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無容器凝固させたFeO-TiO$_2$酸化物の磁気特性

Magnetic properties of containerless solidified FeO-TiO$_2$ oxides

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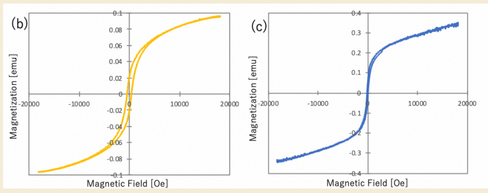
Abstract: This study aimed to develop ferromagnetic lunar regolith simulants for future in-situ resource utilization on the Moon. FeO-TiO$_2$ samples were synthesized by aerodynamic levitation (ADL) and arc melting to simulate high-temperature melting and rapid quenching from meteorite impacts. ADL samples under an Ar atmosphere were paramagnetic ilmenite ($FeTiO_3$), whereas oxygen-containing conditions produced a ferromagnetic solid solution with a Curie temperature of ~140 °C, indicating ilmenite-hematite solid solution formation. Arc-melted samples also showed ferromagnetic hysteresis, and thermomagnetic analysis revealed ferromagnetism from metallic iron precipitation, disappearing between 750-800 °C, with 1.30wt% Fe^0. These results provide insights into ferromagnetism in lunar regolith and guidance for designing ferromagnetic simulants for studies on the Moon's magnetic anomalies.



Keywords: Lunar Regolith Simulants, Ilmenite($FeTiO_3$), Aerodynamic Levitation, Magnetic Properties

1. Introduction

Research and development on the in-situ utilization of lunar resources have been actively advancing in recent years. Concepts for processing lunar regolith to build structures and extract metal resources have been proposed. To realize these concepts, it is necessary to understand the powder properties of lunar regolith under ultra-high vacuum and low-gravity conditions on the lunar surface, and to design suitable processes. It is expected that the gaps between lunar regolith particles differ between ultra-high vacuum and atmospheric environments, leading to variations in powder properties such as dielectric constant and thermal conductivity. Therefore, we have been studying the pressure dependence of the bulk density of lunar regolith simulants. Additionally, it is known that localized strong magnetic fields exist on the lunar surface[1)]. This magnetic anomaly is believed to result from the precipitation of metallic iron in regolith regions rich in Fe, which is thought to occur due to rapid cooling and solidification after high-temperature melting of regolith caused by meteorite impacts[2)]. In these magnetic anomaly regions, it is expected that the gaps between regolith particles differ from those in other areas because of magnetic forces. Consequently, regolith simulants containing particles exhibiting ferromagnetism are required.

From these backgrounds, we simulated the rapid cooling from high-temperature liquid states and the solidification process caused by a meteorite impact using an aerodynamic levitation method and an arc melting furnace, and synthesized $FeTiO_3$ (ilmenite) from FeO-TiO$_2$ powder. We evaluated the magnetic properties of the obtained samples, investigated the cause of ferromagnetism, and examined the process conditions for the creation of ferromagnetic regolith simulants.

2. Experiment

FeO powder and TiO$_2$ powder were mixed at a molar ratio of 1:1 and dry-ground for 30 minutes using a mortar and pestle. The mixed sample was pressed into pellets using a press machine at 19 GPa for 5 minutes. The pellets were then crushed to a suitable size for each melting method. In the aerodynamic levitation (ADL) method, the samples were levitated by Ar gas from a nozzle under both an Ar atmosphere and an oxygen-containing atmosphere. A CO_2 laser with a wavelength of 10.6 μm was irradiated from above the sample to achieve containerless melting, after which the laser irradiation was stopped to rapidly cool and solidify the sample. In the arc melting furnace, the samples were melted under an Ar atmosphere by arc discharge between a tungsten electrode and a copper hearth with a current of 50 A. Cooling water was circulated through the copper mold to rapidly cool and solidify the sample.

Magnetization measurements were performed using a vibrating sample magnetometer (VSM). The samples were vibrated in a magnetic field at a frequency of 80 Hz and an amplitude of 0.50 mm, and the induced electromotive force was detected to measure magnetization. Measurements were conducted at room temperature or during heating up to 800°C, with an applied magnetic field range of $\pm$18 kOe. For heating, the sample was fixed inside a cylindrical alumina ceramic tube, and heated with a heater during measurement to obtain thermomagnetic curves.

3. Result and Discussion

3.1. Magnetic Properties of ADL-Synthesized Samples under an Ar Atmosphere

Figure 1(a) shows the magnetic properties of the sample synthesized by the aerodynamic levitation (ADL) method under an Ar atmosphere. Since the magnetization changes linearly with the applied magnetic field, it is evident from the phase diagram[3] that paramagnetic ilmenite ($FeTiO_3$) has precipitated.

