OS4-2

溶接フラックス用酸化物融体の ELF による熱物性計測

Thermophysical Property Measurement of Welding Flux Oxide Melt by Electrostatic Levitation Furnace (ELF) in ISS

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1. Introduction

We have performed an onboard experiment for investigating thermophysical properties using an electrostatic levitation furnace (ELF) in International Space Station (ISS). Our aim of thermophysical property measurements is to determine the interfacial tension between iron melt and molten oxide, which is crucial in steel production. In such steel production, the welding process much requires the interfacial tension between liquid Fe and molten oxides. For welding processes, a multicomponent oxide system is used as a flux covering on liquid Fe. The compositions of multicomponent oxide are adjusted to control interfacial properties with temperature dependence during welding processes. The oscillating drop method with the core-shell droplet by iron melt and molten oxides is used to measure the interfacial tension between iron melt and molten oxides[1,2)]. For the method to obtain precisely interfacial tension, we need the multicomponent molten oxide density, surface tension, and viscosity. The multicomponent oxide system is difficult to measure on the ground by an electrostatic levitation technique from the problems of compositional change by evaporation under high vacuum conditions. Therefore, we used ELF in ISS to measure them under the pressurized gas condition. For the measurement, we developed the temperature correction procedure for molten oxide samples during measurement in ELF using the normal spectral emissivity of molten oxides[3)]. Our present results of temperature dependence of density of SiO_2-CaO-Mn_3O_4-TiO_2 systems are presented.

2. Experiment

Thermophysical properties of molten oxide of SiO_2:CaO:Mn_3O_4:TiO_2=27:7:13:53 wt.% were measured using ELF in ISS. The ELF system has been previously described[4-6)]. In this report, we focus on density and the temperature dependence of molten SiO_2-CaO-Mn_3O_4-TiO_2 system of model composition used as the welding flux. Our samples, which contained many elements, were synthesized as follows: The staring materials were commercially available high-purity (99.99%) powders of SiO_2, CaO, Mn_2O_3, TiO_2. These powders were dried in a 400 ℃ oven for 24h. Their masses were measured to adjust the compositional ration of SiO_2:CaO:Mn_3O_4:TiO_2=27:7:13:53 wt.%. Then, the starting powders were pre-sintered on the water-cooled Cu plate using a CO_2 laser. The pre-sintered oxides were completely melted using the aerodynamic levitation method with a CO_2 laser to make 2-mm diameter spherical shape samples. The spherical samples were subsequently weighted, transferred into special sample holders, and launched to the ISS. Our molten oxide density measurements for the SiO_2-CaO-Mn_3O_4-TiO_2 system followed the same procedures as previously reported[7,8]. During the measurement processes, we monitored the sample temperature using an emissivity vale set to 1 and recorded the monitored temperature. For the monitored temperature by setting emissivity to 1, we employed our proposal approach[3] using the normal spectral emissivity to correct the sample's temperature during the measurement processes. The density of molten oxide of SiO_2-CaO-Mn_3O_4-TiO_2 system was also measured using the samples synthesized simultaneously by the aerodynamic levitation (ADL) method as ELF samples. The measurement processes are same as the same those previously reported[9,10], but the measured temperature was corrected using the normal spectral emissivity obtained by EML experiments[3].

