SPACE WEATHERING EFFECTS REVELAED THROUGH IN SITU ION IRRADIATION AND HEATING OF THE MURCHISON METEORITE IN THE TRANSMISISON ELECTRON MICROSCOPE.

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Introduction: Solar wind irradiation and micrometeoroid bombardment are the primary space weathering processes that alter airless planetary regoliths [1]. In addition to returned sample analysis, analog laboratory experiments help constrain the microstructural, chemical, and optical effects of space weathering. Ion irradiation experiments simulating solar wind irradiation and pulsed laser or heating experiments simulating micrometeoroid impacts are typically performed *ex situ* [e.g., 2,3]. However, in the last decade, *in situ* transmission electron microscopy (TEM) has been leveraged to monitor real-time morphological, microstructural, and chemical modifications resulting from simulated space weathering in lunar soils, meteorites, and individual mineral phases. These investigations produced vesiculation, amorphization, chemical heterogeneity, whisker formation, and compositionally diverse Fe-bearing nanoparticles (npFeS, npFeNi, npFeSi, $npFe^0$) [e.g., 4-7].

Understanding space weathering of carbonaceous asteroidal regolith is particularly important for maximizing the science return of the Hayabusa2 and OSIRIS-REx missions, which have visited and collected samples from the surfaces of C-complex asteroids Ryugu and Bennu, respectively. Previous *in situ* TEM studies that utilized carbonaceous chondrites as analogs for C-complex asteroid surfaces focus on the effects of parent body thermal alteration rather than space weathering [e.g., 8]. Here, we performed *in situ* H^+ and He^+ irradiation with subsequent rapid heating experiments on the Murchison CM2 carbonaceous chondrite to investigate the combined effects of progressive solar wind and micrometeoroid bombardment on carbonaceous regoliths.

Methods: Murchison dust was crushed into a fine-grained powder using an agate mortar and pestle and then suspended in methanol. The grains were drop-cast onto DENSsolutions Si_3N_4 Wildfire nanochips (Figure 1). Using the *in situ* capabilities of the 200 keV FEI Tecnai G2 F30 TWIN scanning transmission electron microscope (STEM) at the University of Michigan Ion Beam Laboratory (MIBL), we irradiated one sample with 13.2 keV H^+ up to a total fluence of 1.5×10^{17} H^+/cm^2 using a flux of 9.0×10^{12} $H^+/cm^2/s$ and another sample with 23 keV He^+ ions up to a total fluence of 9.0×10^{16} He^+/cm^2 using a flux of 1.4×10^{13} $He^+/cm^2/s$. Following irradiation, each sample was subjected to three consecutive *in situ* rapid heating events. The samples were heated to 1100°C at a rate of ~100°C/s and immediately brought back down to room temperature (21°C). Microstructural changes were evaluated with a combination of bright field (BF) TEM, high-resolution TEM (HRTEM), and selected area electron diffraction (SAED) before and after ion irradiation and the first and third heating events. Videos of the experiments were acquired in BF TEM mode. Samples were also imaged at fluence intervals during H^+ and He^+ irradiation. Chemical analysis was performed by acquiring energy-filtered (EFTEM) and electron energy loss spectroscopy (EELS) maps using the attached Gatan Continuum ER GIF system.

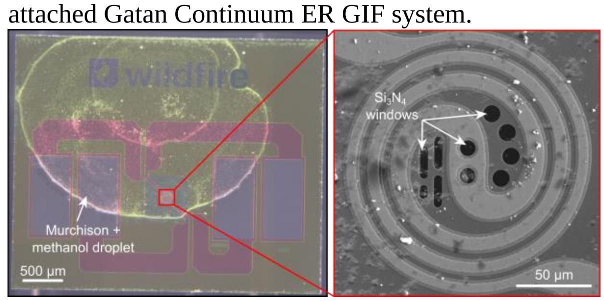

Figure 1. *(Left) Reflected light image of the Wildfire nanochip after sample deposition. (Right) Secondary electron image of the area indicated by the red box at left showing the Si_3N_4 viewing windows and Murchison dust particle distribution.*

Results and Discussion: Microstructural and chemical analysis of the He^+-irradiated and heated sample is ongoing. Here, we discuss results from H^+ irradiation and subsequent rapid heating experiments.

H^+ Irradiation: Vesiculation was not observed in Murchison grains during H^+-irradiation. Even after achieving the final fluence of 1.5×10^{17} H^+/cm^2, vesicles were not present. This finding contrasts with results from [9] in which irradiation of olivine with 1 keV H^+ at a flux on the order of 10^{13} $H^+/cm^2/s$ caused blistering at a fluence of 1.0×10^{17} H^+/cm^2. HRTEM images and SAED patterns suggest that H^+ irradiation causes partial amorphization of phyllosilicates. Some grains display lattice fringes with d-spacings consistent with the (001) plane of cronstedtite (e.g., 7.09 Å). These lattice fringes disappear after the grain has been irradiated to a fluence of 8.6×10^{16} H^+/cm^2 (Figure 2). Amorphization of anhydrous silicate phases (e.g., olivine and pyroxene) was not observed.

