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ORIGINAL ARTICLE

Status of the Electrostatic Levitation Furnace (ELF) in the ISS-KIBO

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Abstract

The electrostatic levitation method is a containerless processing technique that utilizes Coulomb force between a charged sample and the surrounding electrodes. The Japan Aerospace Exploration Agency (JAXA) has been developing this technique for more than 20 years. In 2016, JAXA completed the flight model assembly, and the Electrostatic Levitation Furnace (ELF) for the International Space Station (ISS) was launched to the ISS. The ELF is mainly intended to handle oxide melts that are difficult to levitate on the ground based electrostatic levitator due to gravity and due to insufficient charging. ISS-ELF can measure the thermophysical properties (density, surface tension and viscosity) of high temperature melts above 2000 °C. The thermophysical properties data of materials at high temperature is useful for the study of liquid states and improvement of numerical simulation by modeling the manufacturing processes using the liquid state. Moreover, the interfacial energy of immiscible melts will be measured by creating a core-shell droplet configuration which otherwise cannot be obtained on the ground due to sedimentation. This paper briefly describes the ELF facility and presents the results of a functional checkout that includes the density measurement of molten alumina.

Keywords Containerless processing · Thermophysical property · High temperature melt · Microgravity experiment · Electrostatic levitation

Introduction

Whereas on ground a strong force must be applied to levitate a sample against gravity, in microgravity, samples can be easily handled without containers. This containerless processing has many technological and scientific advantages in materials science (Herlach 1994).

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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 with container walls. The lack of a crucible also suppresses nucleation induced by the walls of a container (heterogeneous nucleation) thus increasing the possibility of producing new functional materials such as glasses or to access metastable states of matter.

Even in microgravity, some positioning forces are necessary to stably levitate a sample against such disturbances as residual gravity and g-jitters. Therefore, space agencies have been developing position control methods (levitation methods) for a long time. Several levitation methods, including acoustic (Trinh et al. 1982), electromagnetic (Saito et al. 1969), aerodynamic (Babin et al. 1995), and electrostatic (Rhim et al. 1993) methods have been applied for containerless processing in space as well as on the ground.

The acoustic method has mostly been used to handle liquid samples at around room temperature. Research on drop dynamics was for instance conducted using the Space Shuttle. Drop oscillation experiments were conducted to validate the theoretical predictions on the oscillation frequency shift versus amplitude (Wang et al. 1994). Drop rotation experiments were also performed to investigate the bifurcation phenomena due to sample rotation (Wang et al. 1996). Acoustic levitation was recently used to determine the secondary Bjerknes force between rigid particles (Castro and Hoyos 2016).

An electromagnetic levitator developed by DLR (Deutsches Zentrum für Luft- und Raumfahrt) was also used in the Space Shuttle missions, in which several thermophysical properties of molten metals/alloys such as density, heat capacity, surface tension, viscosity, and electrical conductivity, were successfully measured (Egry et al. 1998; Egry 2000). An Electromagnetic Levitator (EML) is currently operational in the Columbus module aboard the International Space Station (ISS) (Seidel et al. 2011; Luo et al. 2016).

The Japan Aerospace Exploration Agency (JAXA) has been designing and developing a microgravity electrostatic levitation furnace since 1993. Through a sounding rocket experiment conducted in 1997 (Yoda et al. 2000; Yu et al. 2001) and following ground-based research (Paradis et al. 2002), several key technologies necessary for stable sample positioning and scientific observation have been developed. As a result, an electrostatic levitation furnace for the ISS (ISS-ELF) has been fabricated (Fuse et al. 2013; Ishikawa et al. 2014; Tamaru et al. 2015) and brought to the ISS. The main targets of the ISS-ELF are molten oxides, which are very difficult to levitate and melt with the ground based electrostatic levitator. Moreover, interfacial energy of immiscible melts will be measured by creating a core-shell droplet configuration which cannot be obtained on the ground due to sedimentation. The first trial of this idea was conducted on immiscible Co-Cu alloys using the EML in TEXUS 44 sounding rocket (Egry et al. 2010). Similar experiments on immiscible iron and slag systems using the ISS-ELF have been planned (Watanabe et al. 2016). This paper describes some features of the ISS-ELF and presents the results of a functional checkout that includes the density measurement of molten alumina.

