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IIIII Review IIIII

Thermophysical Property Measurements of Oxide Melts at High Temperature by Electrostatic Levitation Furnace on the ISS

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Abstract

The electrostatic levitation furnace (ELF) for the International Space Station (ISS) has been developed and sent to the ISS. The main target of the furnace are oxide samples, which are difficult to levitate in 1-G. Thermophysical properties such as density, surface tension, and viscosity of molten oxides at high temperature will be measured using microgravity condition in the ISS. In this paper, the ISS-ELF and the first ISS experiment (thermophysical property measurement of some oxide materials) are introduced.

Keyword(s): Electrostatic levitation, Thermophysical property, Oxide

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1. Introduction

The use of a containerless technique for materials processing has many technological and scientific advantages. The absence of a crucible allows the handling of chemically reactive materials such as molten refractory metals, alloys, semiconductors, or oxides and eliminates the risk of sample contamination in overheated as well as in undercooled states. The lack of a crucible also suppresses nucleation induced by the walls of a container (heterogeneous nucleation) thus increasing the possibility of producing new materials such as glasses. Several levitation methods, including acoustic[1)], electromagnetic[2)], aerodynamic[3)] and electrostatic[4)] have been applied for containerless processing in space as well as on the ground.

Japan Aerospace Exploration Agency (JAXA) has been designed and developed the electrostatic levitation furnace since 1993. Through a sounding rocket experiment conducted in 1997[5,6)] and following ground-based research, several key technologies necessary for stable sample positioning and scientific diagnostics have been developed[7,8)]. Finally, an electrostatic levitation furnace for the International Space Station (ISS-ELF) has been fabricated[9-11)] and has been launched in this summer (Aug. 2015). Molten oxide materials, which are very difficult to be levitated and melted on the ground electrostatic levitators, will be mainly processed in the ISS-ELF. This paper describes some features of the ISS-ELF and thermophysical property measurements of molten oxides.

2. Electrostatic Levitation Method

The electrostatic levitation method utilizes the Coulomb force between the sample and surrounding electrodes to cancel the gravity force. **Figure 1** illustrates the hardware arrangement for the position control on the ground based electrostatic levitation system[4)]. A positively charged sample is levitated between a pair of parallel disk electrodes (top and bottom electrode), typically 10 mm apart, which are utilized to control the vertical position of the specimen. The typical sample size is 2 mm in diameter and an electrical field of around 8 to 15 kV/cm is necessary to levitate it against gravity. In microgravity condition, this

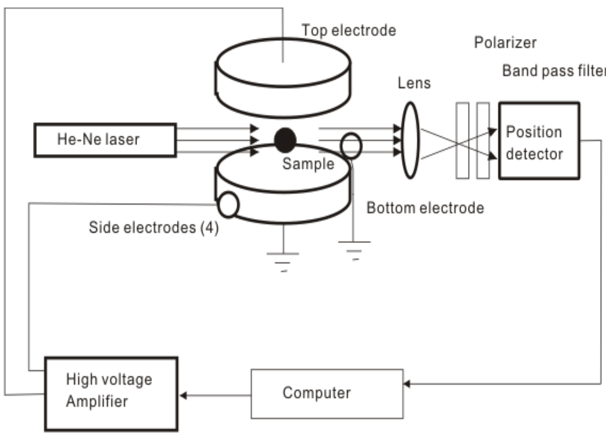

Fig. 1 Schematic drawing of sample position control system on the ground based electrostatic levitator.

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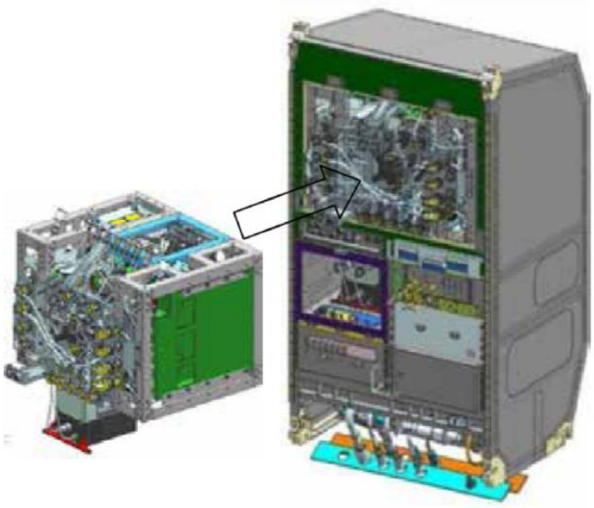

