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Article

Surface microstructures of lunar soil returned by Chang'e-5 mission reveal an intermediate stage in space weathering process

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Abstract

The lunar soils evolution over time is mainly caused by space weathering that includes the impacts of varying-sized meteoroids and charged particles implantation of solar/cosmic winds as well. It has long been established that space weathering leads to the formation of outmost amorphous layers (50–200 nm in thickness) embedded nanophase iron ($npFe^0$) around the mineral fragments, albeit the origin of the npFe0 remains controversial [1,2]. The Chang'e-5 (CE5) mission returned samples feature the youngest mare basalt [3,4] and the highest latitude sampling site [5], providing an opportunity to seek the critical clues for understanding the evolution of soils under space weathering. Here, we report the surface microstructures of the major minerals

including olivine, pyroxene, anorthite, and glassy beads in the lunar soil of CE5. Unlike the previous observations, only olivine in all crystals is surrounded by a thinner outmost amorphous SiO_2 layer (~10 nm thick) and embedded wüstite nanoparticles FeO (np-FeO, 3–12 nm in size) instead of npFe^0. No foreign volatile elements deposition layer and solar flare tracks can be found on the surface or inside the olivine and other minerals. This unique rim structure has not been reported for any other lunar, terrestrial, Martian, or meteorite samples so far. The observation of wüstite FeO and the microstructures support the existence of an intermediate stage in space weathering for lunar minerals by thermal decomposition.

Keywords: Lunar soil, wüstite FeO, nanoparticle, nanophase Fe, amorphous rim

1. Introduction

The lunar soils, made up of a myriad of minute crystalline, non-crystalline mineral fragments and agglutinates, have been exposed to the harsh environment, i.e., space weathering, for billions of years. Space weathering includesthe impact of meteoroids and the irradiations of the solar winds and cosmic rays [1, 6–8]. As a result, rims composed of amorphous Si_xO_y (50-200 nm in thickness) and embedded npFe^0 (2-10 nm in size) usually form on olivine, pyroxene, and anorthite grain surfaces [2, 9, 10]. The randomly-distributed npFe^0 significantly reduces the optical reflection intensity and reddens the reflectance spectra [11–15]. Beyond the rims into the grain cores, the atoms gradually undergo a transition from disorder to order and finally into the crystalline lattice of the hosts [16, 17].

The occurrence of amorphous SiO_2 and npFe^0 inside of the rim is explained by the impact of micrometeoroids and bombardment of solar/galactic cosmic rays and the solar wind that decomposes mineral-like olivine ($Fe_2SiO_4=2FeO+SiO_2$) and further reduce FeO into npFe^0 with the aid of solar-wind-implanted H$^+$ ions [18–20]. SiO_2 is then existent in the form of an amorphous state. Most npFe^0 are pure Kamacite (α-Fe, body-centered cubic) mixed with trace amounts of Ni and Co. Terrestrial laboratory simulations directly create a silica-rich amorphous layer (200 nm in thickness) and npFe^0 (10-30 nm in size) in a vacuum chamber by irradiating olivine grains with a pulse laser beam [21]. It demonstrates that the H$^+$ ions are not necessary in the reduction process while the intermediate phase FeO is not observed. The FeO is also not found in the dust of the Itokawa asteroid either, though these grains are speculated to be in their incipient state of space weathering [22].

The CE5 soils feature high-latitude sampling site (43.058°N and 51.916°W) of

the Moon and the youngest exposure ages [3,4,23-26]. By using aberration-corrected transmission electron microscopy (TEM) and scanning transmissionelectron microscopy (STEM), we examined the microstructures and chemical compositions at nano/atomic scales of 25 soil grains (1-3 μm in size) from Sample CE5C0400YJFM00507 (1.5 g), which mainly includes minerals olivine, pyroxene, anorthite, and glassy bead. We unambiguously identify the wüstite FeO nanoparticles instead of npFe^0 that are embedded in amorphous Si$_x$O$_y$ rims outside the olivine grains. Our systematic study provides clues or constraints on the incipient formation mechanism of rim structure under space weathering.

