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He^+ irradiation and heating of the Murchison meteorite using *in situ* transmission electron microscopy

The Joseph Goldstein Scholar Award funded my research trip to the University of Michigan Ion Beam Laboratory (MIBL). Here, I used *in situ* transmission electron microscopy (TEM) to perform ion irradiation and heating experiments on the Murchison carbonaceous chondrite meteorite to investigate the effects of space weathering on carbon-rich asteroids. Surfaces of airless planetary bodies, like the Moon and asteroids, are continuously altered by energetic solar wind ions (H^+ and He^+) and hypervelocity dust impacts (i.e., micrometeoroid impacts). Collectively known as space weathering, these processes change the spectral, microstructural, and chemical properties of planetary regoliths. Although carbon-rich asteroids are thought to be the most common compositional class of asteroids in our solar system, we know relatively little about the optical and physiochemical effects of space weathering on these bodies. Laboratory simulations of space weathering performed on carbonaceous chondrite meteorites, like Murchison, are one way to better understand this phenomenon.

Typically, laboratory experiments simulating solar wind exposure or micrometeoroid bombardment are performed *ex situ*. Although transmission electron microscopy (TEM) is the preferred analytical tool for characterizing the nanoscale physiochemical products generated by these experiments, relatively few studies have leveraged the *in situ* capabilities of some TEMs to monitor these changes in real-time. With assistance from Dr. Kai Sun, I performed *in situ* experiments with MIBL's 300 kV FEI Tecnai G20 F30 TEM, which is connected to two ion beamlines and equipped with a DENSsolutions MEMS heating holder. Samples were prepped beforehand at Purdue University; fine Murchison dust particles were deposited onto Si_3N_4 Wildfire nanochips using wet-mounting methods (Fig. 1). We irradiated two separate samples with a 23 keV He^+ beam up to a total fluence of ~1.0×10^{17} ion/cm^2 using a flux of ~1.0×10^{13} ions/cm^2/s. An individual grain was monitored in bright field TEM mode for each irradiation. A combination of bright field TEM (BF TEM) images, high-resolution TEM (HRTEM) images, and selected area diffraction (SAED) patterns were acquired before, during, and after the experiment. Quality HRTEM and SAED patterns were sometimes difficult to obtain due to the thickness of individual Murchison particles. Results suggest that vesicles start to form at a fluence of 2.5×10^{16} – 5.0×10^{16} ions/cm^2. With progressive irradiation, the number and size of vesicles increases due to accumulation of implanted He and coalescing of individual vesicles (Fig. 2). After completing irradiation, the vesicles are ~40 nm or less in size and mostly spherical or oblong in shape. SAED patterns indicate that He^+ irradiation induces partial to complete amorphization of the grains.

We also performed *in situ* heating on one of the He^+-irradiated samples. This sample was heated to 1200˚C in 1 second to simulate the thermal shock induced by micrometeoroid impacts and held isothermally for five minutes before returning to room temperature. Heating causes vesicles to burst or coalesce into fewer, but larger vesicles (~40 – >100 nm). Several nanoparticles also form; their sizes range from 10 – <200 nm. Nanoparticle d-spacings suggest pentlandite, troilite, and even pyroxene and olivine mineralogies, however, smaller d-spacings may also be consistent with Fe metal, wüstite, magnetite, or pyrrhotite (Fig. 2). Matrix material is predominantly crystalline after heating, with some localized amorphous regions. Similar to the typical CM chondrite mineralogy, the matrix consists mainly of phyllosilicates whose sinuous and rounded morphology as well as d-spacings are consistent with chrysotile. Future analyses acquired with the TEM's recently-installed electron energy loss spectroscopy detector will allow us to further constrain the composition of nanoparticles and phyllosilicates.

Although it started as a mere idea for a side project, this research has evolved into the third chapter of my dissertation. Monetary support from the Goldstein Award has been instrumental to my project development and PhD progress. This trip to MIBL allowed me to refine my experimental plan for future visits (e.g., mounting particles on the backside of the nanochips; how to prepare FIB sections on the nanochips) and network with a new group of scientists, laying the groundwork for future collaborations

between our research groups. Thank you to the Microanalysis Society for their support.

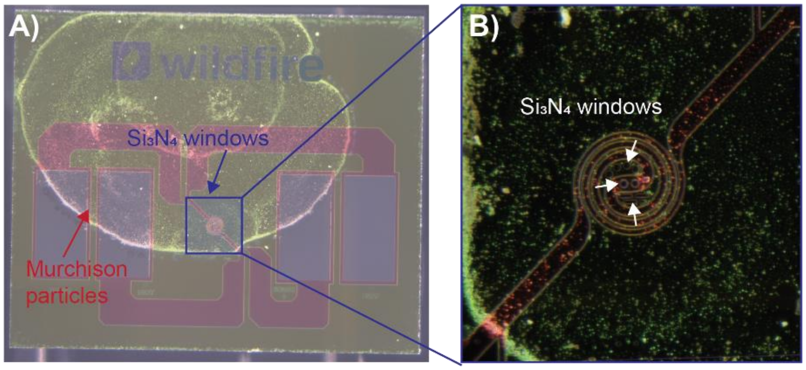

Figure 1. (A) Image of fine Murchison dust mounted on a DENSsolutions Si_3N_4 Wildfire nanochip. (B) A close-up of the Si_3N_4 viewing windows (indicated by white arrows). The green specks are Murchison grains.