Fig. 2 Conceptual drawing of the ISS-ELF installed in the MSPR

electrical field will be minimized to 10 to 100 V/cm. In addition, four spherical electrodes distributed around the bottom electrode are used for horizontal control. Since the electrostatic scheme cannot produce a potential minimum, a feedback position control system is necessary. Position sensing is achieved with a set of orthogonally disposed laser that projected a sample image on a position sensor. The beam of a He-Ne laser is expanded and impinged on a levitated sample. The size of the resulting sample shadow is optimized with a lens to cover the area of the sensor such that a good dynamic range is obtained. In addition, a polarization filter is used to optimize the laser intensity reaching the sensor. The sample position information read by the sensor is then fed to a computer and analyzed by a program. The program uses PID (Proportional Integral Derivative) servo algorithms for implementing the feedback system, thus allowing a sample to maintain a fixed position in time. The computer then computes new values of voltages for each electrode. The typical feedback rate is around 600 Hz for the vertical direction.

3. Electrostatic Levitation Furnace for the ISS (ISS-ELF)

The ISS-ELF will be installed into the Work Volume (WV) of the Multi-purpose Small Payload Rack (MSPR) in the Japanese Experiment Module (JEM), as shown in **Fig. 2**. Detailed explanation about the hardware has been published elsewhere[11]. **Table 1** shows the main specification of the ISS-ELF.

Due to the limitation of space and electrical power, vacuum pumps necessary to get high vacuum environment have been omitted. This is one of the reasons why ISS-ELF will mainly process oxide samples. However, a vent line is connected to the evacuation system provided by JEM so that low vacuum level (around 10^{-3} Torr) can be obtained. This vacuum line will be

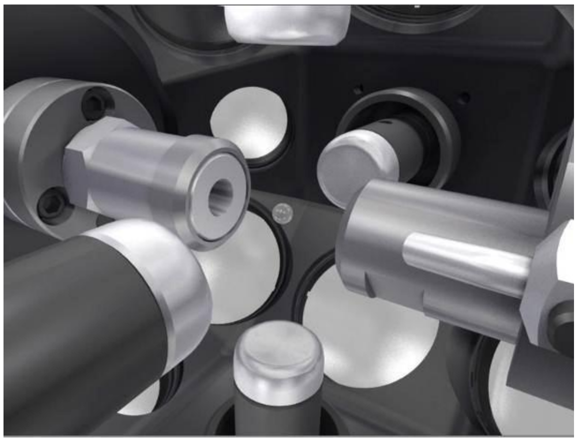

Fig. 3 Arrangement of electrodes in the ISS-ELF.

Table 1 Major Specifications of the ISS-ELF

Item	Spec.
Facility Size	(Main) 590 x 887 x 787 mm (UV Lamp) 226 x 259 x 347 mm
Mass	about 245 kg
Maximum power consumption	550 W
Sample size	φ1.5～2.1 mm
Sample materials	Metals, Alloys, Glasses, Oxides etc.
Positioning Control	• Absolute position accuracy :±300μm • Stability: ±100 μm • Max. control frequency: 1 kHz
Atmosphere	• Ar / N_2 / N_2 + Air up to 2 atm • Low pressure around 10^{-3} Torr
Heating	• 4 semiconductor lasers • Wave Length: 980 nm / • Total power 160 W.
Temperature measurement	Pyrometer (100 Hz) Measurement Range: 300~3000℃
Cameras	• 1B/W camera for density measurement (with telephoto objective lens) • 1 color camera for wide view • 1 color camera combined with a pyrometer
Thermophysical property measurement	Density (image analysis) Surface tension and viscosity (drop oscillation)

used to get Ar gaseous environment in which oxygen concentration is minimized.

