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## Simulation of solar wind space weathering in orthopyroxene

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## ABSTRACT

We have simulated solar wind-based space weathering on airless bodies in our Solar System by implanting hydrogen and helium into orthopyroxene at solar wind energies ( $\sim 1$  keV/amu). Here we present the results of the first scanning transmission electron microscope (STEM) study of one of these simulants. It has been demonstrated that the visible/near infrared (VNIR) reflectance spectra of airless bodies are dependent on the size and abundance of nanophase iron (npFe<sup>0</sup>) particles in the outer rims of regolith grains. However, the mechanism of formation of npFe<sup>0</sup> in the patina on lunar regolith grains and in lunar agglutinates remains debated. As the lattice is disrupted by hydrogen and helium implantation, broken bonds are created. These dangling bonds are free to react with hydrogen, creating OH and/or H<sub>2</sub>O molecules within the grain. These molecules may diffuse out through the damaged lattice and migrate toward the cold traps identified at the lunar poles. This mechanism would leave the iron in a reduced state and able to form npFe<sup>0</sup>. This work illustrates that npFe<sup>0</sup> can be nucleated in orthopyroxene under implantation of solar wind hydrogen and helium. Our data suggest that the solar wind provides a mechanism by which iron is reduced in the grain and npFe<sup>0</sup> is nucleated in the outer surfaces of regolith grains. This formation mechanism should also operate on other airless bodies in the Solar System.

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## 1. Introduction

“Space weathering” is a phenomenon of great interest to planetary scientists. In an extensive review of the literature on “space weathering”, Hapke defines the term as an “aggregate of the physical and chemical changes that occur to material exposed on the surface of an airless body” (Hapke, 2001). The processes that can affect these changes include the solar wind, ultraviolet radiation, meteorite and micrometeorite impacts, and solar and galactic cosmic rays. These phenomena have been extensively studied over the past 30 years using a variety of experimental, microanalytical, and theoretical techniques (Bradley et al., 2014; Domingue et al., 2014; Hapke, 2001; Hornung et al., 2000; Horz and Cintala, 1997; Keller and McKay, 1993; Pieters et al., 2000; Taylor et al., 2010). It has been demonstrated that small particles (< 50 nm) of npFe<sup>0</sup> darken and redden spectral properties while large particles (> 50 nm) of npFe<sup>0</sup> only darken spectra (Lucy and Riner, 2011; Noble et al., 2007). Recently it has also been discovered using scanning transmission electron microscopy (STEM) that hydrogen implanted into silicates generates H<sub>2</sub>O and –OH

(Bradley et al., 2014). The mechanism(s) of npFe<sup>0</sup> formation within low-iron materials and their kinetics are not well understood and remain contentious (Marchi et al., 2010; Pieters et al., 2012). In fact, some investigators have found that hydrogen implantation can cause a depletion in the water content of irradiated samples rather than an increase (Burke et al., 2011).

## 2. Methods

We obtained a sample of orthopyroxene, enstatite (MgSiO<sub>3</sub>), from the Egersund Anorthosite Massif in Norway and cut and highly polished several flat “coupons” of the material for elemental analysis using electron probe microanalysis (EPMA) (Cameca SX51, France) at the University of Wisconsin – Madison (UW–Madison). The orthopyroxene was polycrystalline, containing lamellae of plagioclase, spinel, and minor fractions of magnetite and ilmenite as determined using EPMA (Table 1).

## 2.1. Sample implantation

Once  $\sim 1$  cm diameter wafers of orthopyroxene were highly polished with 0.05  $\mu$ m alumina, plasma source ion implantation (PSII) at the UW–Madison was used to implant hydrogen and

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helium at solar wind energies ( $\sim 1$  keV/amu) into samples of polished orthopyroxene. The present work is similar to that of Brunetto and Strazzulla (2005), which demonstrated a reddening and darkening of the reflectance spectra in the 0.25–2.7  $\mu\text{m}$  spectral range (Brunetto and Strazzulla, 2005). However, the current work utilizes lower ion energies more realistic of those in the solar wind and single mineral samples where the previous work utilized bulk silicate samples. Two of the orthopyroxene coupons were independently subjected to irradiation of hydrogen and helium to approximate solar-wind bombardment using the UW–Madison PSII facility. This is a non-line-of-sight technique (Fig. 1) for energetic ion bombardment of materials (Anders, 2000; Conrad, 1988; Conrad et al., 1990; Mantese et al., 1996; Rej, 1996; Sridharan and Reeber, 1994). In PSII, the samples to be treated are immersed in plasma generated in a vacuum chamber and pulse-biased to a negative potential using a tetrode modulator. This negative voltage bias can range from a few hundred volts to as high as 25 kV, an energy range very suitable for the simulation of solar wind ions. The 1 m<sup>3</sup> chamber was evacuated to a base pressure of about 10<sup>-6</sup> Torr and hydrogen or helium gas is allowed to flow through the chamber at a pressure of approximately 5 mTorr. The gases used were either laboratory-grade hydrogen (99.9%) or laboratory-grade helium (99.9%). The plasma is generated using tungsten filaments to ionize the gas by energetic primary electron impact, which is more suitable for low energy bombardment (< 5 kV). We note that because of this method of ion generation, implanted ions tend to average a broader implant depth and peak at a lower energy than the same nominal species implanted by a conventional accelerator (Vajo et al., 1994). The energy distribution of the implanted ions is likely broadened and reduced in energy by displacement current effects across the expanding plasma sheath. In addition, the non-line-of-sight PSII process provides a “bell-jar” type environment rather than an accelerator type environment, which allows for uniform ion implantation of three-dimensional samples. In this case, we placed the 1 cm<sup>2</sup> mineral samples on a silicon wafer, which acts as the object to be implanted. The mineral samples are, simply speaking, impurities in the silicon wafer and get implanted as well. In the case of a wafer, significant asymmetries can occur at the edges where the electric field is changing rapidly. Focusing of the ions at

the center of the wafer can also occur (Fetherston, 1997). Therefore, the samples of orthopyroxene were placed off-center and away from the edges to maintain a uniform implantation.

