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Simulation of nanophase iron production in lunar space weathering

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ABSTRACT

Nanophase iron (np-Fe⁰) particles produced by space weathering have been widely observed in lunar soil. Current research suggests that np-Fe⁰ could have important effects on the chemical, optical and magnetic properties of the lunar soil. To investigate the relationship between np-Fe⁰ and these properties of lunar soil, simulation of the production process of np-Fe⁰ by space weathering is necessary because of the scarcity of lunar samples for research purposes. New methods using microwave heating and magnetron sputtering techniques to simulate np-Fe⁰ production both in the glass phase and on the grain surfaces, respectively, are investigated in this study. Both the formation and occurrence of np-Fe⁰ are taken into account in the experiment. The X-ray Diffraction (XRD) spectra show that metallic iron has formed in the glass phase produced by microwave heating of ilmenite. Using scanning electron microscope (SEM) and energy dispersive spectrometer (EDS), the size of np-Fe⁰ particles produced in a microwave heating experiment, which is held for 8 min at 1300 °C, is determined to be about 100-500 nm. Compared to the glass of lunar sample 10084, the major composition of the glass matrix is formed by microwave heating compares favorably. In magnetron sputtering experiment the size of np-Fe⁰ particles is about 20-30 nm, and appears on the grain surfaces. The characteristics of np-Fe⁰ produced in the simulations are consistent with those of lunar samples documented in the literature. © 2011 Elsevier Ltd. All rights reserved.

1. Introduction

Unprotected by an earth-like atmosphere and magnetosphere, the exposed lunar surface has been subjected to several harsh space processes for eons. Among them, meteorite impact and energetic charged particle implantation are the dominant forces that reconstructed the lunar surface. Collectively, these processes are known as "space weathering". Meteorite bombardment, especially micrometeorite bombardment, could result in crushing, melting and vaporization of lunar surface materials. Lunar rocks are broken into smaller pieces, accompanied by the formation of considerable glass (typically 40-75%) from bombardment-produced local melting. During the bombardment process, the vapor produced might escape and condense simultaneously. In addition, energetic particles from solar wind, solar flares and cosmic rays are implanted in the lunar soil grains, resulting in radiation damage and sputtering condensation on the grains surfaces (Housley et al., 1974; Keller, 2002).

An important consequence of lunar space weathering is the production of nanophase iron $(np-Fe^{0})$. In studies of lunar samples from Apollo and Luna missions, it is found that lunar

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soil contains abundant np-Fe⁰ both in the agglutinatic glass produced by micrometeorite bombardment and in thin amorphous rims surrounding lunar soil grains formed by vapor deposition and irradiation effects, especially in the mature soil (Keller and Clemett, 2001; Lucey et al., 2006). The difference in states of np-Fe⁰ is considered to be associated with the different space weathering processes. The np-Fe⁰ particles in agglutinatic glass are mainly produced by micrometeorite bombardments, in which lunar surface materials saturated with hydrogen implanted from solar wind are melted, and the ferrous minerals are reduced to metallic iron. In previous studies, the size of np-Fe⁰ particles was found to be about 3-33 nm (Housley et al., 1972, 1973). But recent studies show that the np-Fe⁰ particles in agglutinatic glass are much larger, and most of them are about 50-200 nm in size and finely dispersed (James et al., 2002, 2003; Basu, 2005). The np-Fe⁰ particles appear as inclusions in a thin (50–200 nm) rim surrounding the surfaces of grains, which has resulted from vapor deposition by micrometeorites bombardments and sputtering deposition from the action of solar wind, solar flares and cosmic rays. The size of np-Fe⁰ particles in the rim ranges from several nanometers to tens of nanometers (Keller and McKay, 1992, 1993, 1997: Keller et al., 1998: Wentworth et al., 1999: Basu, 2005).

Due to the presence of the np- Fe^0 particles produced by space weathering, the chemical, optical and magnetic properties of lunar soil are modified. The variation of lunar soil properties

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may depend on the distribution, size and content of np-Fe⁰ in lunar soil (Keller et al., 1998; Keller and Clemett, 2001). Understanding the correlation between the characteristics of np-Fe⁰ particles and the properties of lunar soil is important for the interpretation of remote sensing data, and for future in-situ resource utilization (Hapke et al., 1975; Lucey, 1995). Due to the complexity of the lunar soil, simulation of np-Fe⁰ produced by space weathering in the laboratory is necessary.

