Thermophyical property measurement results of ultra-high melting temperature oxides using Electrostatic Levitation Furnace (ELF) onboard ISS-KIBO

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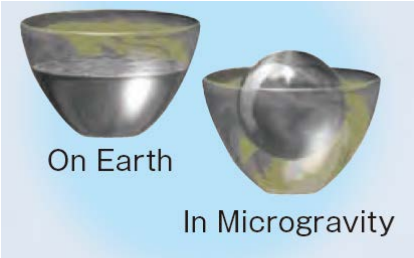
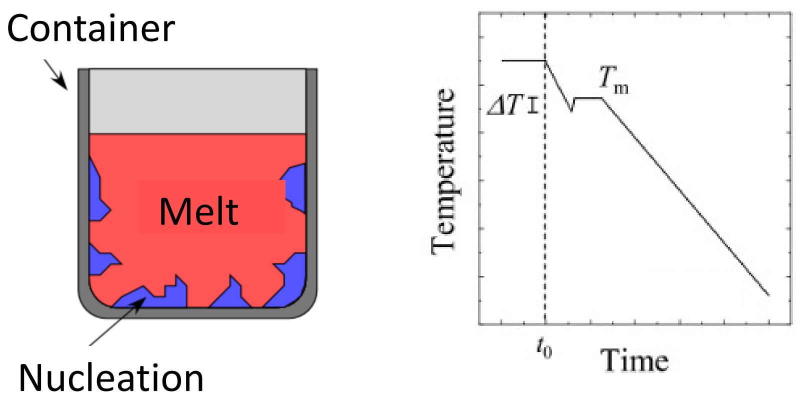
Levitation = Containerless technique

Its advantages :

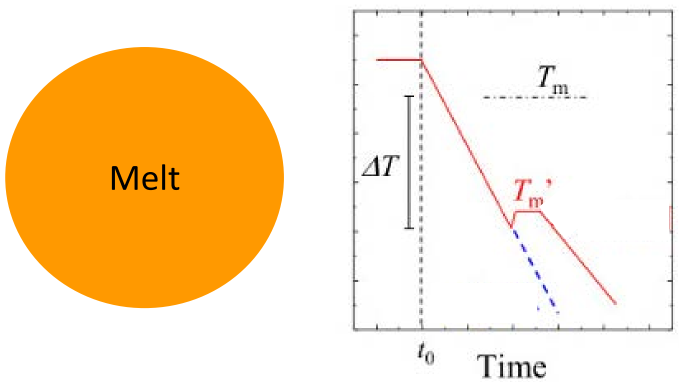
- High temperature liquid, with a melting temperature higher than 2,000 deg. C, can be handled without contamination. Handling without contamination enables accurate measurements of thermophysical properties.
- The lack of a crucible enables larger degree of undercooling and increases the possibility of producing new functional materials such as glasses or to access metastable states of matter.



Container



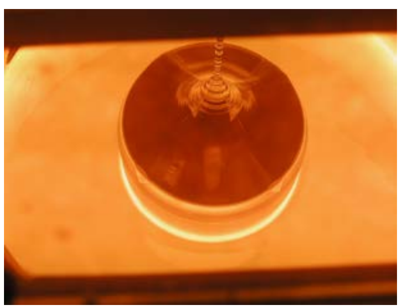
Containerless

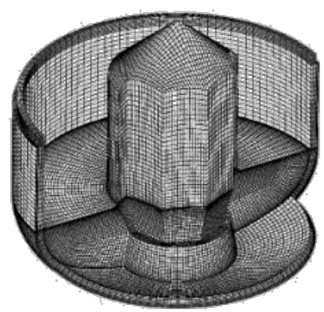


Molten density (ρ), surface tension (γ), viscosity (η)

- Industrial crystals or glasses are manufactured from high temperature melts.
- Accurate thermophysical properties of melts are required for the material processing and structural analysis of melts.