Electrostatic Levitation Furnace (ELF)

Overview

The electrostatic levitation method uses Coulomb force between a charged sample and the surrounding electrodes to stably control the sample position. As this method cannot create a potential minimum to confine the sample, a high-speed real-time feedback control system is needed to stabilize the sample position. After fundamental techniques were developed by Rhim et al. (1993), the electrostatic levitation methods have been utilized in many research efforts. The scientific output achieved by electrostatic levitation are summarized in a couple of reviews (Hyers and Rogers 2008; Paradis et al. 2014). These assets are widely implemented in the development of the ISS-ELF.

Figure 1 shows an overview of the ISS-ELF. It weighs about 220 kg and is installed in the Multi- Purpose Small Payload Rack 2 (MSPR-2) as illustrated in Fig. 2. While the ISS-ELF main body is inserted in the Work Volume (WV) of the MSPR-2, the UV lamp unit is mounted in the Small Experiment Area (SEA). Since the MSPR-2 is shared with a wide variety of experiment facilities, the ISS-ELF must be easily installed and removed from the rack. Electric power, avionics air, cooling water, and communication signals are supplied to the ISS-ELF through the MSPR-2.

The main body of the ISS-ELF consists of three parts as shown in Fig. 3. The right part contains a computer called the "experiment controller" and the left part contains another one named the "position controller". The left part also contains the high voltage amplifiers and gas valve assembly. The center part is occupied by the processing chamber and optical devices that include the heating lasers, a pyrometer, and observation cameras.

All the optical devices are mounted on the chamber as shown in Fig. 4. The chamber is a polyhedron with 26 faces. It can be operated in the range of 500 Pa to 2×10^5 Pa by either evacuating or pressurizing the chamber. The ISS-ELF has capabilities to measure the density (Chung et al. 1996), surface tension, and viscosity (Rhim et al. 1999) of molten samples. The optical devices are described in detail later.

Sample Cartridge and Sample Holder

Details of the sample cartridge are shown in Fig. 5. The sample cartridge can accommodate a sample holder, which contains 15 samples. the size of the spherical sample is

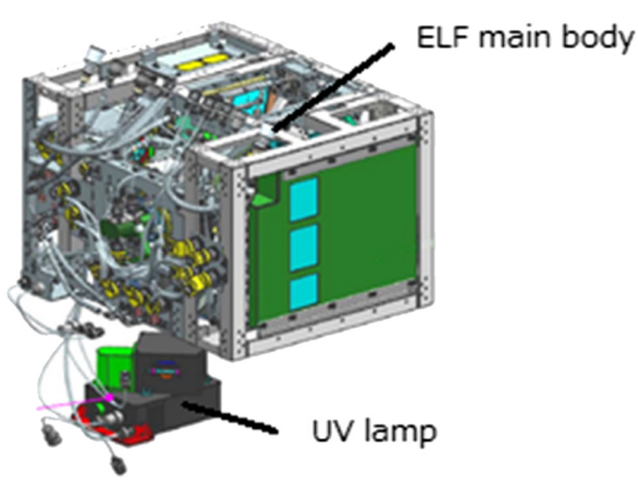

Fig. 1 Overview drawing of the ISS-ELF. The ELF main body and the UV lamp are indicated