At present, three different methods have been reported to simulate np-Fe⁰ particles in lunar soil simulants. The first method uses porous silica gel powders impregnated with ferric nitrate solutions to produce iron in silica (SiO_2) glass. Different sizes (2.3. 6. 25 and 50 nm) of gel pores and normalities (0.001-1.0 N) of ferric nitrate solutions were used to produce the analogs with different np-Fe⁰ sizes and contents (Noble et al., 2003, 2007). By this method, only the np-Fe⁰ in SiO₂ glass phase is simulated. In fact, the composition of the glass matrix varies dramatically. Liu et al. (2007) further developed this method, and synthesized np-Fe⁰ in amorphous silicate glasses with two-component (SiO₂-FeO) and five-component (SiO₂-Al₂O₃-MgO-CaO-FeO) mixtures. The exact content of np-Fe⁰ produced is difficult to control in this method. This process of synthesizing np-Fe⁰ in glass does not simulate the real formation process of np-Fe⁰ in the agglutinatic glass, because the metallic iron is reduced from several chemical materials in the sub-solid state in these experiments rather than directly from the agglutinatic glass. The second method is the direct heating of glass and mineral samples in hydrogen gas. After heating to 1100 °C and maintaining for 3-4 h, sub-micrometer iron metal blebs on the surfaces and within the volumes of glass and mineral grains were produced (Allen et al., 1993). However, the size of the Fe grains is up to 1µm, which is much larger than that in typical lunar agglutinitic glass (50-200 nm). The third method is the irradiation of olivine, pyroxene or their mixture with a pulse laser beam. A solid-state Nd-YAG pulse laser with 20 Hz impulse frequency, 1064 nm wavelength and 6-8 ns pulse duration was used. The analysis of TEM showed that np-Fe⁰ particles (several up to 30 nm in diameter) were widely scattered throughout the amorphous rims (\sim 200 nm in thickness) that had developed along the olivine grain surfaces. However, no such amorphous rims could be identified on the irradiated pyroxene grains surfaces. The radiation experiments have only been performed on selected individual minerals (olivine and/or pyroxene), and the np-Fe⁰ particles that appeared in the irradiated pyroxene were different from those seen in the lunar samples (Yamada et al. (1999); Sasaki et al., 2002, 2003).

Laboratory simulation of np-Fe⁰ production in lunar space weathering should be a more realistic approach to model the production process of np-Fe⁰ in lunar soil. In this study, new experimental methods using microwave heating and magnetron sputtering have been used to simulate production of np-Fe⁰ both in agglutinatic glass and on lunar simulant soil grains, respectively.

2. Experiments

2.1. Microwave heating experiment

The np-Fe⁰ in agglutinatic glass is believed to be produced by micrometeorite bombardment, which melts lunar soil grains via impact heating, transforming reduced Fe²⁺ in the melt to Fe⁰ through reaction with implanted hydrogen. Ilmenite is probably melted first because of its lower melting point and is more easily reduced to metallic iron compared to other silicate minerals in this process. To better simulate this process we propose the use of microwave energy because it could instantly heat and melt

minerals with large dielectric loss while other minerals with small dielectric loss are less affected (Kelly and Rowson, 1995).

As a dominant oxide in lunar soil, ilmenite has a large dielectric loss and can easily be reduced. So, ilmenite is a good candidate to simulate the formation of np-Fe⁰ in agglutinatic glass produced by molten reduction at high temperature. An ilmenite sample from the Panzhihua iron deposit in the southwest of China was prepared for our microwave experiment. The ilmenite sample (with some olivine and pyroxene < 5%) was ground and sieved through a 200 meshes sieve in order to increase its coupling with the microwave energy. To better simulate the dry condition on the lunar surface, the ilmenite sample was pre-heated in an oven to 105 °C for 3 h to remove water from the sample. The sample for each experiment weighed about 30 g.

The sample was then put in a silicon carbide crucible, and heated in a NJZ4-2 microwave furnace operated at 2.45 GHz and 4 kW. During the heating process, the sample temperature was monitored by an infrared thermometer. The furnace was evacuated, purged with argon and then maintained at a low pressure $(\sim 100 \text{ Pa})$. Holding time has an obvious effect on the size of the metallic iron particles. Short holding times may result in an insufficient reaction and produce less metallic iron, while long holding times may cause the metallic iron to combine in the melt, and increase the size of the metallic iron particles. To compare the effect of holding time to the size of metallic iron in the product, four ilmenite samples were heated to 1000 °C and then kept for 2, 5, 8, 10 and 30 min, respectively. Another sample was put in an alumina crucible and was heated to 1250 °C for 30 min as a comparison. In our experiment, the ilmenite samples were quickly heated to 1000 °C in about 10 min, with the pressure in the microwave oven rising continually. Finally, the samples were cooled down rapidly in argon flow to make the material form in a glass phase and also restrain the growth of metallic iron in the products. The products were cooled to below 100 °C after about 1 h.