Material processing

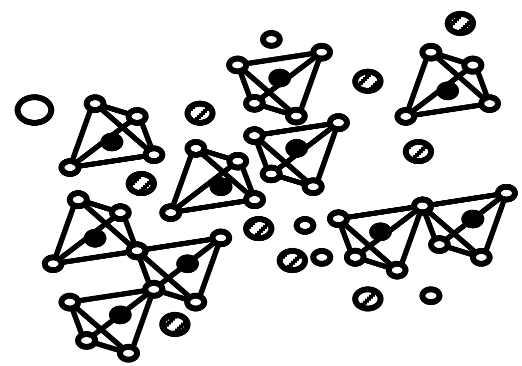




Casting, Wilding, Thermal spraying and so on…

V.V. Kalaev *et al.* J. Cryst Growth, **250** (2003) 203.

Structure of melt

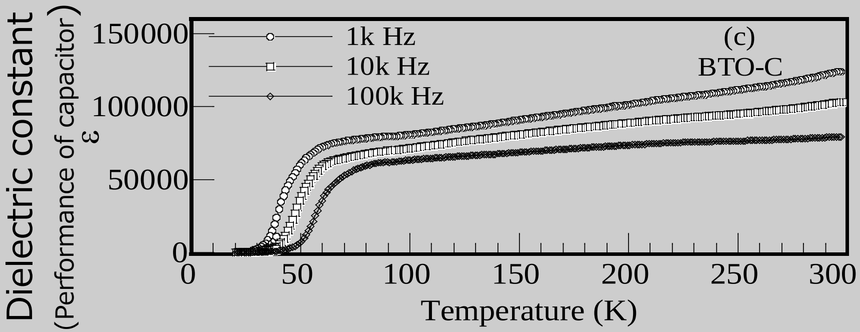


Takeda *et al*. Jap. Assoc. Cryst. Growth, **20** (1993) 27

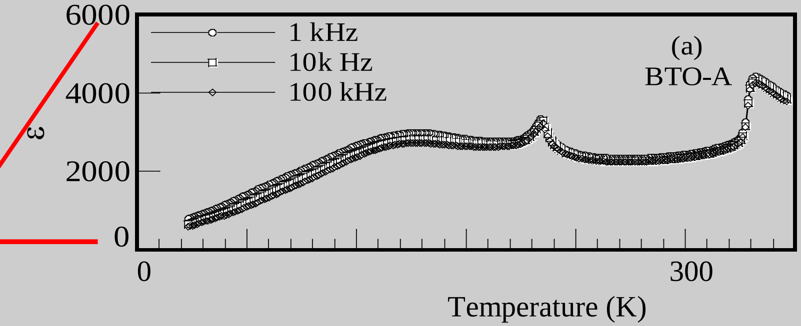
<u>Melting temperature (T_m) of industrial material</u>

Ex.) Si: 1687 K, Fe: 1811 K, SiO_2: 1923 K, YAG: 2213 K, Al_2O_3: 2345 K, Lanthanoid oxides : <u>over 2500 K</u>

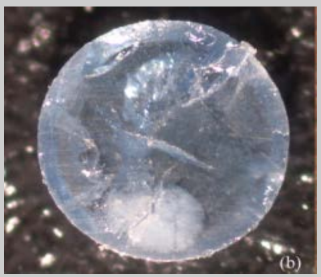
New functional materials processed by levitation technique