The time needed to implant a desired fluence is given by Eq. (1):

$$\text{time} = \frac{\text{Fluence} \cdot \text{Area} \cdot e \cdot (\gamma + 1)}{I_p \cdot \text{RR} \cdot N} \quad (1)$$

where time is the total time of the implantation (s), fluence is the desired final dose (ions/cm<sup>2</sup>),  $e$  is the charge on an electron (Coulombs per electron),  $\gamma$  is the secondary electron emission coefficient (SEEC) (electrons emitted per incident ion),  $I_p$  is the average current per “on-time” of the pulse (A), RR is the repetition rate (Hz) and  $N$  is the number of atoms per molecule of the precursor gas (i.e.  $N=1$  for He,  $N=2$  for H<sub>2</sub>). SEEC's used were taken from a study of electron emission from clean metal surfaces (Baragiola et al., 1979). These values do not change drastically for other elements ( $\pm$  at most 0.2) at these energies and the choice of the value to use will not greatly affect the calculation of time needed for the dose (a 30% error in  $\gamma$  only results in an error of 12% in Eq. (1)). The values of SEEC's on Cr were generalized to 0.25 for H<sub>2</sub><sup>+</sup> and 0.5 for <sup>4</sup>He in the current implantations. It should be remembered here that we are implanting a pure silicon wafer with the mineral samples acting as an impurity.

The impingement of energetic ions on a material's surface can initiate a variety of mineral disruption events as illustrated in Fig. 2. Ion implantation is dominant at higher energies and leads to the entrapment of implanted species atoms in the near-surface regions of the target material. At high doses, coalescence of the atoms of the implanted species can lead to void/bubble nucleation [e.g. (David et al., 2014; Klimenkov et al., 2013)]. Sputtering, on the other hand, leads to the “erosion” or recession of target surface and is more pronounced at lower energies and higher doses.

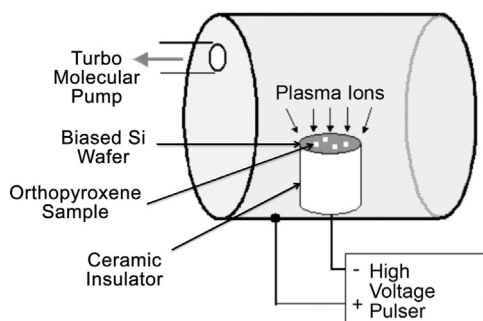
After each polished sample was implanted, they were removed from the PSII chamber and transferred to a sputter coater in a separate facility. The implanted surface was sputter-coated with a 60 nm thick protective, conductive layer of gold. STEM sample preparation was accomplished using a focused ion beam (FIB) (CrossBeam, Zeiss Corp. Germany), which allowed us to prepare the STEM samples from selected areas of the matrix, which were free from the inclusions and lamellae using the standard methods (Tomus and Ng, 2013). The FIB's SEM was used to target the orthopyroxene matrix material, and a protective layer of carbon approximately 3  $\mu\text{m}$  thick, and 3  $\mu\text{m}$  wide by 20  $\mu\text{m}$  long was deposited in an appropriate location using ion beam assisted deposition of carbon. Once the carbon layer was deposited, standard TEM lift-out sample preparation methods were used to fabricate an electron transparent lamella approximately 8  $\mu\text{m}$  deep and 12  $\mu\text{m}$  long (Fig. 3) (Tomus and Ng, 2013). Final polishing was performed at low angle with a 5 keV gallium beam. Further cleaning was performed after gentle asher cleaning of the sample, using a NanoMill<sup>®</sup> (Fischione). This cleaning was performed using 900 eV Ar for 15 min per side.

## 2.2. Analytical methods

Once STEM samples were fabricated with the FIB, they were analyzed in the TITAN STEM (FEI Corp.) at the UW–Madison. We utilized electron energy loss spectroscopy spectrum imaging (EELS SI) using a 24.5 pA probe with size < 0.12 nm at 200 keV. We took all precautions to avoid sample damage during these experiments including short EELS integration time (15–200 ms) and limiting probe current. For this reason we did not use energy-dispersive X-ray spectroscopy spectrum imaging (EDX SI) because it demands several orders higher electron dose than EELS SI. Electron energy

**Table 1**  
Average EPMA results from 10 spots in the orthopyroxene matrix given in oxide weight percent. The sum of the measurements is 100.587.

Na <sub>2</sub> O	MgO	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	K <sub>2</sub> O	CaO	TiO <sub>2</sub>	Cr <sub>2</sub> O <sub>3</sub>	FeO	MnO
0.002	27.283	5.578	52.06	0.006	0.354	0.36	0.107	14.589	0.248



**Fig. 1.** Schematic illustration of the PSII process at the UW–Madison that was used for energetic ion bombardment of highly polished orthopyroxene samples. The samples are placed approximately halfway out on an 8-in. silicon wafer, which acts as the object being implanted. The ions follow the arrows orthogonal to the surface of the silicon wafer.

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