IN SITU HEATING EXPERIMENTS ON PATINA COATED LUNAR ROCK 76015.

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Introduction: We are undertaking a series of *in situ* heating experiments in a transmission electron microscope to constrain several characteristics of space-weathered sufaces from airless bodies. These studies are focused on understanding 1) the preservation and modification of solar flare particle tracks in silicates, 2) thermal effects associated with the evolution and loss of solar wind implanted species, and 3) alteration of vapor-deposited and solar wind damaged rim layers on rocks and grains. These studies are particularly important for analyses of returned samples from the Moon, Itokawa, and future sample return missions. Here, we report on a dynamic *in situ* heating experiment from a lunar sample and evolution of solar wind implanted gases and associated microstructural changes.

Samples and Methods: We used a FEI Quanta3D focused ion beam (FIB) instrument at JSC to prepare a thin section extracted from lunar sample 76015 that was collected from the Apollo 17 Station 6 boulder The region of interest was dominated by orthopyroxene (opx) with little to no patina covering its surface. To avoid ion-beam damage to the opx surface, we first used electron beam-assisted deposition to lay down the first 500 nm of the carbon protective strap, followed by several micrometers of ion beam-assisted deposition of additional carbon. We performed an *ex situ* lift-out of the section and placed the section on one of the elements of a specialized heating substrate and attached the section to the substrate by depositing small carbon straps with the FIB (Fig. 1). The heating chips used in this study are manufactured by Norcada Inc. and consist of microelectromechanical systems (MEMS) platforms that utilize electron transparent silicon nitride windows to support the samples and provide uniform heating while enabling transmission electron microscope (TEM) imaging. The MEMS chip was loaded into a Hitachi "Blaze" heating holder and analyzed using a Hitachi HF5000 at the University of Arizona. The Hitachi HF5000 is a 200 keV aberration-corrected scanning and transmission electron microscope (STEM) equipped with brightfield (BF), darkfield (DF), and secondary electron (SE) detectors as well as Oxford dual-side-entry 100 mm^2 Si-drift detectors giving a large (1.7 sr) solid angle.

For the dynamic heating run, we heated the sample in steps with pauses at certain temperatures to allow the sample to equilibrate. The heating profile began at room temperature with a 1°C/s ramp to 200°C with additional pauses at 400, 600, 700, and 800°C. The elapsed time for the experiment was ~25 min. We monitored the sample by acquiring simultaneous BF, DF, and SE STEM images. Temperature calibration for the MEMS chips is achieved using the melting point of metallic Ge.

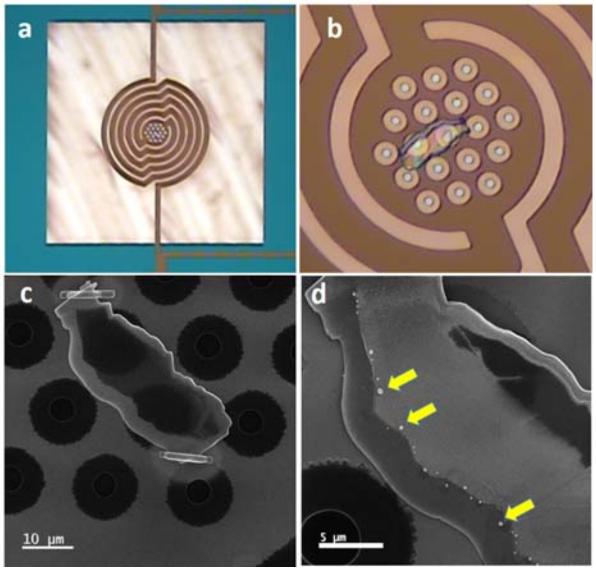

Figure 1. a) the MEMS heating chip. b) reflected light image showing the FIB section plced on the heating chip. c) SEM image of the FIB section attached to the heating chip with C straps. d) FIB section after heating to ~280°C showing the development of Ga metal particles at the interface between the protective strap and the opx (yellow arrows).