BaTiO$_3$ capacitor manufactured by ELF



BaTiO$_3$ capacitor commonly used



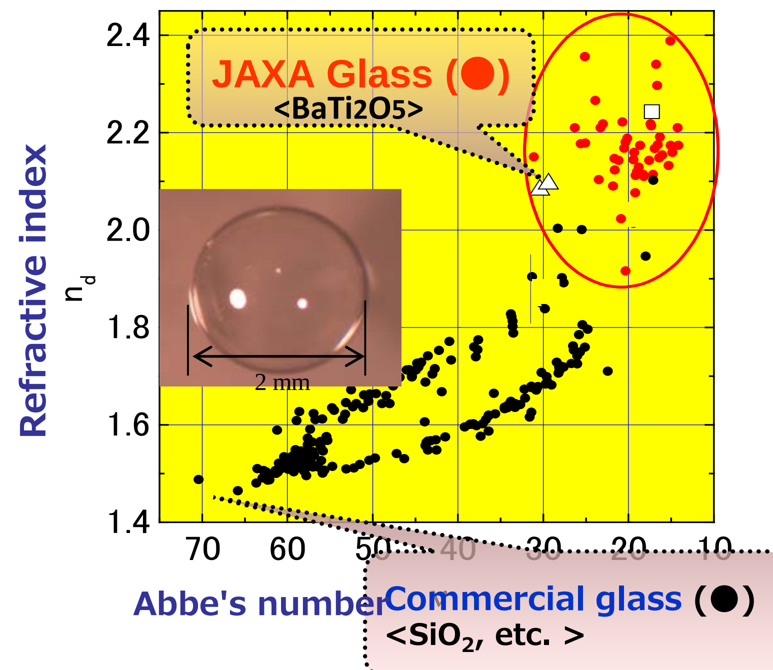
<u>The dielectric constant is **30 times more than conventional dielectrics**</u>
-> Ultra-compact capacitor
by TDK Corporation

Single crystal with hexagonal crystal system (high-temperature phase)

J. Yu, P.-F. Paradis, T. Ishikawa, and S. Yoda, "Huge Dielectric Constant of Transparent Hexagonal BaTiO3 Obtained by Containerless Processing" Ferroelectrics, 301(2004), 199–201.

<u>Creation of high refractive-index glass</u>
<u>(Highest refractive index of 2.4)</u>

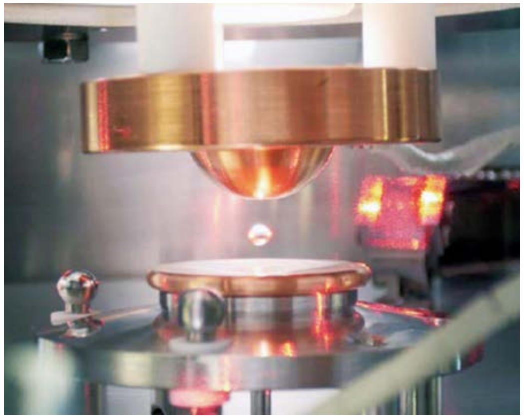
-> Ball lens for high-density DVD
by Nippon Sheet Glass Co., Ltd.



A. Masuno, J. Yu, J. Jpn Soc. Microgravity Appl. 26 (2009),134

Levitation techniques

Electrostatic levitation on the ground (in vacuum)

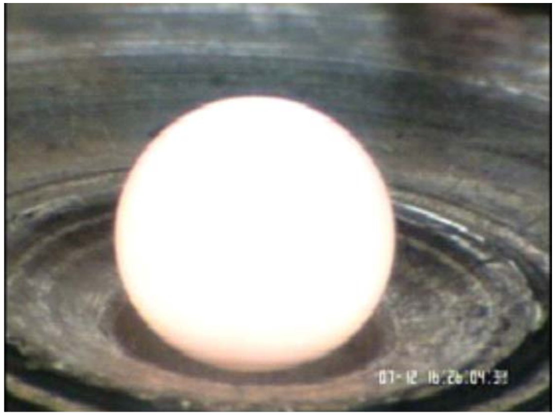


Easy evaporation ☹

Electromagnetic levitation

Only conductor ☹

Aerodynamic levitation



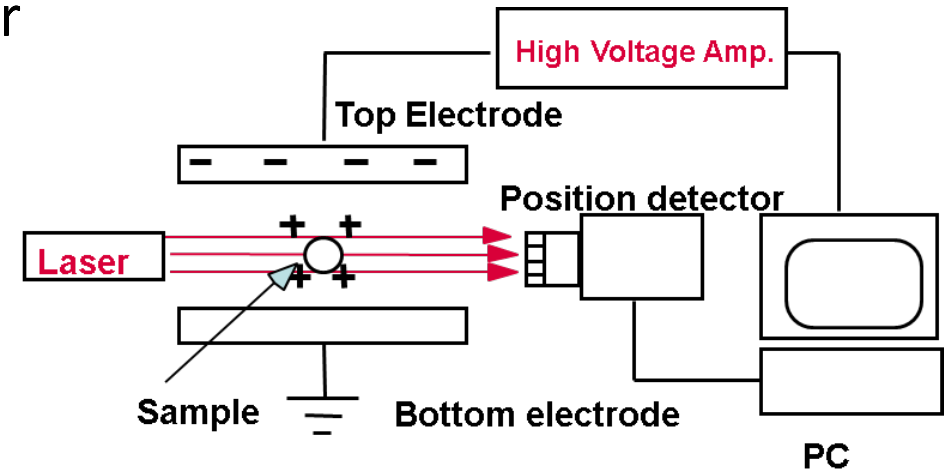
Can not observe whole image
Large temperature gradient ☹