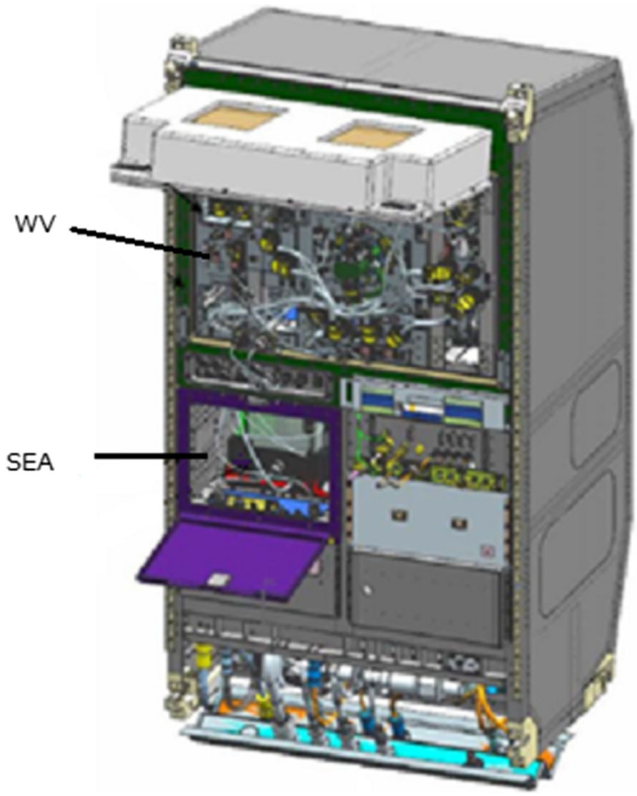

Fig. 2 Drawing of the ISS-ELF installed in the MSPR-2. The main part of the ISS-ELF is installed in the work volume (WV), while the UV lamp is set in the small experiment area (SEA)

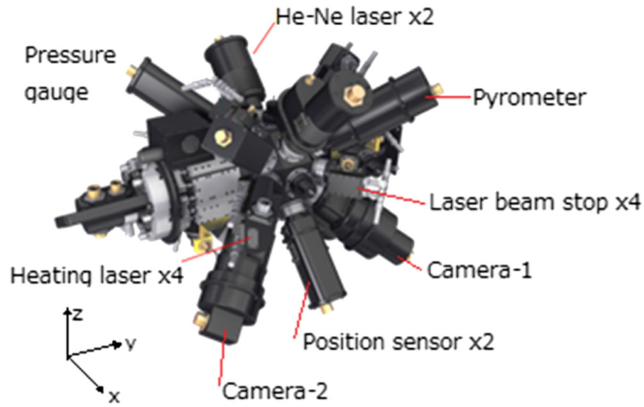

Fig. 4 Drawing of the ISS-ELF chamber and installed optical devices. Coordinates of the ISS-ELF (x, y, and z directions) are indicated

approximately 2 mm in diameter. The sample cartridge contains six electrodes between which a sample is levitated. Since there is no strong G-vector in microgravity, the size of these six electrodes is identical. The separation between the electrodes is 15 mm along the y-axis and 30 mm along both the x and z-axis. The sample cartridge has two pushing rods. At the insertion of the sample, the upper rod pushes out the sample from the sample holder and carries it to levitation filed. After the experiment, the processed sample is carried back to the sample holder by the lower rod. By rotating the sample holder, samples are exchanged from the processed sample to an unprocessed one. These operations are conducted from ground through tele-commands, thereby enabling to carry out experiments on all 15 samples continuously and without the requirement of crew operation. The sample cartridge is inserted into the chamber by crew members, as shown in Fig. 3.