Since there is no strong G-vector in the ISS, electrodes for position control are isotropically arranged. Observation windows are placed three dimensionally to minimize the chamber size (**Fig. 3**).

Four semiconductor lasers (wave length 980 nm) with high efficiency (as high as 50 %) are used for sample heating. Without these high efficiency lasers, ISS-ELF could not be designed. However, oxide samples generally have low absorption on this wave length. Therefore, compatibility between samples and the heating laser should be checked as a part of sample preparation. Temperatures of the levitated

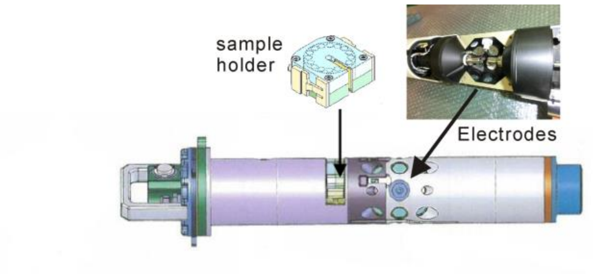

Fig. 4 Details of sample cartridge. It contains electrodes and a sample holder.

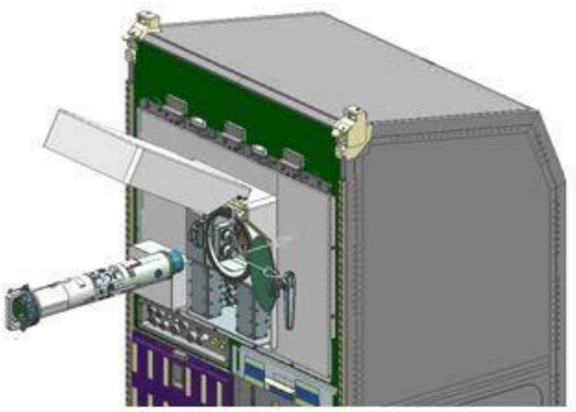

Fig. 5 Sample cartridge inserted into the ISS-ELF chamber.

samples will be measured by a pyrometer which measures the light intensity from the sample at 1.5 μm in wave length. The spectral emissivity of the sample at this wave length should be measured on the ground to get an accurate temperature in the ISS beforehand.

Samples are installed in a sample holder, which is inserted to the sample cartridge (**Fig. 4**). The sample cartridge also contains electrode assembly in which a sample is levitated. Finally, the sample cartridge is inserted into the ISS-ELF chamber, as shown in **Fig. 5**.

Except for the initial hardware installation into the MSPR and sample exchange, all ISS-ELF operations will be conducted from the ground via tele-communication.

4. Thermophysical Property Measurement in the ELF

Since the levitated sample takes a spherical shape, the volume of the sample can be easily obtained from the image analyses of the magnified sample pictures [12]. Once the volume is known, density can be calculated using the mass of the sample (which will be measured after the experiment).

Surface tension and viscosity can be measured using oscillating drop method [13]. In this method, a sample is molten and brought to a selected temperature. Then, a $P_2(\cos\theta)$-mode of

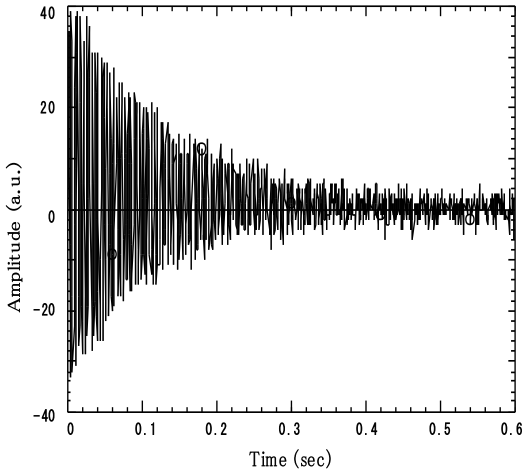

Fig. 6 Fluctuation on vertical diameter of levitated droplet measured by power meter.

