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Phase-dependent space weathering effects and spectroscopic identification of retained helium in a lunar soil grain

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Abstract

The solar wind is an important driver of space weathering on airless bodies. Over time, solar wind exposure alters the physical, chemical, and optical properties of exposed materials and can also impart a significant amount of helium into the surfaces of these bodies. However, common materials on the surface of the Moon, such as glass, crystalline silicates, and oxides, have highly variable responses to solar wind irradiation. We used scanning transmission electron microscopy (STEM) with electron energy loss spectroscopy (EELS) to examine the morphology and chemistry of a single grain of lunar soil that includes silicate glass, chromite and ilmenite, all present and exposed along the same surface. The exposure of the silicate glass and oxides to the same space weathering conditions allows for direct comparisons of the responses of natural materials to the complex lunar surface environment. The silicate glass shows minimal effects of solar wind irradiation, whereas both the chromite and ilmenite exhibit defect-rich rims that currently contain trapped helium. Only the weathered rim in ilmenite is rich in nanophase metallic iron ($npFe^0$) and larger vesicles that retain helium at a range of internal pressures. The multiple exposed surfaces of the single grain of ilmenite demonstrate strong crystallographic controls of planar defects and non-spherical $npFe^0$. The direct spectroscopic identification of helium in the vesicles and planar defects in the oxides provides additional evidence of the central role of solar wind irradiation in the formation of some common space weathering features.
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Keywords: Space weathering; Solar wind; Scanning transmission electron microscopy; Ion irradiation; Helium

1. INTRODUCTION

Solar wind irradiation contributes a significant amount of hydrogen and helium to the surfaces of airless bodies and can change optical, chemical, and physical properties of individual rocks and soil grains. The space weathered rims of these grains, as the affected regions are known, are altered significantly by this irradiation as well as other interactions with the space environment, such as micrometeoroid bombardment (Pieters and Noble, 2016). Different materials respond in different ways to irradiation and bombardment, and the altered rims at grain surfaces can include a variety of features, including amorphous layers, layers of variable composition, nanophase metallic iron inclusions ($npFe^0$), and vesicles (e.g., Keller and McKay, 1997). Grains from within the same soil sample can demonstrate significant variability in rim thicknesses and composition (Keller and McKay, 1997), indicating that bulk measurements of the degree of space weathering experienced by a soil do not necessarily reflect the complex processing experienced by the lunar regolith. Recent work on samples returned from asteroid Itokawa show large variations in surface space weathering features on different grains and even different parts of the same grain (Matsumoto et al., 2015, 2016; Noguchi et al., 2014). The grain-to-grain variations can make it challenging to distinguish between the effects of long exposure times, differences in material

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properties, and changes in the dominant space weathering process affecting any single grain. Comparisons between individual grains may reflect either different weathering processes or different material responses to the same process.

The presence of helium and other noble gases in space weathered material provides evidence of the influence of the solar wind in altering surface material, but the specific features created by irradiation may not always be distinguishable relative to other processes. For instance, the amorphous rim on a pyroxene grain may differ in composition from the substrate because it is a vapor deposited layer or because the composition was altered due to differential sputtering during irradiation. Some phases allow for more clear discrimination between the various processes, and the Si-free composition of ilmenite, the most common oxide on the lunar surface, means that material that was part of the original substrate can be clearly distinguished from material that was deposited by melt splashes or vapor deposition and that does contain Si (Bernatowicz et al., 1994; Christoffersen et al., 1996). Studying the composition and morphology of ilmenite rims, where solar wind effects can potentially be separated from other processes (Bernatowicz et al., 1994; Christoffersen et al., 1996), and comparing ilmenite to other phases known to have experienced the same exposure conditions can lead to a better understanding of the role of irradiation in creating commonly seen space weathering features.