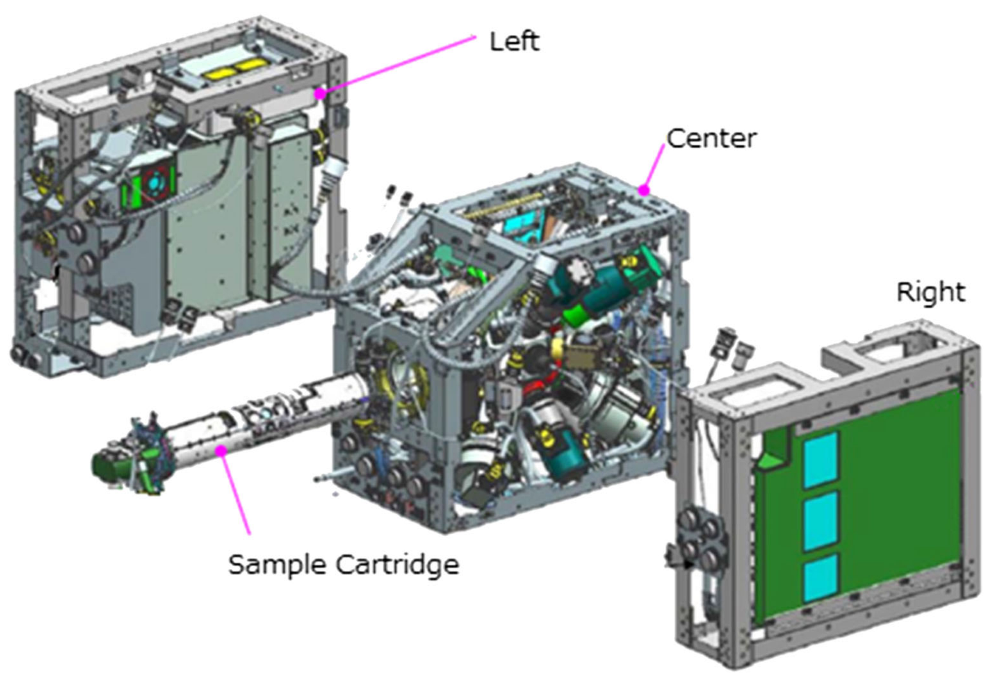

Fig. 3 ISS-ELF in its shipping and launch configuration. The main part of the ISS-ELF installed in the WV is divided into three parts. The sample cartridge is also separately shipped

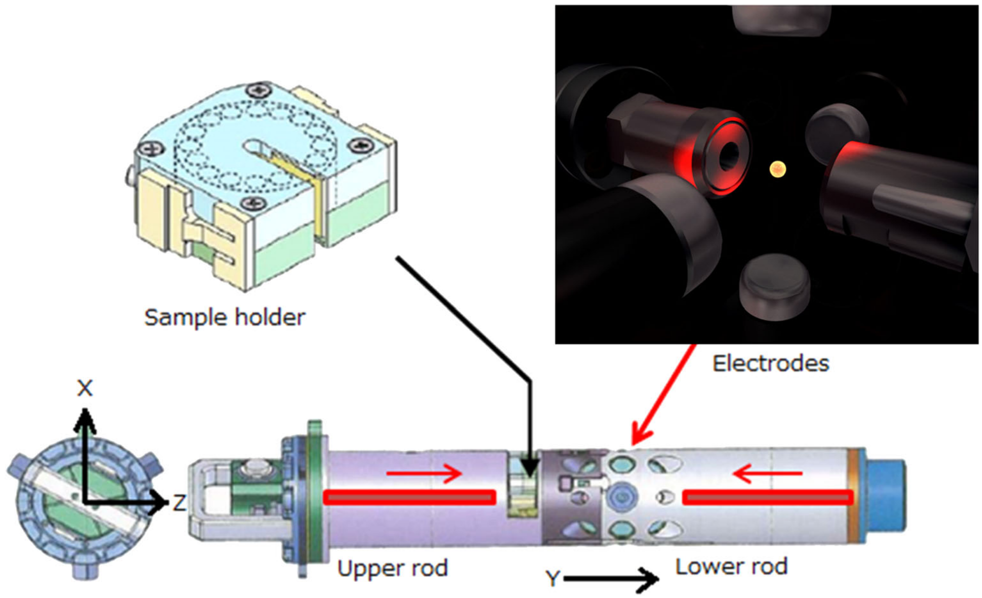

Fig. 5 Conceptual drawing of the sample cartridge. A sample holder (left top) is inserted in the cartridge and upper rod transfer a sample to the levitation field (right top). The lower rod is used to return the sample back to the holder. Coordinates of the ISS-ELF (x, y, and z directions) are indicated

Experiment and Position Control

The overall experiment sequence is controlled by the experiment controller. It activates and deactivates all other devices. It opens and closes gas valves to maintain atmospheric conditions in the chamber. It also measures the temperatures of all devices for safety. Finally, it records experiment and house-keeping data, and transfers these data to the ground.

The position controller is dedicated to the position control of the levitated samples. It also moves the rods and rotates the sample holder in the sample cartridge for sample insertion and retrieval.

The sample position control method is the same as the original technique described by Rhim et al. (1993). A collimated laser beam (around 20 mm in diameter) from a projection laser (He-Ne laser with 638 nm wavelength) projects a shadow of the sample on a position sensor (HAMAMATSU Intelligent Vision System) where vertical and horizontal positions are measured. Two sets of the projection laser and position sensor system are orthogonally placed to measure the tridimensional sample position (x, y, and z). The position signals are sent to the position controller where the sample position data are compared with the setting position (x_0, y_0, and z_0). The position control voltages (V_x, V_y, and V_z) are then calculated using the following PD (Proportional- Differential) control algorithm:

$$\begin{aligned} V_x &= P_x(x - x_0) + D_x(x - x_0)' \\ V_x &= P_y(y - y_0) + D_y(y - y_0)' \\ V_z &= P_z(z - z_0) + D_z(z - z_0)' \end{aligned} \quad (1)$$

where P and D indicate the proportional and differential control parameters respectively. This feedback control is conducted with a frequency of 1000 Hz for stable levitation of a sample. Control parameters can be adjusted from the ground.

