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無容器浮遊法で作成した Fe 系酸化物の磁性

Magnetic properties of Fe-based oxides prepared by containerless method

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1. Introduction

We have performed using an electrostatic levitation furnace installed in ISS an investigation of the thermophysical properties of the molten SiO_2-CaO-FeO system which is the basis for the steel-making slags. Since the oxide system includes Fe ions, it is interested in magnetic properties for the solidified oxide systems. The magnetic properties of the oxide system including Fe ions would depend on the atmospheric oxygen conditions because of the reaction for $4Fe^{3+} + 2O^{2-} \rightleftarrows 4Fe^{2+} + O_2$ at the interface between liquid and atmospheric gas state. Since under microgravity conditions the atmospheric gas around molten oxide has no convections, the difference in the magnetic properties of the oxides solidified on the ground is expected. From the background, we investigate the magnetic properties of the SiO_2-CaO-FeO oxide system solidified under microgravity and on the ground using the magnetization curve and its temperature dependence.

2. Experiments

2.1. Preparation of solidification samples of SiO_2-CaO-FeO system under microgravity and on the ground

Master samples of SiO_2:CaO:FeO = 20:20:60 mass% (Ox2) and SiO_2:CaO:FeO = 40:40:20 mass% (Ox6) were prepared from reagent grade SiO_2, CaO and FeO powders. Master samples were prepared by drying SiO_2, CaO, and FeO powders at 300°C for 14 hours and evaporating the H_2O contained in the raw powder. A weighed mixed oxides were placed in a cooled copper crucible, sintered by CO_2 laser irradiation at 1000°C in dry air, and prepared by the aerodynamic levitation (ADL) method. A sintered sample of approximately 2mm in diameter was levitated using an aerodynamic nozzle, and containerless melting and solidification was performed. After these procedures, selected samples without voids and air bubbles by X-ray radiograph checking were installed in ISS. These samples were levitated and melted in the dried air (N_2/O_2 mixture gas) atmosphere by the electrostatic levitation furnace (ELF) in ISS[1)] for the density measurements of the molten state and then they were solidified by stopping the irradiation of heating laser of 980nm wavelength with natural cooling. On the other hand, on the ground samples were levitated and melted in the air atmosphere by an aerodynamic levitation system with a CO_2 laser of 10.6 μ m-wavelength and after completely melting solidified by stopping the irradiation of the CO_2 laser.

Sample temperature was monitored by a monochromatic pyrometer with an emissivity of 1 both in ELF and on the ground. After measurements, we corrected the temperature emissivity of 1 to real temperature using the emissivity of the molten SiO_2-CaO-FeO measured by our proposed methods [2)]. **Figure 1** shows an example of temperature profiles during melting and solidification. As a result, the recalescence phenomenon, in which crystallization occurs due to the release of latent heat of solidification from the supercooled melt in both the aerodynamic levitation and the ELF was observed for Ox2, but not for Ox6. Therefore, Ox2 was found to be crystalline state, while Ox6 was glass state with an amorphous structure.

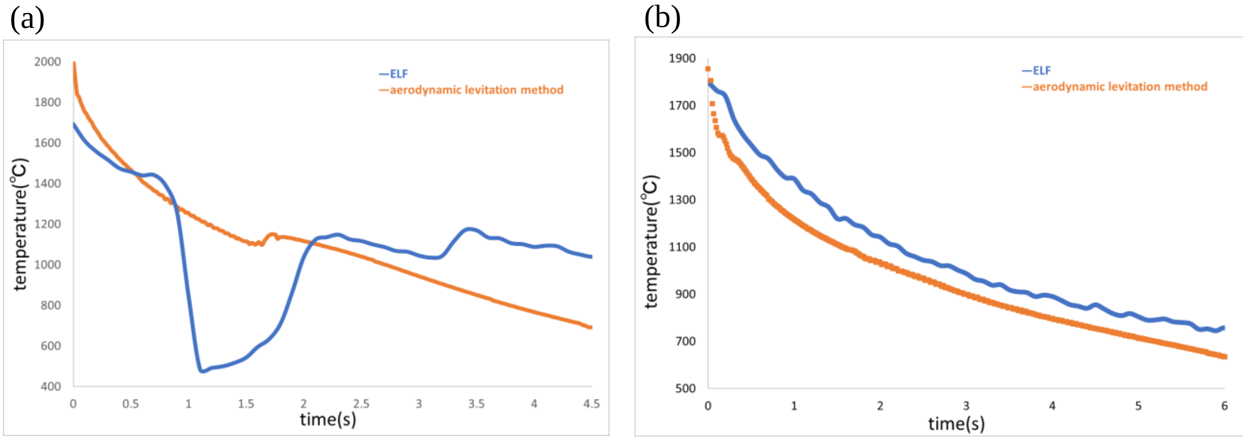

Figure1. Temperature profiles during cooling in ADL and ELF for (a) Ox2 and (b) Ox6 samples.

2.2. Magnetic property measurement from the magnetization curve by VSM method

The oxide samples using the above procedure in the ELF and on the ground were investigated for their magnetic properties. The magnetic properties investigations were performed using a vibrating sample magnetometer (VSM) from room temperature to 1200℃. From the VSM measurements, we obtained the magnetization curve in which the magnetization of samples is changed with the external magnetic fields. From the magnetization curve and its temperature dependence, we can identify the type of magnetic properties and the origin of magnetic properties. Moreover, the sample surfaces and inside were observed by scanning electron microscopy (SEM) with energy-dispersed X-ray analysis (EDX) for investigation of morphology and segregation of ions.