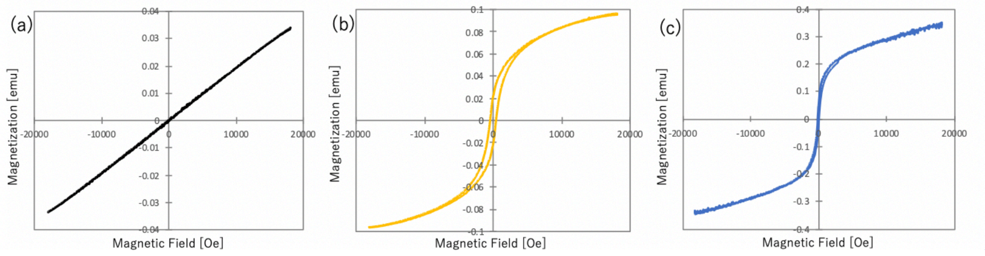


Figure 1. Magnetic curves of the synthesized samples at room temperature. (a) Sample synthesized by the ADL method under an Ar atmosphere. (b) Sample synthesized by the ADL method under an oxygen-containing atmosphere. (c) Sample synthesized by the arc melting furnace under an Ar atmosphere.

3.2. Magnetic Properties of ADL-Synthesized Samples under an Oxygen-Containing Atmosphere

As shown in **Figure 1(b)**, the sample melted under an oxygen-containing atmosphere exhibited a ferromagnetic hysteresis loop. To investigate the origin of the ferromagnetism, magnetization measurements were performed while heating the sample. The sample was heated until it lost its magnetization, and from the high-field region of the hysteresis loops at each temperature, an approximate straight line was obtained by the least squares method; the intercepts were then plotted (**Fig.2(a)**). The Curie temperature was estimated to be approximately 140°C. Based on the oxygen-containing atmosphere and the Curie temperature, it is considered that part of the FeO transformed into Fe_2O_3, forming a continuous solid solution with ilmenite ($FeTiO_3$)[4]. Samples recovered by the Chang' e 5 mission have been found to contain precipitates of metallic iron (Fe) and magnetite ($Fe_3O_4 = FeO{\cdot}Fe_2O_3$) within spherical iron sulfide particles formed by meteorite impact[5]. It is therefore quite possible that ferromagnetic ilmenite present in lunar regolith forms a solid solution with a small amount of hematite.

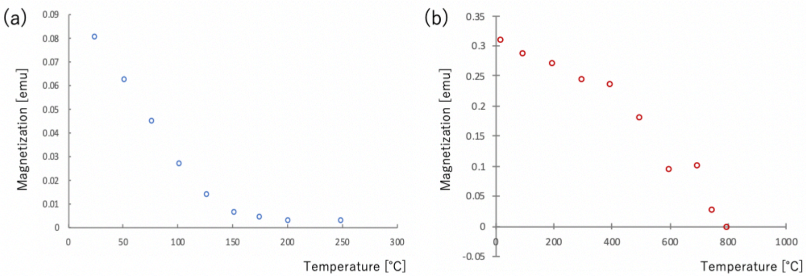


Figure 2. Thermomagnetic properties of the synthesized samples exhibiting ferromagnetism. (a) Sample synthesized by the ADL method under an oxygen-containing atmosphere. (b) Sample synthesized by the arc melting furnace under an Ar atmosphere.

3.3. Magnetic Properties of Samples Synthesized by the Arc Melting Furnace

As shown in **Figure 1(c)**, the sample melted in the arc melting furnace also exhibited a ferromagnetic hysteresis loop. Analysis of the thermomagnetic properties (**Fig.2(b)**) showed that the magnetization disappeared between 750°Cand 800°C, indicating that the ferromagnetism originated from the precipitation of metallic iron. From the hysteresis loop at room temperature (**Fig.1(c)**), a straight line was fitted to the high-field region, and the ferromagnetic hysteresis loop was extracted to calculate the mass saturation magnetization. The mass saturation magnetization (M_s) due to metallic iron in the synthesized sample was 2.70 emu/g. Given that the M_s of pure Fe was measured as 207.31 emu/g, the metallic iron content in the synthesized sample was estimated to be 1.30 %.

4. conclusion

In this study, motivated by the need for ferromagnetic lunar regolith simulants in future in-situ resource utilization, FeO–TiO_2 oxide samples were synthesized via rapid cooling and solidification using the aerodynamic levitation (ADL) method and an arc melting furnace, simulating the high-temperature melting and rapid quenching caused by meteorite impacts. The ADL method under an Ar atmosphere yielded paramagnetic ilmenite, whereas under an oxygen-containing atmosphere, a ferromagnetic solid solution with a Curie temperature of approximately 140°C was obtained. In contrast, the arc melting furnace produced metallic iron, with its content estimated to be about 1.30 wt%. These results not only provide valuable insights into the formation process and origin of ferromagnetism in ilmenite within lunar regolith, but also offer practical guidance for the design and processing conditions of ferromagnetic regolith simulants required for studies on regolith particle behavior and bulk properties in magnetic anomaly regions on the Moon.

Conflicts of Interest

The authors state no conflict of interest.

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