Heating Lasers

The sample is heated by four semiconductor lasers (980 nm, 40 W power each). In order to obtain an excellent temperature homogeneity of the sample, these lasers are arranged in a tetrahedral heating arrangement around the sample (Schroers et al. 2004). Each laser power can be controlled by commands from the ground thorough the experiment controller. Laser beam-stops are installed to absorb laser beams and protect the chamber wall. Laser beams are focused (around 0.4 mm in diameter) at the sample position.

Optical Devices

Sample temperature can be measured by a commercial pyrometer (IMPAC IGA140) thorough a couple of sapphire windows. The pyrometer measures the radiation intensity (1.45–1.8 μm in wavelength) from the sample. Since the emissivity setting on the pyrometer can't be changed from the ground commanding, it remains 1.0. The measurement temperature range is from 300 °C to 3,000 °C, with a 100 Hz frequency interval. Actual sample temperature can be determined using the temperature plateau after recalescence and known melting temperature of the sample. The pyrometer contains a built-in video camera with which it can be easily confirmed that the sample stays in the measuring spot of the pyrometer (Fig. 6a).

The ELF has two other cameras as shown in Fig. 4. The camera-1 (color) offers a wide view of the levitation field, including the electrodes used to observe sample behavior from insertion to retrieval (Fig. 6b). The camera-2 (a black and white camera with a non-telecentric zoom lens) gives a magnified sample image with UV backlight (Fig. 6c). Using the video image (taken with a 60 Hz frequency interval) from this camera, density of the sample can be calculated (Chung et al. 1996).

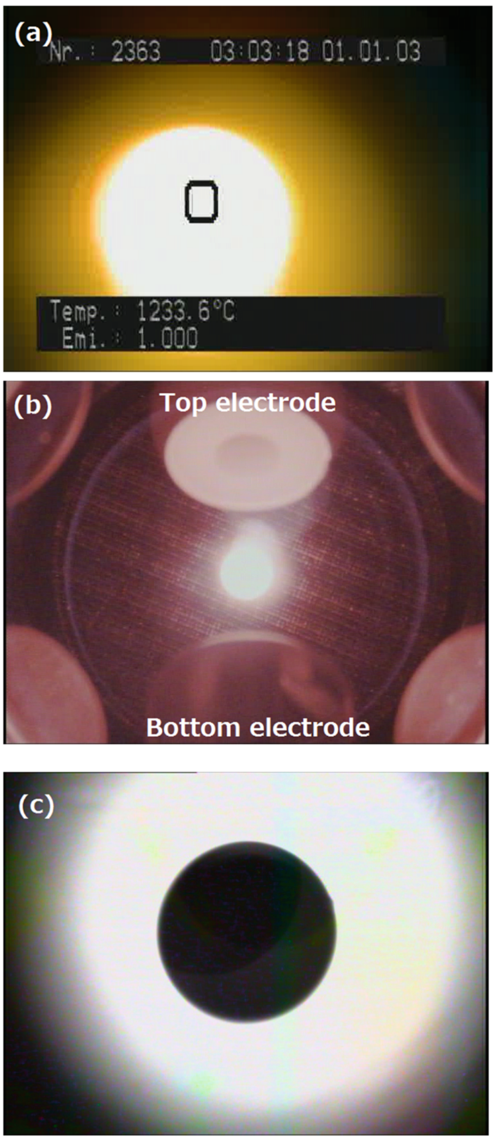

Fig. 6 Levitated sample in the ISS-ELF; **a** image taken by a camera through the pyrometer, **b** image taken by camera-1, **c** magnified sample image by camera-2 with UV back light for density measurement. A black square in Fig. 6a is an area where the temperature is measured

Surface tension and viscosity of the sample can be measured using the oscillation drop method. The measurement technique is same as the one described in a reference (Rhim et al. 1999). The drop oscillation of the molten sample is excited by superimposing sinusoidal voltage on the electric field for a couple of seconds. After the termination of excitation, the oscillation amplitude on the sample is gradually decays by its viscosity. A collimated laser beam, which creates a drop shadow for position sensing, is divided and part of the beam is introduced to a circular photo-detector. The oscillating drop amplitude can be measured as a fluctuation of the total leaser power at the photo-detector, with a 5000 Hz frequency interval. From the measured signal, the characteristic oscillation frequency of the sample and time constant of amplitude decay are obtained, from which surface tension (Lord Rayleigh 1879) and viscosity (Lamb 1932) can be calculated. The viscosity can be measured over the 0.5 to 100 mPa·s range.