2.2. Magnetron sputtering experiment

Besides the np-Fe⁰ found in agglutinatic glass, metallic iron found on the rims of lunar soil grain surfaces might be caused by vapor deposition from micrometeorites bombardment and sputtering deposition from solar wind radiation. Magnetron sputtering is a physical vapor deposition (PVD) technique, which might simulate the process of iron deposition on the lunar soil grains more realistically. In the magnetron sputtering process, the target is bombarded by energetic ions generated in an electromagnetic field. The ejected atoms escape from the target and eventually deposit on the sample surface (Kelly and Arnell, 2000). All types of minerals such as olivine, pyroxene, plagioclase, ilmenite and quartz in lunar soil have been found surrounded with amorphous rims that contain np-Fe⁰. Because the chemical, physical and geological properties of the Chinese Lunar Regolith Simulant (CLRS-2) prepared by Chinese Academy of Sciences are similar to Apollo 11 high-Ti basaltic lunar soil, CLRS-2 is selected for the magnetron sputtering experiment. CLRS-2 was the high-Ti lunar soil simulant of CAS series. Major components of CLRS-2 are pyroxene, plagioclase, olivine, ilmenite and volcanic glass, with their particle sizes ranging from 80 to 110 μ m (Zheng et al., 2009).

In our experiment, a magnetron sputtering ion plating system was used. To better simulate the dry condition on the lunar surface, the CLRS-2 sample was pre-heated in an oven to 105 °C for 3 h to remove water from the sample. The sample for the experiment weighed about 50 g and was placed on a 10×10 cm box inside the platform in the system. The size and abundance of the np-Fe⁰ particles in the final product could be controlled by the sputtering power and the sputtering time. In general, the size of np-Fe⁰ increases as the power increases, and the abundance of

np-Fe⁰ increases as the time increases. The cavity is evacuated to a pressure of 3×10^{-3} Pa over about 3 h. There are three steps in the magnetron sputtering experiment: ion cleaning, transition and iron-plate. The initial ion cleaning process requires a lower power (100 W) in order to prepare the sample particle surfaces for improving the attachment between grains and the iron atoms to be sputtered. After 10 min of ion cleaning, the power was increased to 400 W for 6 min for plating the iron. The power required by the iron-plating process was also 400 W, and in this third phase, iron atoms bombarded by argon ions escaped from the target (99.9 wt% Fe) and then deposited onto the CLRS-2 grain surfaces. The iron-plating process lasted for 10 min.

3. Result and discussion

3.1. Np-Fe⁰ in glass

X-ray diffraction (XRD) was used to identify the formation of glass and metallic iron phase in the sample. The XRD spectra with discrete peaks showed that a glass phase had been formed in the product, and the Fe⁰ peak in the spectra also showed that metallic iron blebs had been produced (Fig. 1). Scanning electron microscope (SEM) was used to characterize the products. These six products with different heating times were prepared for microscopy by embedding the particles in epoxy resin and sectioning the resin into thin slices. The SEM images and the energy dispersive spectrometer analysis showed that the ilmenite had reacted and produced a series of Fe-Ti-O, Ti-O and Fe-O components. As olivine and pyroxene were present in the initial ilmenite samples, there is some black glass with Si-Mg-Al-Ti-Fe composition in the final products. Some metallic iron grains were produced and dispersed randomly in the glass phase. Out of the six final products, the one, which had been held for 8 min at 1300 °C, produced the most submicron metallic iron grains, with

50

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Ilmenite

Ilmenite after microwave heating

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sizes of about 100-500 nm. Those, which were held for 2 and 5 min, were found to form fewer metallic iron grains, while the products, which had been held for 10 and 30 min in silicon carbide crucible, were found to form more large metallic iron grains with sizes of a few microns. The product, which was put in the alumina crucible. was rarely found to form any metallic iron (Fig. 2).

Some metallic iron particles in the black glass were formed in the product after microwave heating as shown in backscattered electron (BSE) image of electron microprobe (Fig. 3). Comparing this with the composition of agglutinatic glass in Apollo 11 sample 10084, the black glass in the microwave experiment has a similar chemical composition, especially regarding the major chemical components (Table 1). These results suggest that the microwave heating technique might be a good method to simulate the production of np-Fe⁰ in the glass phase by reducing ferrous materials into metallic iron.

In these microwave heating experiments, due to the power available to the microwave and the efficient microwave-absorbing properties of ilmenite, the samples were quickly heated to 1300 °C and reacted. The samples, which were put in the silicon carbide crucible, melted completely and produced a few metallic iron grains, while the sample, which was put in the alumina crucible at 1250 °C, did not melt completely and produced hardly any metallic iron. In the high temperature process the silicon carbide crucible may diffuse some C and reduce the iron in the ilmenite from Fe^{2+} and Fe^{3+} . The holding time of 8 min may be best for forming many submicron metallic iron grains in our six microwave experiments. Microwave heating of the ilmenite sample with some olivine and pyroxene could produce multicomponent glass with metallic iron, which is similar to the agglutinatic glass with np-Fe⁰ in lunar soil samples. Therefore, microwave heating of ilmenite may be a feasible method to simulate the production of np-Fe⁰ in agglutinatic glass in lunar soil simulants.

