TEM ANALYSIS OF SPACE WEATHERING FEATURES IN AN ITOKAWA SOIL GRAIN WITH A POLYPHASIC MINERALOGY. M. S. Thompson[1], T. J. Zega[2], J. Y. Howe[3], [1]NASA Johnson Space Center, Mail Code XI3, 2101 E NASA Parkway, Houston, TX, 77058, michelle.s.thompson@nasa.gov, [2]Lunar and Planetary Laboratory, Department of Planetary Sciences, University of Arizona, 1629 E. University Blvd, Tucson, AZ, 85721, [3]Hitachi High-Technologies America Inc., 22610 Gateway Center Drive, Suite 100 Clarksburg, MD 20871.

Introduction: Grains on the surfaces of airless bodies are exposed to micrometeorite impacts and irradiation by energetic particles from the solar wind. Such space weathering alters the microstructure, chemical composition, and spectral characteristics of surface materials [1,2]. The changes in these properties make it difficult to characterize planetary surfaces with remote-sensing techniques, and present challenges for matching meteorites to their parent body asteroids.

Sample-based analyses of space weathering have traditionally focused on lunar soils and asteroidal and lunar regolith breccias [3-5]. The Hayabusa mission to near-Earth asteroid Itokawa enables comparison of space weathering characteristics in returned samples from a known asteroidal body. Previous analyses indicate that samples from Itokawa show weathering characteristics consistent with formation via solar wind irradiation and micrometeorite impacts e.g., [6-9]. These features include: nanophase Fe (npFe) particles, partially and completely amorphous rims on soil grains, melt splashes, vesiculated textures, and solar flare tracks. Here we report detailed chemical and microstructural analyses of a polyphasic grain returned by the Hayabusa mission. Analysis of this grain indicates space weathering processes have affected several mineral types in the same assemblage, providing the opportunity to compare the response of each phase to space weathering.

Samples and Methods: We were allocated Itokawa sample RA-QD02-0248 from the NASA JSC collection, which was provided as an intact grain measuring ~148 µm in size. We embedded the grain in low viscosity epoxy and prepared it with the hybrid ultramicrotome-focused ion beam (FIB) technique developed by [10]. We created ultramicrotome sections of part of the grain, leaving the bulk for preparation by FIB. Four FIB sections were extracted from locations around the exterior edge of the grain. Both the FIB and microtomed sections were analyzed using the transmission electron microscope (TEM).

We prepared three FIB sections using the FEI Helios Nanolab 660 FIB-SEM located at the University of Arizona. One FIB section was prepared using a Hitachi NB5000 Dual FIB at Hitachi High-Technologies Co. in Naka, Japan. We analyzed two of these FIB sections in the 300 keV Hitachi HF3300 at the University of Toronto, equipped with a bright-field (BF), dark-field (DF), and secondary electron (SE) detectors, and a Bruker silicon-drift energy dispersive x-ray spectrometer (EDS) system. All four FIB sections were also analyzed using the 200 keV aberration-corrected Hitachi HF5000, currently being installed at the University of Arizona. The HF5000 is a TEM/STEM equipped with BF, DF, SE detectors, and Oxford dual side-entry silicon-drift X-ray detectors giving a large (2.0 sr) solid angle. The microtome thin sections were analyzed using the JEOL 2500SE TEM located at Johnson Space Center, equipped with BF and DF detectors and a Thermo thin-window Li-drifted Si EDS detector.

Results: The grain is composed of regions of plagioclase, olivine, and pyroxene, shown in Fig. 1. The sites for sections 1-4 were selected to ensure examination of each mineral type and all exterior sides of the grain, providing a three dimensional analysis of weathering characteristics. Section 3 was found not to sample the exterior rim of the grain and as such those results have not been included here.

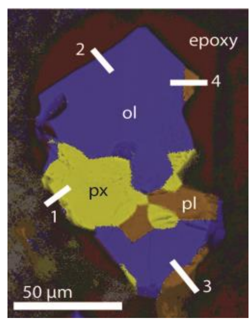

Figure 1: EDS phase map collected in the FIB of the grain surface after microtoming. Mineral phases present in the grain include olivine (blue), pyroxene (yellow), and plagioclase (orange). The location of extracted FIB sections are shown by white lines (1-4).

Section 1: This section is composed of pyroxene and olivine and both minerals exhibit features characteristic of space weathering (Fig. 2). The pyroxene contains a rim of non-uniform thickness (up to ~60 nm) that is continuous along the exterior edge of the grain exposed in the FIB section. High resolution TEM images (HRTEM) show that the rim contains pockets of nanocrystallinity interspersed with amorphous zones. EDS maps indicate there is a depletion in Ca, Fe, and Mg in the nanocrytalline rim, compared to the average of the grain interior. There is also a region in the rim that is enriched in Si, occurring ~5 nm below the exterior of the grain (Fig. 2), similar to that described in [8]. TEM images of the olivine portion of the grain indicate it has a rim of uniform thickness (~30 nm) that exhibits difference in contrast compared to the underlying material. HRTEM images however, show the rim maintains short-range order. EDS maps show a gradual depletion in all elements over the width of this rim approaching the edge of the grain.