Experimental Procedure

After the sample cartridge is inserted by the crew, all experiment operations are conducted from the ground. The upper rod transfers the sample from the sample holder to the levitation area through the top electrode. The sample gets a positive charge when it hits the bottom electrode. After the sample position is stabilized by position control, the sample is melted by heating lasers. An UV lamp is used to maintain the sample's charge using a photoelectric effect. After the thermophysical property measurements are conducted, the sample is solidified by turning off the heating lasers. The processed sample is retrieved by the lower rod. After all the samples in the holder are processed, the crew replaces the sample holder with a new one.

On-Orbit Results

The ISS-ELF was launched dismantled by two flights (HTV-5 and OA-4). Then, it was assembled in the ISS and installed in the MSPR-2 in February 2016. Since then, a functional checkout and initial experiments using metal and oxide samples has been conducted. The samples for initial checkout (listed in Table 1) have thus far been successfully levitated. Oxide samples including Al_2O_3 and 40wt%Er_2O_3-$CaAl_2O_4$ were melted under dry air, while zirconium samples were melted in an Ar (with 99.9999% purity) gaseous environment. Since the verification of the surface tension and viscosity measurements have not been finished yet, the results on sample position control

Table 1 Estimated surface charge of the samples levitated in the ISS-ELF

Sample	Mass (mg)	Charge ($\times 10^{-12}$C)
SUS	32.2	10–50
Zr	20–28	6–10
Al_2O_3	15–17	2–20
40 wt%Er_2O_3-$CaAl_2O_4$	10–11	10

(at room temperature and high temperature) and density measurements are explained here.

Levitation of Solid Samples

Figure 7 shows a time-position (y-direction; see Fig. 5) history of a stainless-steel sphere during sample insertion. Sample position data were recorded at intervals of 0.1 s. The amount of the surface charge on the sample can be estimated using this sample position profile.

Sample motion in the y-direction (y) is governed by the following simple equation:

$$my'' = \frac{QV_y}{L_y} \tag{2}$$

where m is the sample mass, V_y is the control voltage between the top and bottom electrodes determined by Eq. 1, L_y is the distance between the top and bottom electrodes, and Q is the charge of the sample.

Numerical simulations are conducted by changing Q so that the simulation agrees with the measured sample motion. The best simulation result is plotted in Fig. 7. The sample charge is estimated to be 1.65×10^{-11} C. Table 1 lists the amount of surface charge on the initial checkout samples. The values range from 10^{-12} to 10^{-11} C. In ground experiments, a surface charge around 10^{-10} C is necessary for levitation against gravity. In microgravity, the amount of a sample's charge can be reduced to around 1% of 10^{-10} C.

Sample position stability was analyzed using image analysis. Still images of stainless-steel sphere such that shown in Fig. 6c were captured from a video recorded for 30 seconds (900 frames). Each image was analyzed, and the centroid of the sample was determined. Sample movement in 30 seconds was found to be less than 40 μm.

Levitation of High Temperature Melts and Density Measurement

Alumina (Al_2O_3) samples were used to check the capabilities of heating and density measurement. Each sample was levitated in dry air (2×10^5 Pa in pressure) and heated by four semiconductor lasers. When the sample was fully molten, all lasers were powered off to cool the sample. Figure 8 shows a typical temperature - time profile measured by the pyrometer. The position stability became worse (up to 400 μm) than that at room temperature (less than 40 μm) due to disturbances induced by the heating lasers. However, by tuning the control parameters, the position