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Fig. 1. XRD spectrum of the ilmenite samples before and after microwave heating (kept for 8 min at 1300 °C).





Fig. 2. Images a–e are SEM images of products after ilmenite was heated by microwave energy and correspondingly kept for 2, 5, 8, 10 and 30 min at 1300 °C in a silicon carbide crucible. The image f is the product after ilmenite was heated by microwave energy and kept for 30 min at 1250 °C in an alumina crucible. The arrows point to the np-Fe⁰.



Fig. 3. BSE image of electron microprobe. Melted borders are found surrounding the grains. The white spots are the metal iron grains. Also shown are arrows pointing to the black glass, with some small metal iron particles (white spots) dispersed in the glass at random.

3.2. Np-Fe⁰ on grains surfaces

Magnetron sputtering was used to simulate np-Fe⁰ production on grains surfaces of sample CLRS-2. Metallic iron in the sample after magnetron sputtering was not identified by XRD, which might indicate that the content of metallic iron was lower than the detection limit (1%) of the XRD. Through other analyses, the sample after magnetron sputtering showed the following special characteristics:

- Formation of np-Fe⁰ particles: the sample after magnetron sputtering was examined by transmission electron microscopy (TEM). The results showed that np-Fe⁰ particles had deposited on some CLRS-2 grain surfaces. The average size of np-Fe⁰ is about 20–30 nm (Fig. 4). The size of the metallic iron blebs in the magnetron sputtering experiment is in the range of the actual size of the np-Fe⁰ particles observed in lunar soil (3–33 nm).
- 2) Change of magnetic properties: the sample after magnetron sputtering appeared to be weakly magnetized. Grains in the product could be attracted by a small magnet because of the appearance of metallic iron particles. Comparing the magnetic susceptibility between the samples before and after magnetron sputtering, the magnetic susceptibility of the sample after magnetron sputtering had increased 19.6 percent (Fig. 5).
- 3) Increased Fe content: an atomic absorption spectrometer (AAS) was used to measure the Fe content in the samples before and after magnetron sputtering. The result showed that the average Fe content of the sample after magnetron sputtering was 0.68 percent higher than that of the sample before

Table 1

Composition of glass in experimental sample and lunar sample 10084.

	TiO ₂	FeO	Al_2O_3	SiO ₂	CaO	MgO	MnO	Cr ₂ O ₃	Na ₂ O	K ₂ O
Experimental sample	3.9	20.0	2.7	50.1	11.4	12.0	1.4	-	-	-
Lunar sample 10,084 ^a	2.96	10.6	17.4	44.5	13.2	8.69	-	0.31	0.36	0.09

^a The composition data of lunar 10084 come from Taylor et al. (2001).



Fig. 4. TEM images of the np-Fe⁰ on the silicate minerals surfaces of the CLRS-2 formed by magnetron sputtering. The arrows point to the np-Fe⁰ particles, which are brighter than silicate minerals.



Fig. 5. Magnetic susceptibility of the samples before and after magnetron sputtering.



Fig. 6. Fe content in the CLRS-2 samples before and after magnetron sputtering.

magnetron sputtering (Fig. 6). The increased Fe content could be approximately considered as the amount of metallic iron in the sample after magnetron sputtering, which was consistent to the result of the XRD.

As a physical vapor deposition process, magnetron sputtering could make np-Fe⁰ particles to deposit on CLRS-2 grains surfaces. The result has shown that the magnetron sputtering technique is a useful method to simulate np-Fe⁰ production on grains surfaces.

4. Conclusion

It is widely believed that the formation of np-Fe⁰ particles in lunar soil is the result of micrometeorite bombardment as well as energetic particles from solar wind, and cosmic rays. In this paper we report two new methods to simulate the occurrence of np-Fe⁰ both in agglutinatic glass and on lunar soil grains surfaces in a laboratory environment.

Microwave heats the ilmenite samples to high temperatures at a very rapid rate, with effects similar to the rapid heating by micrometeorite bombardment on the lunar surface. Our results show that it could realistically simulate the production of np-Fe⁰ in agglutinatic glass used on ilmenite samples with size, distribution and chemical composition similar to that of lunar soil.

The magnetron sputtering technique offers a satisfactory method to simulate the vapor deposition and sputtering deposition on lunar soil grain surfaces. It can produce np-Fe⁰ particles adhered to the grain surface with a similar size range. Based on these two techniques, the variation in the properties of lunar soil caused by space weathering can be further studied.

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