3. Result and discussion

Figure 2 shows the sample surface morphorogy by SEM observations. The appearance of the sample surface differs depending on the composition, with Ox2 showing crystals precipitated on the surface. On the other hand, the surface of Ox6 is quite uniform, and this image confirms that it is formed of glass state. **Figure 3** shows the magnetization curve by VSM measurements of Ox2 and Ox6 samples made by the ADL method and the ELF onboard the ISS. The results show that both samples of Ox2 have ferromagnetic properties. On the other hand, Ox6 does not have ferromagnetic properties, it can be confirmed that the magnetization slightly changes with the value of the magnetic field, and in particular, the ELF sample has a paramagnetic behavior in which the magnetization is proportional to the applied magnetic field. In addition, a comparison of the Ox2 magnetization of the ELF sample with that of the ADL sample showed that the saturation magnetization of the ELF sample was smaller than that of the ADL sample. The reason for this is thought to be that the ELF and ADL methods both melted in air composition gas, but in ELF no gas convection conditions around the molten samples, and the different atmosphere around the sample would result in a difference in the presence of Fe^{3+} state in the sample[3,4].

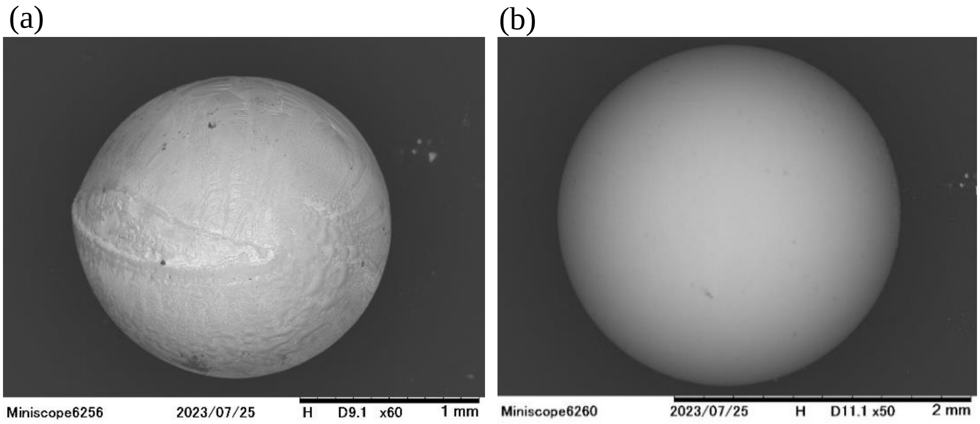

Figure2. SEM images of samples solidified in ELF under microgravity for (a) Ox2 and (b) Ox6 compositions.

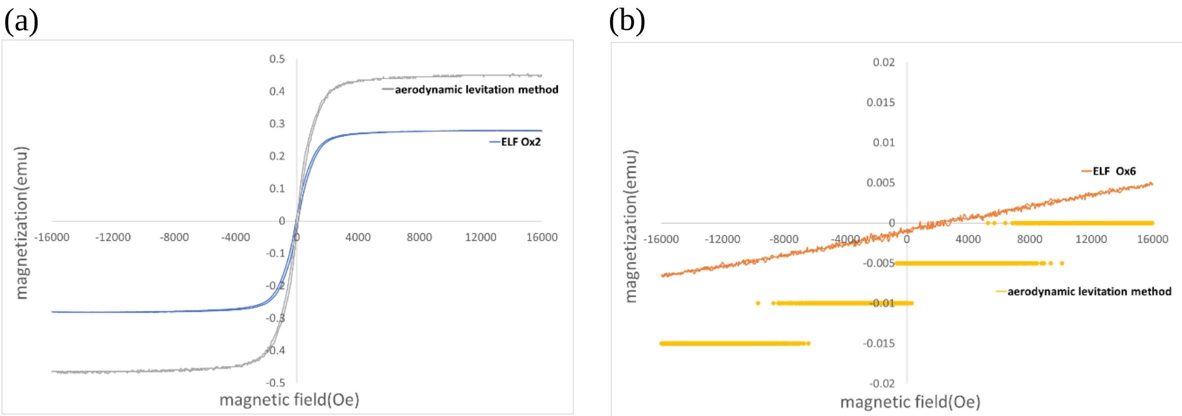

Figure 3. Magnetization curve using VSM at room temperature of samples solidified by the ADL method and ELF for (a) Ox2 and (b) Ox6 compositions.

4. Conclusion

SiO_2-CaO-FeO oxides were solidified using the aerodynamic levitation method on the ground and ELF under microgravity, and the magnetic properties were compared ADL samples with those of an ELF sample of the same composition. Both samples exhibited magnetization with hysteresis loops depending on composition. The comparison revealed a significant difference in the magnitude of magnetization between the ADL sample and the ELF sample, with the ELF sample being smaller. The cause of this difference is thought to be the difference in the presence of Fe^{3+} state that causes the magnetism, but the details are not yet known. We will investigate the details of the origin of the magnetization of the oxide system through temperature dependence of magnetization-curve and compositional analysis in the future.

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