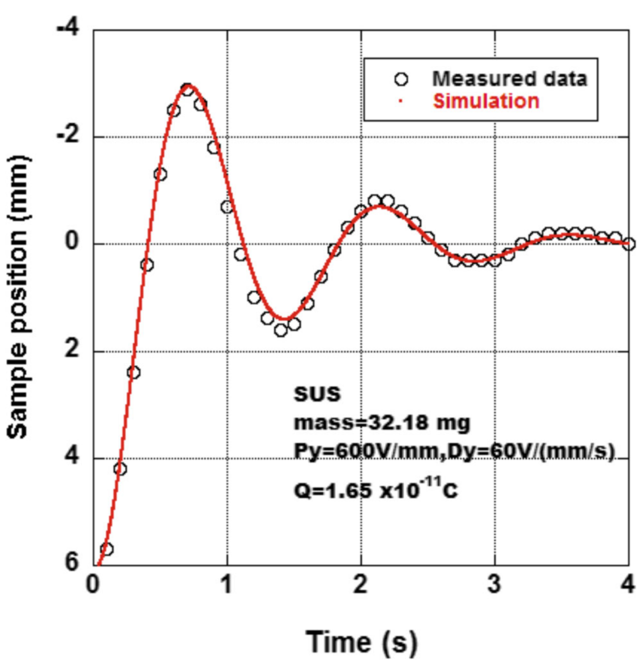

Fig. 7 Movement of sample during sample insertion. Measured data was recorded with 0.1 s interval. Numerical simulation results are shown in red line

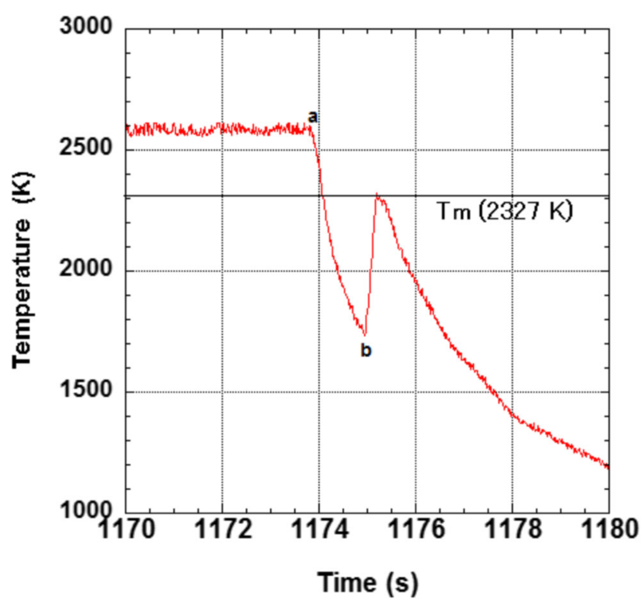

Fig. 8 Temperature profile of molten alumina sample during rapid cooling. Heating lasers were turned off at point a and recalescence occurred at point b

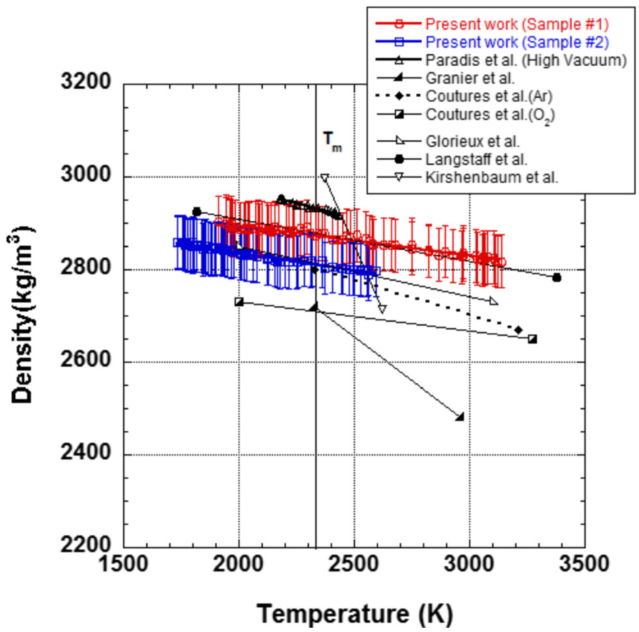

Fig. 9 Measured density of molten alumina as a function of temperature (Density data of sample-1 and sample-2 are plotted with 2% error bar. Some literature values are also plotted)

fluctuations were minimized to less than 100 μm so that the sample temperature could be properly measured.

The sample moved mainly in the x-z plane with a circular motion whose frequency was less than 1 Hz. No surface oscillation was observed due to this fluctuation.

