide grains [6,7]. To better understand how sulfides are space weathered and to decipher the origin of characteristics like Fe-whiskers, here we report results of simulated micrometeoroid bombardment of pentlandite via in situ heating in the TEM.

Methods: We dropcast grains of pentlandite suspended in isopropanol onto microelectromechanical systems (MEMS) heating chips with SiN support films. In situ heating using the Hitachi Blaze heating holder was performed under vacuum in the aberration-corrected 200 keV Hitachi HF5000 scanning TEM (STEM) at the University of Arizona. The HF 5000 is equipped with bright-field (BF), dark-field (DF), and secondary electron (SE) detectors as well as dual Oxford 100 mm^2 windowless silicon-drift energy dispersive X-ray spectroscopy (EDX) detectors and a Gatan Quantum Imaging Filter (GIF) for electron energy-loss spectroscopy (EELS). We subjected sulfide grains to fast (<0.5 s) thermal pulses up to 1100 °C (up to three total pulses) to simulate the short-duration, high-temperature effects of micrometeoroid impacts [8]. We imaged the samples before and after heating, recorded videos of the samples during the thermal events, and analyzed changes in grain chemical composition by collecting EDX maps. We then used the FEI Helios NanoLab 660 focused ion beam scanning electron microscope (FIB) at the University of Arizona to extract an electron transparent thin section spanning multiple whisker-like structures that developed on the sample surface during heating. We performed high resolution (HRTEM) imaging and selected area electron diffraction (SAED) to understand the whisker microstructure. We also collected EDX maps and linescans to understand changes in sample chemistry, and EELS spectrum images to identify changes in Fe oxidation state as a result of heating.

Results and Discussion: After the first thermal pulse, many pentlandite grains developed microstructural features that protruded from the surface of the samples. These structures are striated, enriched in Fe and Ni and depleted in S, and exhibit near-identical morphologies and sizes to the whiskers observed in returned samples [6,7]. Subsequent thermal pulses contributed to the destruction of some of the whiskers. TEM observations of the FIB section indicate that the whiskers are predominantly amorphous with pockets of nanocrystallinity. Some of the whiskers contain crystalline euhedral or lenticular particles ranging in size from 20-100 nm in length. EDX maps of the FIB section indicate that the nanoparticles are strongly enriched in Fe and Ni, and that some whiskers are completely depleted in S while others show migration of S into isolated pockets at the exteriors of the whisker structures. There are also micron-scale features below the grain surface that are depleted in S and enriched in Fe and Ni, suggesting that the migration of S as a result of heating events is not solely surface-correlated. These results suggest that micrometeoroid bombardment could serve as a formation mechanism for Fe-bearing whiskers on the surfaces of airless bodies.

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