2. Materials and methods

2.1 Sample preparation

The CE-5 lunar soils (CE5C0100YJFM00103, ~1.5 g) are allocated by the China National Space Administration. The lunar soils are scooped by the CE5 lander and separated in an ultraclean room at the extraterrestrial sample curation center of the National Astronomical Observatories, Chinese Academy of Sciences. All the soils are kept in a nitrogen-filled glove box. To avoid chemical contamination and ion-bombing-induced amorphization, we do not employ the focused ion beam (FIB) to cut the bulk samples except the large glassy bead. The pristine fine grains were mounted on the lacey carbon-coated copper grids by directly dipping the grids straight into the lunar soil. Small grains can cling onto the copper grids by electrostatic adsorption, allowing us to study the grains with an average size of ~1 μm and obtain high-resolution lattice fringe and accurate chemical composition. Meanwhile, we analyze the grains having sharp edges because they may undergo short exposure time under micro-meteoroids impact or sloar wind. Mineral types are initially identified by the real-time chemical composition via energy-dispersive X-ray spectroscopy (EDS).

2.2 Glassy bead and standard sample preparation

The slices samples are cut from the large glass bead (~100 μm) and Fe standard sample by the FIB technique. The lamella was thinned down to 100 nm thick at an accelerating voltage of 30 kV with a decreasing current from the maximum 2.5 nA, followed by fine polish at an accelerating voltage of 2 kV with a small current of 40 pA. The standard samples of FeCO$_3$ and Fe$_2$O$_3$ were ground to fine powder in a glove box and then dispersed on lacey carbon-coated copper grids.

2.3 STEM, EDS and EELS measurements

Transmission electron microscopy (TEM) and scanning transmission electron microscopy (STEM) observations were performed on Spectra 300 (Thermo Fisher

Scientific) double-Cs-corrected transmission electron microscope equipped with a field-emission electron source and a monochromator, operated at an accelerating voltage of 300 kV. High-angle annular dark-field (HAADF)-STEM and bright-field (BF)-STEM images were collected with a probe convergence angle of 30 mrad. EDS data were collected with a super-X EDS detector. Electron-energy loss spectroscopy (EELS) data were acquired using a Gatan image filter (GIF) (Gatan Inc.) with a dispersion of 0.05–0.15 eV per channel and collection semi-angle of 40 mrad. The energy resolution of EELS was estimated to be 0.4-0.7 eV from the full width at half maximum of zero-loss peak.

3. Results and discussion

Most of the measured grains have sharp edges and typical crystallographic habits rather than the rounded surface of grains from the Apollo and Luna missions [27]. We start with olivine as it is the most sensitive to space weathering among the Moon's minerals [28–31]. Fig. 1a displays a HAADF-STEM image for a typical olivine grain. The enlarged upper region 1 and lateral region 2 are shown in Fig. 1b and c, respectively. The grain is entirely covered by amorphous layers with thicknesses of ~10 nm and uniformly embedded nanoparticles (see Fig. 2a for details). EDS analyses reveal that the amorphous layer is only composed of O and Si as shown in Fig. S1a (online). The atomic ratio of O and Si is ~2.0, indicating the formation of amorphous SiO_2 (a-SiO_2) layer. The average composition of the olivine is quantified to be $(Mg_{0.25}Fe_{0.75})_2SiO_4$, within the range of olivine in CE5 lunar soil [5]. The nanoparticles are richer in Fe in comparison to the host matrix, see Fig. S1b (online).