3. Result and discussion

Figure 1 shows our measurement results of molten oxide of SiO_2:CaO:Mn_3O_4:TiO_2=27:7:13:53 wt.%. ELF density measurements were performed under dry air conditions with 2 atm pressure to avoid composition changes due to evaporations. However, after samples were returned to the ground, we found that several of the m had lost over 1% of their masses, regardless process conditions. Therefore, we omitted the density for samples with a mass loss of more than 1% in the results. The uncertainty of density measurements using ELF in ISS is 2.5% which is the same as that presented by Ishikawa et al.[11]. Alternatively, the uncertainty of density measurements using ADL technique is 1.8%[9,10]. The resolution of the levitated molten sample images recorded by the observation camera with the backlight system causes the variation in density measurement uncertainty. Because the camera system difference, the employed ADL system has the smaller uncertainty in volume measurements of levitated molten samples. The temperature during measurements was corrected using the normal spectral emissivity of 0.76 and the correction factor of 1.14 to produce the density results at the various temperature shown in **Figure 1**. The maximum temperature difference between the original and corrected temperatures was about 170K. we determined the density-measured temperature range against the liquidus temperature by correcting the temperature during the procedures. **Figure 1** shows the liquidus temperature of SiO_2:CaO:Mn_3O_4:TiO_2=27:7:13:53wt.% simulated using Thermo-Calc of a software package. The density measurement range covers a large undercooled regions by comparing the density-measured temperature to the liquidus temperature. However, the obtained density cannot correspond to the liquid or the amorphous in undercooled temperature regions. No glass transition signals were observed from the cooling curves during the experiment. In the future, we will clarify the liquid state region in undercooled regions by combining the oscillating drop results. **Figure 1** also shows the plots of density results measured by ADL. For the ADL result, almost the constant temperature dependence of density was shown in the figure. We conclude that the temperature dependence of the molten-oxide multicomponent system is determined, and we found that the composition of molten oxides is almost non-expanding with increasing temperature. Moreover, we found that if an amorphous state of SiO_2:CaO:Mn_3O_4:TiO_2=27:7:13:53 wt.% oxides exists in undercooled temperature regions, the amorphous density and temperature dependence are identical to the liquid state. For a multicomponent molten slag system containing SiO_2, temperature coefficient is in the order of 10^{-4} [12,13]. By comparing the slag system containing SiO_2, the a of our SiO_2-CaO-Mn_3O_4-TiO_2 oxide system is extremely small. Chemical bonding related to the melting temperature dominates the volume expansion for the single element oxide case. For high melting temperature oxides, because the chemical bonding between metal elements and oxygen is strong, the volume expansion with increasing temperature is small. However, numerous reasons for the temperature coefficient, a, variations exist for a multicomponent slag system. The short-range atomic arrangement network in a multicomponent molten oxide containing SiO_2 aggregates, and the aggregated network does not make free volume space as temperature increases. If the rigid network is in a molten state, the viscosity will also be typically affected by temperature. To test this hypothesis, we should carefully analyze the temperature dependence of viscosity in the future.

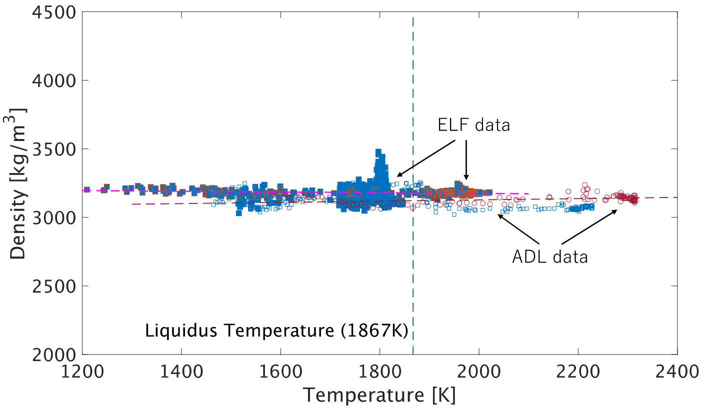

Figure 1. Temperature variation of molten SiO_2-CaO-Mn_3O_4-TiO_2 system measured by ELF in ISS and ADL method on ground.

4. Conclusion

We corrected the temperature monitored with emissivity set at 1 to the real temperature during density measurement by ELF in ISS using our proposal procedure by the normal spectral emissivity for molten SiO_2-CaO-Mn_3O_4-TiO_2 system, and we found the temperature dependence of molten oxides of SiO_2:CaO:Mn_3O_4:TiO_2=27:7:13:53 wt.%. The multicomponent oxide system of SiO_2-CaO-Mn_3O_4-TiO_2 has small temperature coefficient of temperature dependence of density, which means that the molten oxides of SiO_2:CaO:Mn_3O_4:TiO_2=27:7:13:53 wt.% has a small volume expansion system in molten states, according to the results of density measurements in ELF in ISS. Moreover, our density measurement was widely covered in undercooled temperature regions. In the regions, the amorphous sate was present, but we were unable to identify the difference between the liquid and amorphous states from the temperature dependence of density and cooling curves. In the future, we will analyze and discuss the temperature dependence of density combined with the temperature dependence of viscosity measured simultaneously with density measurements based on our findings.

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