Rapid Heating: Subjecting the H^+-irradiated Murchison particles to one rapid heating event (1x heated sample) causes widespread formation of vesicles and nanoparticles (Figure 3). In addition, phyllosilicates become completely amorphous. Individual vesicles are spherical and ovoid in shape and range in size from ~10-60 nm in their longest diameter. Considering vesicles are a characteristic feature of space weathering in lunar and Itokawa regolith grains, this experiment suggests

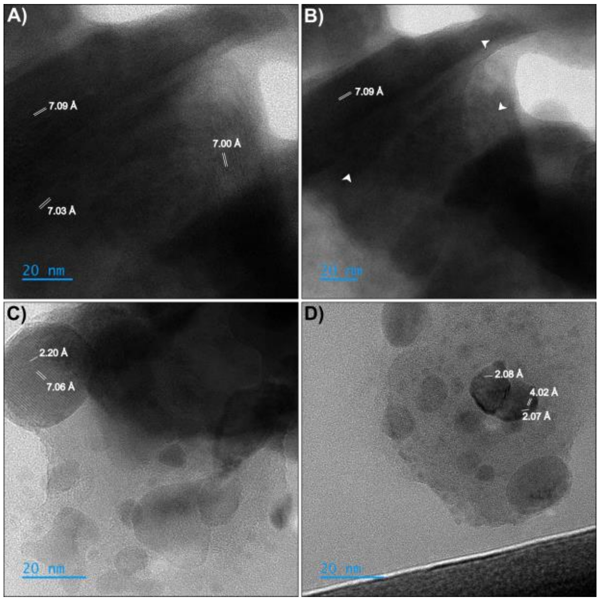

Figure 2. *A) BFTEM image of Murchison particle before H^+ irradiation showing crystalline phyllosilicate lattice fringes with d-spacings consistent with cronstedtite. B) BFTEM image after irradiating up to a fluence of 8.6×10^{16} H^+/cm^2 showing partial amorphization of phyllosilicates. White arrows indicate regions where lattice fringes are no longer present. C) BFTEM image of the 3x heated sample showing nanoparticles set in an amorphous silicate matrix. D-spacings of the top left nanoparticle are consistent with pentlandite. D) BFTEM image of the 3x heated sample showing nanoparticles coalescing due to successive rapid heating.*

that heating events may aid in the formation of vesiculated textures on airless bodies.

The majority of nanoparticles in the 1x heated sample are ≤ ~15 nm in size. Performing two additional rapid heating events (3x heated sample) causes existing nanoparticles to coalesce and grow in size, similar to what was observed in consecutive rapid heating of lunar soil grains [8]. Figure 2D shows two merging nanoparticles in the 3x heated sample with different crystallographic orientations. Nanoparticles in both the 1x and 3x heated samples exhibit spherical to subhedral shapes [8]. Interestingly, some vesicles that were present in the 1x heated sample are no longer present in the 3x heated sample (Figure 3). We hypothesize that these vesicles were destroyed by heating-induced outgassing of implanted H^+ [10].

In contrast to the predominantly Fe metal nanoparticles found in returned lunar samples, measured d-spacings and EELS/EFTEM elemental maps from this study suggest that rapid heating of Murchison forms nanoparticles with a variety of compositions that may include Fe metal, pentlandite, troilite, and pyrrhotite. A similar diversity in nanoparticle mineralogy was observed in laser irradiated Murchison samples and Ryugu returned samples [e.g., 11-13]. Collectively, these results show that in situ ion irradiation and heating techniques can be used to successfully reproduce characteristic features of space weathering and confirm the complex nature of space weathering of carbonaceous asteroidal regoliths.

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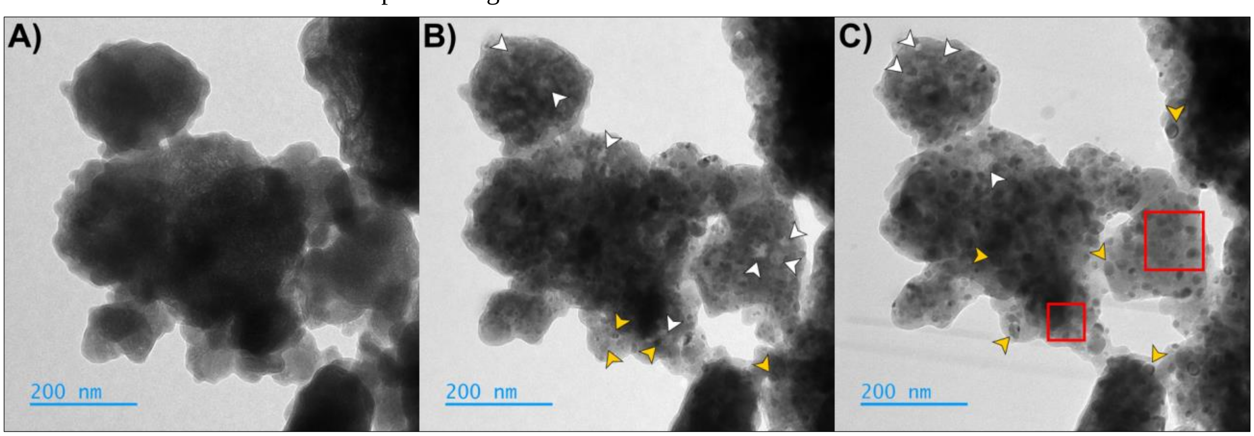

Figure 3. *BFTEM images of a Murchison particle after (A) H^+ irradiation, (B) 1x rapid heating, and (C) 3x rapid heating. (A) No vesicles form from H^+ irradiation. (B) White arrows indicate vesicles formed from 1x rapid heating. (C) Red boxes indicate regions where vesicles were destroyed from 3x rapid heating. White arrows denote vesicles. Yellow arrows show examples of subhedral nanoparticles in both (B) and (C).*