The EDS line mapping was performed to describe the element distribution from surface to interior of olivine. The profile along the red arrow of Fig. 1d is shown in Fig. 1e. It confirms that the outmost amorphous layer is solely made up of Si and O again, and the nanoparticles within the matrix consisting of the olivine and MgO are FeO. Therefore, the np-FeO are embedded inside the olivine just beneath the amorphous layer, which is supported by the thickness calculations in Fig. S2 (online). The size of nanoparticles spans from 3 to 12 nm with a dominant range of 6-8 nm, see the histogram of Fig. 1f. The fast-Fourier transform (FFT) pattern (Fig. 1g) based on a high-resolution transmission electron microscopy (HRTEM) image (Fig. S3, online) suggests that apart from the olivine phase, the wüstite fcc-FeO nanoparticles be embedded inside as the diffraction rings, matching well with the simulated ones.

We then carried out the HRTEM imaging to probe into the details of the amorphous layer, nanoparticle and the interior olivine. In Fig. 2a, a multiple-layered rim

consisting of zone I, II, and III is observed, in which three typical areas labeled as green, red and white squares are selected for further measurements. The results are shown in Fig. 2b, c, respectively. In the green square area, the outmost layer is homogeneously amorphous with a thickness of ~10 nm, see an enlarged HRTEM image and the corresponding FFT pattern in Fig. 2b. The HRTEM image for an np-FeO (red square) particle is shown in Fig. 2c. The lattice fringe is $d = 2.11$ Å. Several important d values are measured to be 2.45, 2.11, and 1.49 Å from Fig. 2c, which well match the spacing values of $(1\bar{1}0)$, (002), and $(2\bar{2}0)$ planes of wüstite FeO, respectively. These results confirm the embedded nanoparticles are fcc FeO. Fig. 2d is the enlargement of the HRTEM image for the olivine host (white square) and its FFT pattern along the zone axis of $[10\bar{3}]$. The diffraction spots are well indexed to be orthorhombic olivine. Although a few FeO and Fe_3O_4 nanoparticles have been observed along with a large amount of npFe0 in Apollo lunar soils [32], they are suggested to be generated by the diffusion of O atoms from the O-rich matrix to the surface. In contrast, the npFe^0 are not observed at all in our samples and the wüstite FeO nanoparticles are the only inclusion embedded in the amorphous rim. This unique rim structure has not been reported in any other lunar, terrestrial, Martian, or meteorite samples to the best of our knowledge.

Fig. 3a shows the HAADF image and elemental mapping of surface area. It reveals that the Si and O are richer in the outmost layer, and the Fe and Mg below a-SiO$_2$, consistent with line scanning in Fig. 1e. To determine the valence of Fe, we acquired the EELS for np-FeO and olivine marked by blue and red circles in Fig. 3b. The Si L-edge, O K-edge, Fe L-edges of FeO and olivine are plotted in Fig. 3c, in which the weaker Si and O intensity for FeO indicates that the np-FeO are embedded in the host olivine. The features of Si L-edge for the olivine and the amorphous SiO_2 layer are compared with several reference spectra. It indicates that they are similar to those for the SiO_2 rather than Si (Fig. S4a, online). Importantly, the Fe L_3-edges of np-FeO and olivine have the same energy of 708.45 eV, which is identical to 708.42(4) eV for the $FeCO_3$ standard sample (Fig. S4b, online), and larger than 708.08(5) eV for the Fe standard sample. Furthermore, we analyze the intensity L_3/L_2 ratios based on the method given in Fig. S5 (online). The L_3/L_2 ratios for the np-FeO and olivine are 3.99(11) and 4.01(12), respectively. Both values are close to the ratio of 4.03(5) in the $FeCO_3$ standard sample while much larger than the ratio of 2.99(7) in the Fe standard sample. All the Fe L_3-edge energy and L_3/L_2 ratios are tabulated in Table S1 (online).

It demonstrates that the nanoparticles are FeO rather than Fe [33–35]. Besides, we notice pores, 1-2 nm in diameter, exist in these np-FeO (Fig. S6 online), which could act as natural collectors for noble gases.