Only the electrostatic levitation under the micro gravity environment can levitate and melt ultra-high temperature oxides.

☺

Electrostatic Levitation Method

- Developed by W.-K. Rhim @JPL.
- Use Coulomb force between charged sample and electrodes.
- Need High speed feed back for position control.
- Less disturbances



Rhim et al., *Rev. Sci. Instrum.*, 64 (1993),2961.

Type	Voltage (Electric discharge)	Atmosphere (Evaporation)	sample
On the ground [1]	High ($8kVcm^{-1}<$) (occur)	Vacuum (difficult to control)	Metals and few oxides
On ISS	low ($<3kVcm^{-1}$) (does not occur)	Pressurized gas or vacuum (easy to control)	Metal, alloy, and oxides

[1] Ishikawa et al., *J. Jpn Soc. Microgravity Appl.*, 18 (2001),106

Electrostatic levitation furnace in the International Space Station (ISS-ELF)

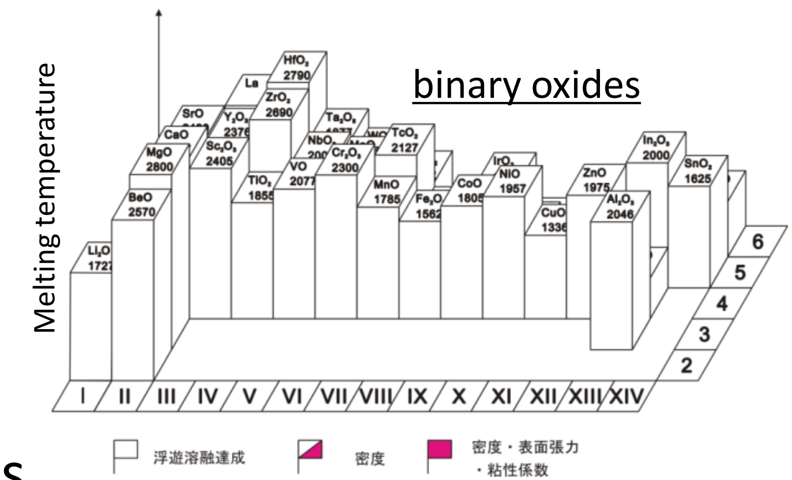
Mission Objectives of the ISS-ELF

1. Measure thermophysical properties of molten oxides whose T_m is over 2000 K.

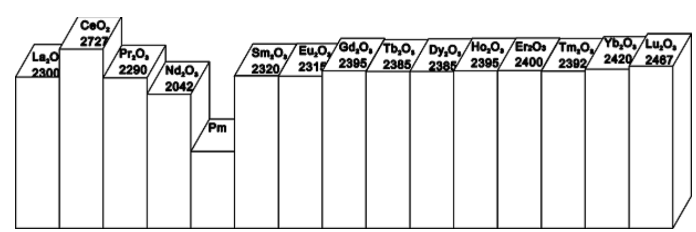
- The database of thermophysical properties will be open to the public.
- Alloy is also available.