Heating lasers also create torque and rotate the sample (Rhim et al. 1999). Sample rotation creates centrifugal force in the sample and reduces the advantages of microgravity environment (Paradis et al. 2003). Even though no rotation control device is implemented with the ISS-ELF, noticeable sample rotation induced by the heating laser has not been observed. Since the experiments were conducted in the pressurized gaseous environment, aerodynamic drag between sample and surrounding gas was large enough to prevent sample rotation.

When the heating lasers were turned off, the sample was cooled by radiation and conduction through the surrounding gas. After the sample reached the undercooled temperature, it exhibited recalescense (a sudden temperature rise to its melting temperature) and solidified. After the experiment, the sample was successfully retrieved to the sample holder, which was later returned to Earth from the ISS.

Magnified sample images recorded during the time period from a to b in Fig. 8 were analyzed to obtain the volume as a function of temperature. The masses of the processed samples were measured on the ground. Finally, the density data of alumina as a function of temperature were obtained. Figure 9 shows the measured density of molten alumina as a function of temperature. Two alumina samples were used to validate density measurements. The first sample (sample-1) was heated above 3,000 K and quickly cooled down to ensure the sample stability at high temperature. The second sample (sample-2) was heated around 2,600 K and maintained as this temperature for

Table 2 Literature values of the density of liquid alumina

Density at T_m (10^3 kg·m^{-3})	Temperature coeff. (kg·m^{-3}·K^{-1})	Temperature (K)	Reference
2.87	−0.064	1913–3139	Present work (run-1)
2.81	−0.074	1732–2597	Present work (run-2)
2.9	−0.09	1900–3240	Langstaff et al. (2013)
2.93	−0.12	2175–2435	Paradis et al. (2004)
2.81	−0.107	2000–3100	Glorieux et al. (1999)
2.71	−0.0678	2000–3275	Coutures et al. (1994)
2.80	−0.151	2327–3210	Coutures et al. (1994)
2.72	−0.28	2323–2965	Granier and Heurtault (1983)
2.69	−0.79	2320–3100	Zubarev et al. (1969)
2.55		2327	Wartenberg et al. (1936)
3.06		2327	Ikemiya et al. (1993)
3.06	−0.965	2323–3023	Shpil'rain et al. (1973)
2.98	−1.15	2325–2775	Elyutin et al. (1973)
3.03	−0.752	2323–2673	Rasmussen (1972)
3.04	−1.15	2323–2828	Mitin and Nagabin (1970)
3.05	−1.127	2375–2625	Kirshenbaum and Cahill (1960)
3.05		2327	Kozakevitch (1960)
2.97		2327	Kingery (1959)

10 minutes to check the long term sample stability and influence of evaporation. The difference between sample-1 and sample-2 is about 2%, which is within the experimental uncertainties of our measurement system (Ishikawa et al. 2005).

Measured density data (sample-1) can be compared with our earlier results obtained using a ground-based electrostatic levitator (G-ESL) under a high vacuum condition (Paradis et al. 2004). All data are shown in Fig. 9. Data obtained with the ISS-ELF cover a temperature range wider than those with the G-ESL. This result is reasonable because 1) the G-ESL being operated under high vacuum, the molten alumina sample became unstable due to evaporation and limited the highest temperature; 2) the cooling speed in high vacuum is lower than that with gaseous environment, which resulted in lower undercooling. The density of sample-1 is around 2% lower than that of G-ESL. In microgravity, voids in the sample are hard to be removed if they exist, and they may make the density value lower. This might be a cause for the density difference between ISS-ELF and G-ESL as well as sample-1 and sample-2. The alumina samples returned to the ground will be analyzed to confirm the existence of voids. However, measured data for sample-1 show good agreement with those obtained by Langstaff et al. (2013) using an aerodynamic levitator, and other literature values listed in Table 2 which indicate that the influence of voids was very limited. As a result, the validity of the density measurement with the ISS-ELF has been confirmed.

Conclusions

The ISS-ELF has been developed, fabricated and installed in the ISS. It is currently operational, and some oxide materials have been levitated and molten in microgravity. Molten oxides could be heated as high as 3,000 K. Density measurement capability has been checked with alumina samples. The functional check for surface tension and viscosity measurement is currently being conducted, and detailed results will be reported later.

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