

**PROBLEMS AT THE LEADING EDGE OF SPACE WEATHERING AS REVEALED BY TEM COMBINED WITH SURFACE SCIENCE TECHNIQUES.** R. Christoffersen<sup>1</sup>, C. A. Dukes<sup>2</sup>, L.P. Keller<sup>3</sup>, Z. Rahman<sup>1</sup>, and R. A. Baragiola<sup>2,†</sup>, <sup>1</sup>Jacobs, NASA Johnson Space Center, Mail Code XI, Houston, TX 77058, USA, ([roy.christoffersen-1@nasa.gov](mailto:roy.christoffersen-1@nasa.gov)), <sup>3</sup>NASA JSC, Houston, TX, 77058, USA, <sup>3</sup>University of Virginia, Laboratory of Atomic and Surface Physics, Charlottesville, VA 22904, USA, <sup>†</sup>deceased.

**Introduction:** Both transmission electron microscopy (TEM) and surface analysis techniques such as X-ray photoelectron spectroscopy (XPS) were instrumental in making the first characterizations of material generated by space weathering in lunar samples [1,2]. Without them, the nature of nanophase metallic Fe (npFe<sup>0</sup>) correlated with the surface of lunar regolith grains would have taken much longer to become recognized and understood. Our groups at JSC and UVA have been using both techniques in a cross-correlated way to investigate how the solar wind contributes to space weathering [e.g., 3]. These efforts have identified a number of ongoing problems and knowledge gaps. Key insights made by UVA group leader Raul Baragiola during this work are gratefully remembered.

**Threshold Fluences for Solid-State Amorphization by the Solar Wind.** Early application of TEM to the Apollo samples showed that solar wind ions produced partial to completely amorphous “rims” up to ~150 nm thick on regolith mineral grains [1]. Later TEM work revealed additional layers of vapor or sputter deposited material adding to the complexity of rim formation processes [4]. The width of the ion-processed portions of the rims can be developed as one type of “exposure clock” for measuring how long a given grain has been completely uncovered on the regolith surface [5]. This development, however, is hindered by incomplete experimental calibration of the critical ion fluences for amorphization of key minerals under solar wind irradiation conditions. We will review why past experimental design has played a role in keeping this knowledge gap open.

**Depth Evolution of Surface Composition Changes Produced by The Solar Wind:** During in-situ ion irradiation under solar wind conditions in the XPS, surface reduction occurs in Fe-bearing silicates and oxides (e.g., ilmenite) [6,7]. This is typically interpreted to occur by the preferential sputtering of oxygen [6]. High-resolution field-emission TEM (FE-STEM) indeed confirms that a layer of surface-correlated npFe<sup>0</sup> grains a few nm thick is produced as part of the process [3]. What FE-STEM also shows, however, is that as the outer ~100 nm of some irradiated minerals amorphizes and/or become more ion processed, compositional gradients deepen and become complex to a degree not so easily explained by preferential sputtering alone [7]. In ilmenite in particular, the gradients show a significant resemblance to those in irradiated grain rims on natural

lunar ilmenite [8]. Currently, these observations frame a knowledge gap regarding how the solar wind actually creates the integrated thickness of the npFe<sup>0</sup>-bearing, optically-active layers on space weathered grains over time.

**Solar Wind Sputter Erosion.** Solar wind sputter erosion can potentially remove layers of space weathered material from grain and rock surfaces. We used XPS in-situ surface analysis, combined with ex-situ FE-STEM, to track changes in microstructure and bulk surface composition of mature lunar mare soil 10084 under step-wise in-situ ion irradiation by 4 kV He<sup>+</sup> and Ar<sup>+</sup> [3]. XPS showed surface major element ratios changing from a unique surface mono-layer composition towards the bulk composition of space weathered grain rims in the soil [3]. FE-STEM found no evidence of sputtering removing or altering existing grain rims in the sample even up to He<sup>+</sup> fluences equivalent lunar surface exposure times of 10<sup>4</sup>-10<sup>5</sup> years. The results point to a surface “cleaning” effect of the solar wind, but not to significant sputter erosion of rims over the exposure lifetime of lunar grains.

**Solar Wind Sputter Deposition.** Solar wind sputter deposition has been proposed to deposit dark optically-active surfaces layers on regolith grains [9]. We repeated the famous “Hapke experiment” [9] using a mm-scale lunar orthopyroxene substrate cantilevered over a homogeneous lunar glass sputtering target [10]. Sputtering by 5 keV Ga<sup>+</sup> ions produced a 70 nm-thick amorphous sputter deposit on the orthopyroxene. FE-STEM analyses show that the deposit is completely amorphous, contains no npFe<sup>0</sup>, and is compositionally homogeneous with no significant element enrichments/depletions relative to the sputtered source. It does not resemble the npFe<sup>0</sup> surface deposits on lunar grain rims, suggesting sputter deposition may not play a significant role in the latter’s formation.

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