2. Search for Novel materials

- samples return to Earth by solidification from undercooling.
- Analysis about solid state properties
- Consider the possibility of industrial applications



rare earth oxides

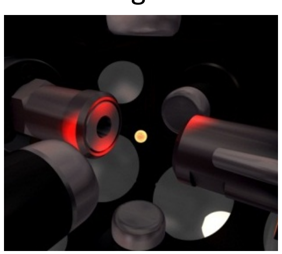


Overview of the ISS-ELF

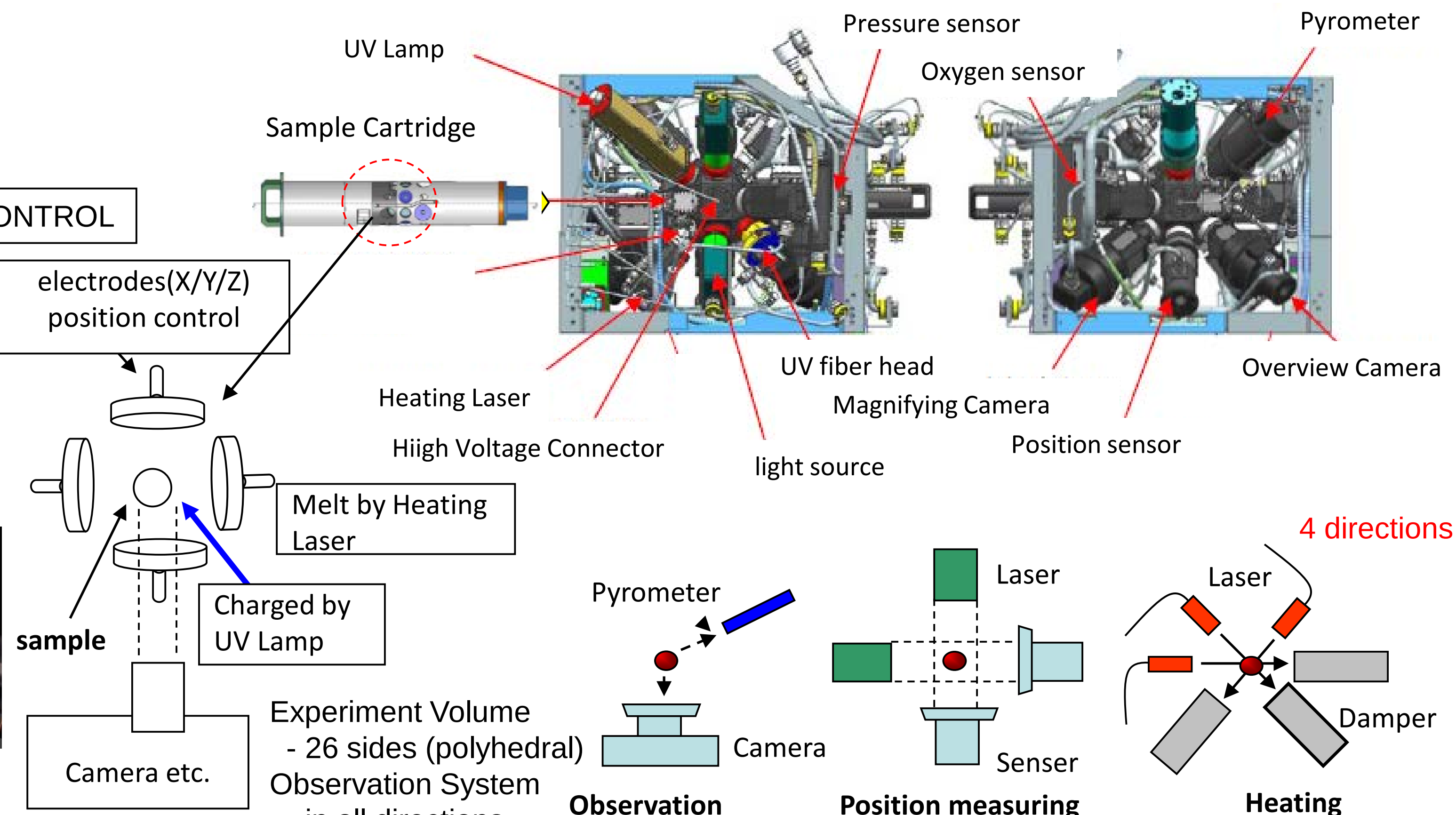
- Sample size : about 1.5-2.1mm in diameter
- Voltage between electrodes: Max. 3 kV
- Atmosphere : pressurized air, N2, Ar (2 atm.), Vacuum(approx. 300Pa)