drop oscillation is induced to the sample by superimposing a small sinusoidal electric field on the levitation field. Here, $P_2(\cos\theta)$ is a Legendre polynomial of 2nd order. An oscillation detection system measures the fluctuation of the vertical diameter of the molten sample with a 5000 Hz sampling frequency. The transient signal that followed the termination of the excitation field is shown in **Fig. 6**. This signal was analyzed to obtain ω_c and τ. The surface tension could be found from Eq. (1)

$$\omega^2 = \frac{8\gamma}{\rho r^3}. \tag{1}$$

Similarly, using the decay time τ given by the same signal, the viscosity η is found by

$$\eta = \frac{\rho r^2}{5\tau}. \tag{2}$$

5. Measurement of Molten Oxides in the ISS-ELF

Since oxide materials are hard to be electrically charged, electrostatic levitations of these samples on the ground are very hard, even though a few samples could be levitated and melted in 1-G condition [14–17]. Therefore, thermophysical property measurement of oxide melts is set as a main research target of ISS-ELF. Measurement of density, surface tension, and viscosity are planned on the simple oxides materials (metal element plus oxygen system) listed in **Table 2**.

Before conducting thermophysical property measurement of above mentioned samples, the measurement system has to be evaluated with the sample whose thermophysical properties are known. Currently, zirconium and erbium doped $CaAl_2O_4$ are selected for this evaluation purpose and will be processed in the

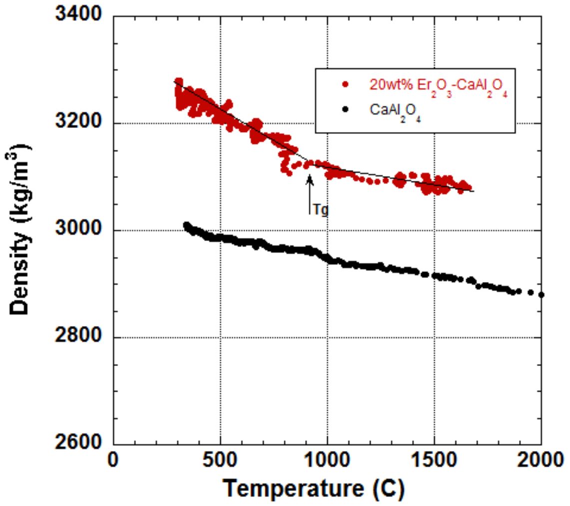

Fig. 7 Density of 20wt% Er_2O_3-$CaAl_2O_4$ (red) and non-doped $CaAl_2O_4$ (black) vs temperature.

ISS-ELF. The $CaAl_2O_4$ is a unique sample which can be levitated even in 1-G [18]. A small amount of erbium is doped to improve the absorption of heating lasers. **Fig. 7** shows density of 20 wt% Er_2O_3-$CaAl_2O_4$ and non-doped $CaAl_2O_4$ [18] measured on the ground ELF. No literature value was found for the density of 20 wt% Er_2O_3-$CaAl_2O_4$. Due to the addition of heavy erbium atoms, the density of 20 wt% Er_2O_3-$CaAl_2O_4$ is around 6% larger than that of $CaAl_2O_4$. The glass transition temperature (Tg) of 20 wt% Er_2O_3-$CaAl_2O_4$ was determined by the temperature coefficient of density to be around 900 degree C, which showed a good agreement with that of non-doped $CaAl_2O_4$ [18]. Density data of 20 wt% Er_2O_3-$CaAl_2O_4$ will be obtained in the ISS-ELF and evaluated by comparing this reference data.

Table 2 Oxide samples to be processed in the ISS-ELF.

No.	Sample	Melting Temperature (K)
1	Fe_2O_3	1838
2	Er_2O_3	2773
3	Sm_2O_3	2593
4	Eu_2O_3	2588
5	Tb_2O_3	2665
6	Ho_2O_3	2668
7	Al_2O_3	2319
8	Gd_2O_3	2668
9	Y_2O_3	2649
10	ZrO_2	2963
11	Lu_2O_3	2740
12	HfO_2	3063
13	Tm_2O_3	2665
14	Yb_2O_3	2693

6. Conclusions

Some features of the ISS-ELF and thermophysical property measurement plan are briefly described. The ISS-ELF will be operational onboard the ISS in this winter.

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