Results and Discussion: We observed no changes in the sample up to 200°C. At ~280°C we began to see the growth and coalescence of Ga metal into discrete μm-sized particles from the protective carbon strap (Fig. 1d). Ga metal has a melting point of ~30°C. No obvious changes to the opx were observed until the 600°C pause when nanophase Fe particles began to form within the opx (Fig. 2) at a depth of ~0.4 μm and deeper in the section (relative to the rock surface). Previous dynamic heating experiments on lunar soil grains using similar heating substrates observed that nanophase Fe metal inclusion began to form at ~575°C [1]. With heating to 700°C the number and density of nanophase Fe inclusions continued to increase across the surface of the FIB section, especially along microcracks (Fig. 3). During the heating ramp between 700°C and 800°C, we observed the formation of submicrometer-sized pores and vesicles in the opx starting at ~750°C and becoming denser and more numerous up to 800°C where the experiment was ended (Fig. 4). The vesicles are concentrated in the outer ~1 μm of the section and likely form by the nucleation and growth of bubbles that eventually burst initially at 750°C. The formation temperature of these vesicles is consistent with gas release profiles from stepped-heating experiments from noble gas studies of lunar soils [e.g. 2]. These studies show that >50% of the noble gases are released at temperatures <800°C. The presence of vesicles far beyond the implantation depth of solar wind species (Fig. 4) implies inward diffusion of these species over longer timescales on the lunar surface.

Conclusions: Stepped heating of a lunar sample *in situ* in a STEM allowed detailed observations of the microstructural evolution of a space weathered rock surface. We observed the formation of nanophase Fe metal particles and the formation of vesicles in opx from the surface of lunar rock 76015. These preliminary data and our follow-on analyses will allow us to further explore thermal processing of regolith materials affected by impact processes.

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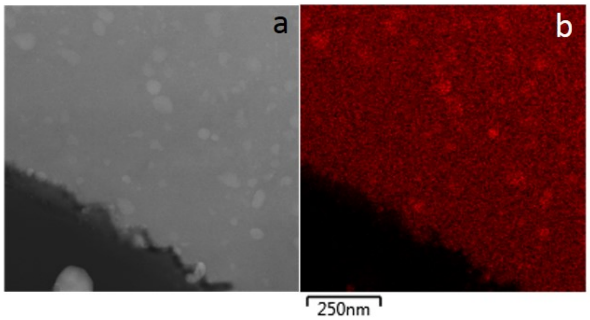

Figure 2. a) annular darkfield image of the FIB section heated to 800°C showing numerous nanophase Fe metal grains distributed throughout the opx. b) Energy-dispersive x-ray Fe map of the region in a).

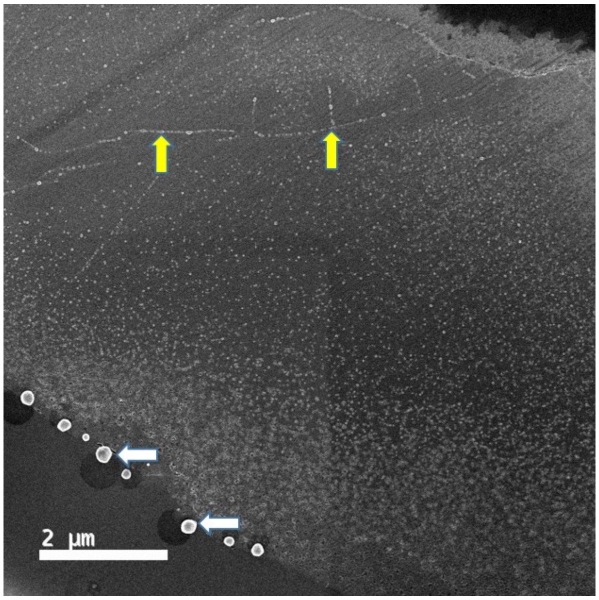

Figure 3. Secondary electron image (SEI) of the FIB section heated to 800°C showing Ga metal droplets at the opx-carbon strap interface (white arrows), and numerous nanophase Fe metal grains distributed throughout the opx, but especially along cracks (yellow arrows).

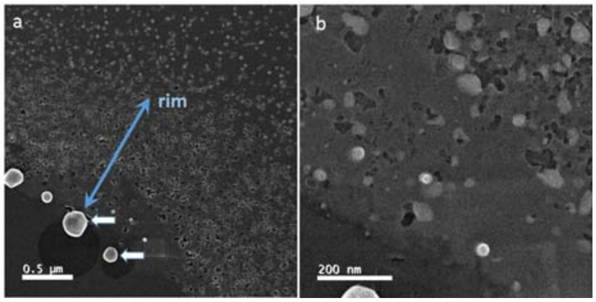

Figure 4. A SEI showing the irregular nature of the vesicles in the opx sample and the 10-20 nm nanophase Fe metal particles in the opx heated to 800°C. The vesicles are concentrated in an ~1 μm wide rim (blue arrow).