Helium is ubiquitous in the Solar System, and measurements of its abundance and isotope ratios are important for understanding formation and evolution of many materials. Among common phases present on the Moon, ilmenite is the most retentive of helium (Signer et al., 1977) and highly resistant to amorphization by ion irradiation (Borg et al., 1976). However, the mechanism of trapping and storage of helium in ilmenite and other phases and the conditions of formation of vesicles related to build-up of solar wind implanted material has not been well studied. In addition to lunar samples, implanted solar wind ions have been measured in meteoritic regolith breccias (Suess et al., 1964), micrometeorites and interplanetary dust particles (IDPs) (e.g., Nier and Schlutter, 1992; Kehm et al., 2002; Rajan et al., 1977), and "fossil" micrometeorites in (ancient) deep sea sediments (e.g., Merrihue, 1964; Heck et al., 2004), and used to distinguish presolar or interstellar grains (e.g., Heck et al., 2007; Pepin et al., 2011), but studies of helium abundance have not established how the implanted He is retained, i.e., whether interstitially, in vesicles, or both. Helium is also a key component of the exospheres of the Moon and Mercury (Wieler, 2002), and establishing how or when trapped helium is released from soils could improve our understanding of lifetimes and cycling of exospheric gases.

Characterization of the nano-scale effects of space weathering requires the use of (scanning) transmission electron microscopy (S/TEM) and associated analytical techniques (e.g., Christoffersen et al., 1996; Keller and McKay, 1993, 1997; Noble et al., 2005; Noguchi et al., 2011; Thompson et al., 2016). A number of studies have shown that electron energy loss spectroscopy (EELS) in a S/TEM is a valuable technique for identifying and measuring the helium in nano-scale cavities in a number of materials, including laboratory irradiated metals and semiconductors (e.g., David et al., 2014; Fréchard et al., 2009; Walsh et al., 2000) and radiogenic minerals (Seydoux-Guillaume et al., 2016). Spectroscopic signatures of hydrogen in vesicles in the space weathered rim of a pyroxene grain from an IDP were identified using this technique (Bradley et al., 2014). In the present study, we were able to examine a lunar soil grain in which silicate glass, chromite, and ilmenite are exposed along the same surface. The sections were prepared for analysis in the STEM using a focused ion beam microscope (FIB), which leads to sections in which multiple space-exposed edges of a single grain can be viewed. Results shown here show a range of space weathering features in the three exposed phases, including crystallographic orientation effects in the ilmenite. The EELS data show conclusively the presence of helium in vesicles in the space weathered rim of the ilmenite and in small defects in both the ilmenite and chromite.

2. MATERIALS AND METHODS

The soil grain examined here is from sub-mature soil 71501,288. The soil has been previously characterized by the Lunar Soil Characterization Consortium (LSCC) (Taylor et al., 2001) and in a number of bulk, mineral separates, and single grain noble gas analyses (Becker and Pepin, 1989; Nier and Schlutter, 1994; Benkert et al., 1993; Heber et al., 2003; Nichols et al., 1994; Pepin et al., 1999). Exposure ages and crater counts for the collection region group around 100 Ma and are linked to secondary impacts associated with Tycho crater (Arvidson et al., 1976). Plagioclase grains from this soil have an average of 6.7×10^8 tracks/cm^2 for 68 individual grains (Wieler et al., 1980), which gives an exposure age of ~15 kyr (Berger and Keller, 2015).

A small amount of the soil was dispersed on a sticky carbon substrate, coated with a thin layer of carbon, and examined in a scanning electron microscope (SEM). The grain described here is an ilmenite dendrite crystal, with silicate glass and quench crystals filling regions between ilmenite limbs (Fig. 1). Thin chromite lamellae are inter-grown with the ilmenite in some areas, and all three phases are exposed at the original surface of the grain. Two sections from the grain were prepared by focused ion beam microscopy (FIB) with a FEI Nova 600 FIB-SEM. Regions of interest on the grain were coated with a thick ion beam deposited carbon film (1–2 μm) over a thin layer of evaporated carbon before ion milling to prevent damage to the grain surface by the ion beam. A Ga$^+$ ion beam voltage of 30 kV and variable probe current was used for sectioning. Both the top and bottom space-exposed surfaces of the original grain are visible in section 1 (Fig. 1b), with a layer of the carbon tape substrate attached to the bottom surface, while only the bottom surface of section 2 was maintained intact after thinning (Fig. 1c). The sections were attached to a Cu half-grid using a Pt weld and thinned to electron transparency; final thinning was done at 30 kV, 40 pA. The final thickness was calculated to be ~130 and