We checked more olivine grains, and found that they exhibit the same features of L-edge and L_3/L_2 ratio as the abovementioned grain (Fig. S7, online). In the thin slice of glassy beads, see Fig. S8 (online), np-FeO are found in amorphous matrix as revealed by the EDS and EELS analyses. We have checked the morphology of pyroxene grains, however, found there are no np-FeO and a-SiO$_2$ layers, see Fig. 4a, b. Meanwhile, the chemical compositions of nine selected areas at the surface are identical to interior parts as shown in Fig. 4c. For Ca-rich anorthite, identical chemical compositions are observed at the surface and inside, see Fig. 4d-f. More details are shown in Figs. S9-S11 (online). All observations imply that our measured samples do not undergo severe space weathering as Apollo and Luna samples did on the Moon.

It is interesting to compare our results to a recent paper about the rim structure and morphology of lunar soil returned by CE-5 [35], in which the authors found the npFe^0 (~35 nm) rather than wüstite FeO within the uppermost amorphous Si$_x$O$_y$ layers (5-20 μm in thickness) of large olivine grains. A notable distinction is that the solar flare trails are found in their olivine grains, but they are not present in our smaller grains at all. Based on the size and sharp edge of our samples, we propose that our samples are more likely to be small pieces of grains cracked off bigger olivine.

4. Conclusion

In summary, our findings reveal two major characteristics of CE5 lunar soil under space weathering. First, a multiple-layer-rim structure consisting of (1) outmost a-SiO$_2$ layer, (2) mixture of a-SiO$_2$, np-FeO, and MgO, (3) mixture of np-FeO, MgO, and olivine, (4) host olivine, is observed. Second, the npFe^0 and volatile elements S from vapor deposit are not observed within the rim, nor are the solar flare tracks inside the grain. As far as we know, such peculiar rim structures embedded FeO nanoparticles are not reported in the previous literatures. Given that the npFe^0 is the final product of decomposing olivine, we suggest that wüstite FeO may serve as an intermediate state of the thermal decomposition process, and the FeO may further transform into npFe^0 with the aid of cosmic radiation or solar flare. Our findings imply that our studied samples do not undergo severe space weathering, and the underlying mechanism deserves further investigation.

Conflict of interest

The authors declare that they have no conflict of interest.

Acknowledgments

Author contributions

Supplementary materials

Supplementary data are available online.

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Fig. 1. Morphology and chemical composition of olivine from CE5. (a) HAADF-STEM image of an olivine grain. The upper region 1 and lateral region 2 are enlarged in (b) and (c), respectively. The thin amorphous layer is labeled by white arrows. (d) HAADF image showing location (red arrow) of EDS mapping for olivine edge. (e) Line profile of atomic fraction in the red arrow zone in (d). Zero position is at the outermost surface. (f) Histogram of the nanoparticles' sizes in (b) and (c), showing their size in the range of 3-12 nm. (g) FFT pattern of an area of mixed nanoparticles and olivine matrix. The right panel is a simulated diffraction pattern of wüstite fcc-FeO.

Fig. 2. Structural details of amorphous SiO_2 layer, np-FeO and olivine host. (a) HRTEM image around the olivine surface. There are three typical zones (I, II, and III) within the rim. Green, red, and white areas are selected for detail analysis. (b)–(d) HRTEM and indexed FFT patterns of selected amorphous SiO_2 layer, np-FeO and olivine host, respectively.

Fig. 3. Elemental mapping and calibration of Fe's valence. (a) HAADF image of surface area and elemental mapping of Si, O, Fe, and Mg. (b) HAADF-STEM image during EELS acquisition. FeO and olivine areas are marked by blue and red circles, respectively. (c) EELS of Si L-edge, O-K edge, and Fe-L edge of marked np-FeO and olivine in (b). In the right panel, the Fe-L edge of Fe standard sample is plotted for comparison.

Fig. 4. Morphology and chemical composition of pyroxene and Ca-rich anorthite. (a)-(c) HAADF images and nine selected areas at pyroxene's surface. Zoom-in image around areas 2 and 6. EDS of nine areas. (d)-(f) HAADF image and eight selected areas at Ca-rich anorthite's surface. Zoom-in image around areas 4 and 8. EDS of eight areas.

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