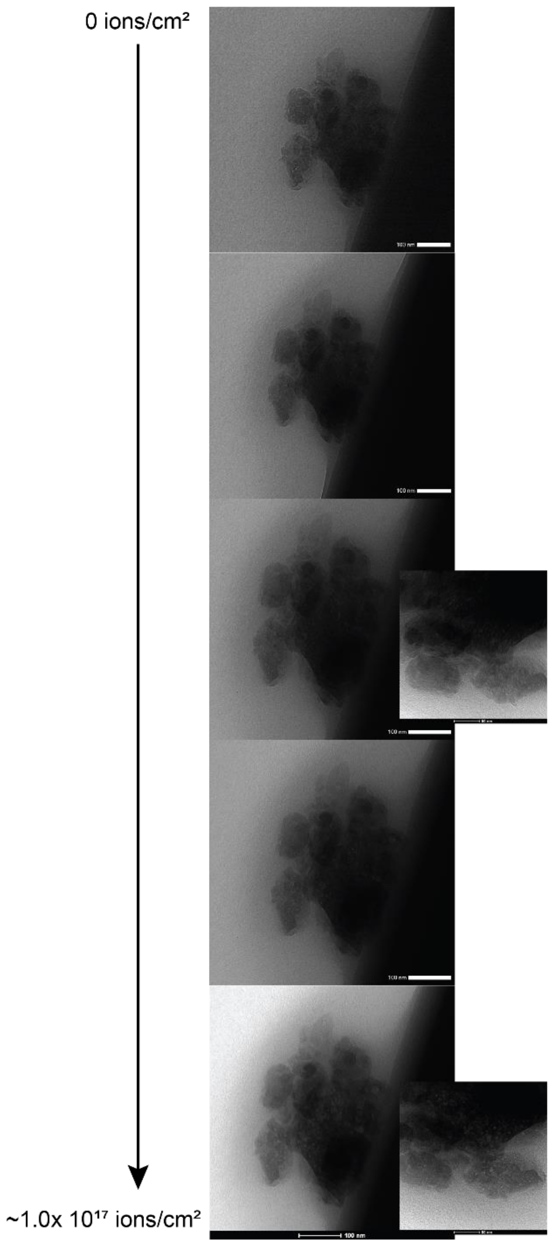

Figure 2. A series of BFTEM images of a Murchison grain irradiated with He^+ to increasing fluences. The bottom image was acquired after irradiating up to the total fluence of ~1.0×10^{17} He^+/cm^2. Note the formation of small vesicles which grow larger and more visible with progressive irradiation. The inset BFTEM images zoom in on portions of the larger grain.

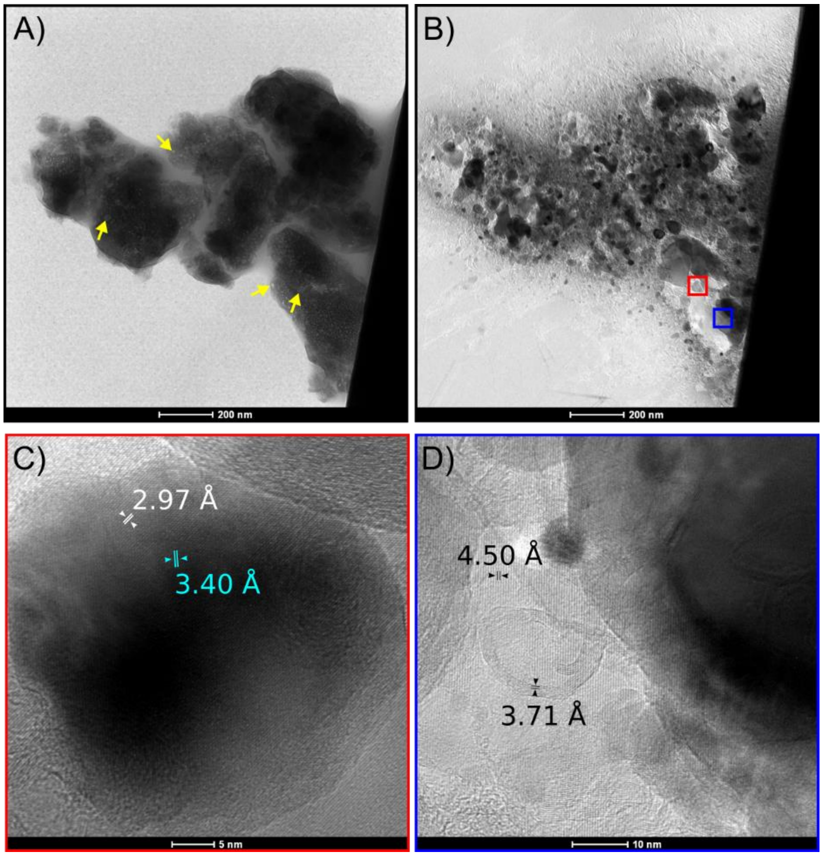

Figure 3. (A) BFTEM image of a Murchison grain after He^{+} irradiation. Yellow arrows indicate vesicles which appear as light-contrast circular features. (B) BFTEM image of the same grain after heating. The dark-contrast features are possible Fe-bearing nanoparticles. (C) HRTEM image of the region indicated by the red box in (B). Measured d-spacings are consistent with pyrrhotite or magnetite. (D) HRTEM image of the region indicated by the blue box in (B). The thin, sinuous or rounded crystalline features are phyllosilicates. The pyllosilicate d-spacing (3.71 Å) suggests chrysotile mineralogy. The other measured d-spacing (4.50 Å) is consistent with either pentlandite or pyroxene.