Section 2: This section is composed of olivine. Similar to Section 1, TEM images show a continuous rim of uniform thickness, ~50 nm, on the exterior edge of the olivine, though the rim remains crystalline. EDS maps indicate a gradual decrease in intensity from all elements over the width of the rim.

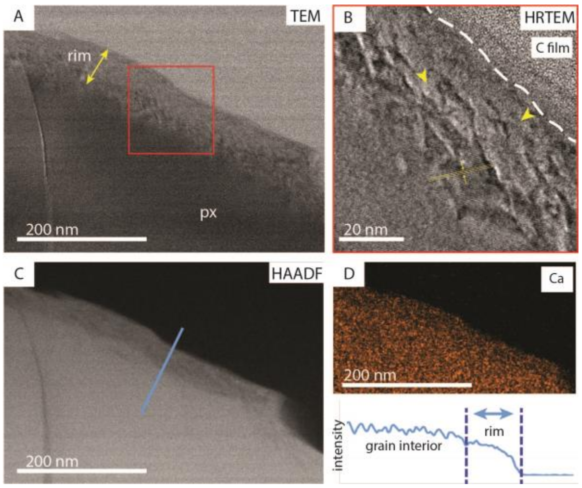

Figure 2: A) TEM image showing the partially amorphous rim, B) HRTEM image of area outlined by the red box in A). Yellow lines highlight lattice fringes in areas of crystallinity and yellow arrows indicate areas of amorphization, C) High angle annular dark field (HAADF) image showing the rim. Element concentration profile was extacted along the blue line, D) Ca EDS map (top) and the results of the intensity profile extracted in C), showing lower Ca concentration in the rim.

Section 4: This section includes both plagioclase and olivine grains. HRTEM images of the plagioclase show a rim of uniform thickenss (~25 nm) that is completely amorphous. TEM images of the olivine, similar to observations from other sections, indicate there is a structurally disordered rim which, upon examination with HRTEM, maintains short-range order although long-range order has begun to disappear.

Microtome Sections: Regions of the pyroxene grain exhibit a partially amorphous rim, ~50 nm in thickness, containing localized areas of nanocrystallinity.

Discussion: Our results indicate that this grain experienced space weathering effects on the surface of Itokawa, primarily through irradiation by solar wind ions. The FIB sections analyzed in this work were extracted from opposing sides of the grain, and each exhibits characteristics consistent with solar wind irradiation. These results suggest the grain has been tumbled or overturned on the surface of Itokawa, with all sides eventually being exposed to the solar wind.

The degree of amorphization and thicknesses of the rims vary with mineral phase. Section 1 has pyroxene and olivine grains in direct contact with one another, indicating each mineral has likely recieved the same irradiation dose. While pyroxene exhibits localized zones of partial amorphization, olivine maintains its crystallinity, which has been observed previously [9]. Model predictions of amorphization timescales made using programs like Stopping and Range of Ions in Matter (SRIM) suggest complete amorphization of olivine (up to 20 nm depth) in as little as 100 years [11]. It is possible this grain experienced a short exposure timescale, however, it should be noted that SRIM accounts for the chemical compositions of the irradiated target, but not crystal structure. While experimental irradiation of olivine and pyroxene show amorphization occurs at similar doses, these fluences were much higher than appropriate for the solar wind [12]. Our results suggest that crystal structure may play an important role in determining amorphization thresholds at low dose and/or short exposure timescales.

In addition to microstructural measurements, chemical data also indicate that the response to irradiation varies between olivine and pyroxene grains. In olivine, the disordered rim exhibits a gradual decrease in concentration of all elements over the rim, indicating the crystal may first become less dense, with no preferential sputtering, before amorphization occurs. In contrast, pyroxene shows a depletion in Fe, Mg, and Ca, but a localized enrichment in Si in the rim. While olivine appears to experience depletion in all elements uniformly with exposure to irradiation, the pyroxene structure may trap certain elements, perhaps in localized regions that maintain crystallinity. Further work should be completed to understand the differences in mineral response to irradiation, particularly at low doses.

References: [1] Hapke B. (2001) *J. Geophys. Res-Planet., 106*, 10,039-10,073. [2] Pieters C. M. and Noble S. K. (2016) *J. Geophys. Res-Planet., 121*, 1865-1884. [3] Keller L. P. and McKay D. S. (1997) *Geochim. Cosmochim. Ac., 61*, 2331-2341. [4] Noble S. K. et al. (2005) *Meteorit. Planet. Sci., 40*, 397-408. [5] Noble S. K. et al. (2011) *Meteorit. Planet. Sci., 45*, 2007-2015. [6] Noguchi T. et al. (2011). *Science 333*, 1121-1125. [7] Noguchi T. et al. (2014). *Meteorit. Planet. Sci. 49*, 188-214. [8] Thompson M. S. et al. (2014) *Earth. Planet. Space., 66*, 1-10. [9] Keller L. P. and Berger E. L. (2014) *Earth. Planet. Space., 66*, 1-7 2044. [10] Berger. E. L. and Keller L P. (2015) *Microscopy Today, 23*, 18-23. [11] Christoffersen R. and Keller L. P. (2015) *LPS XXXXIV* Abstract #2084. [12] Christoffersen R. and Keller L. P. (2011) *Meteorit. Planet. Sci., 4*, 950-969.