THREE-AXIS CONTROL

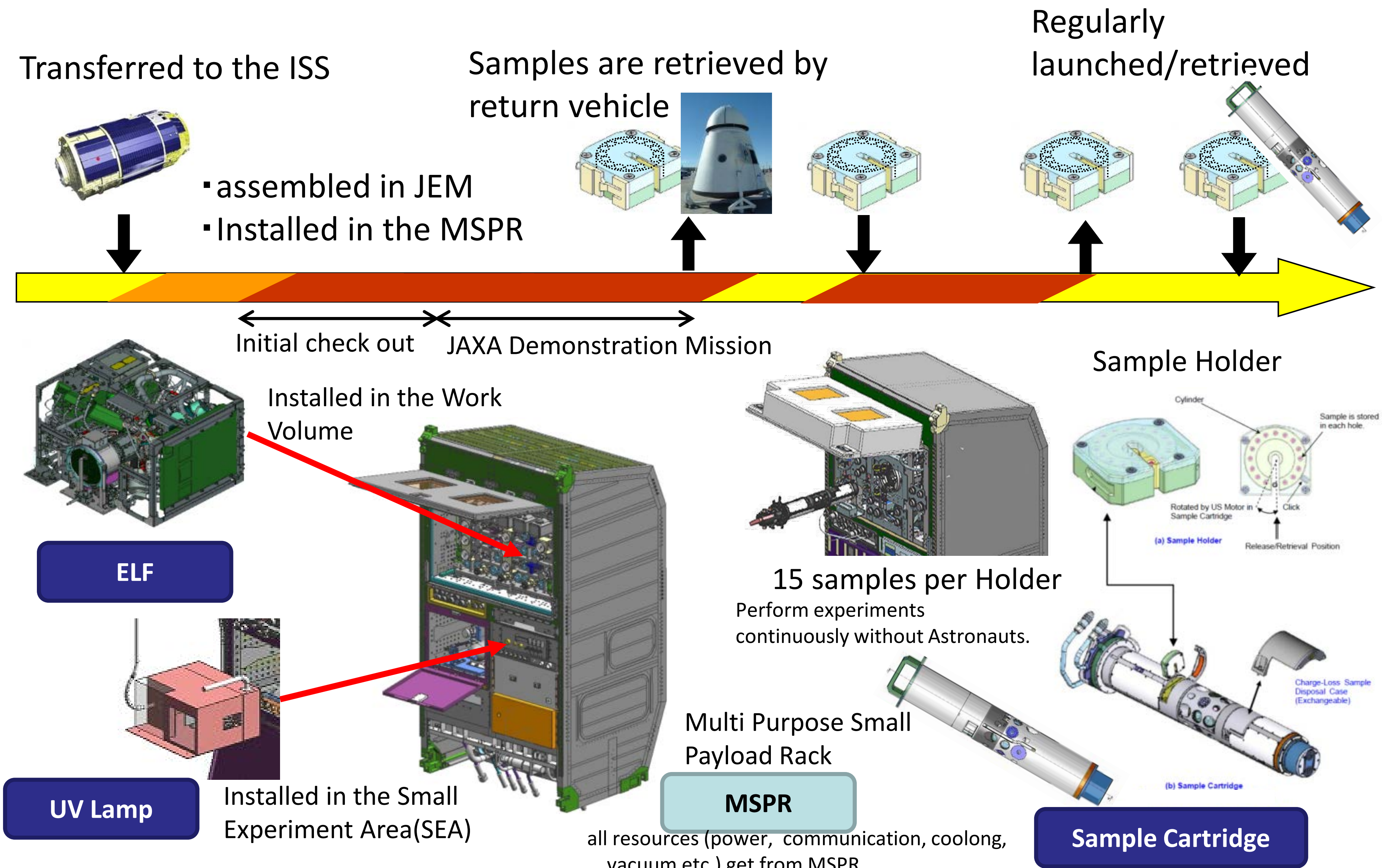
On the ground



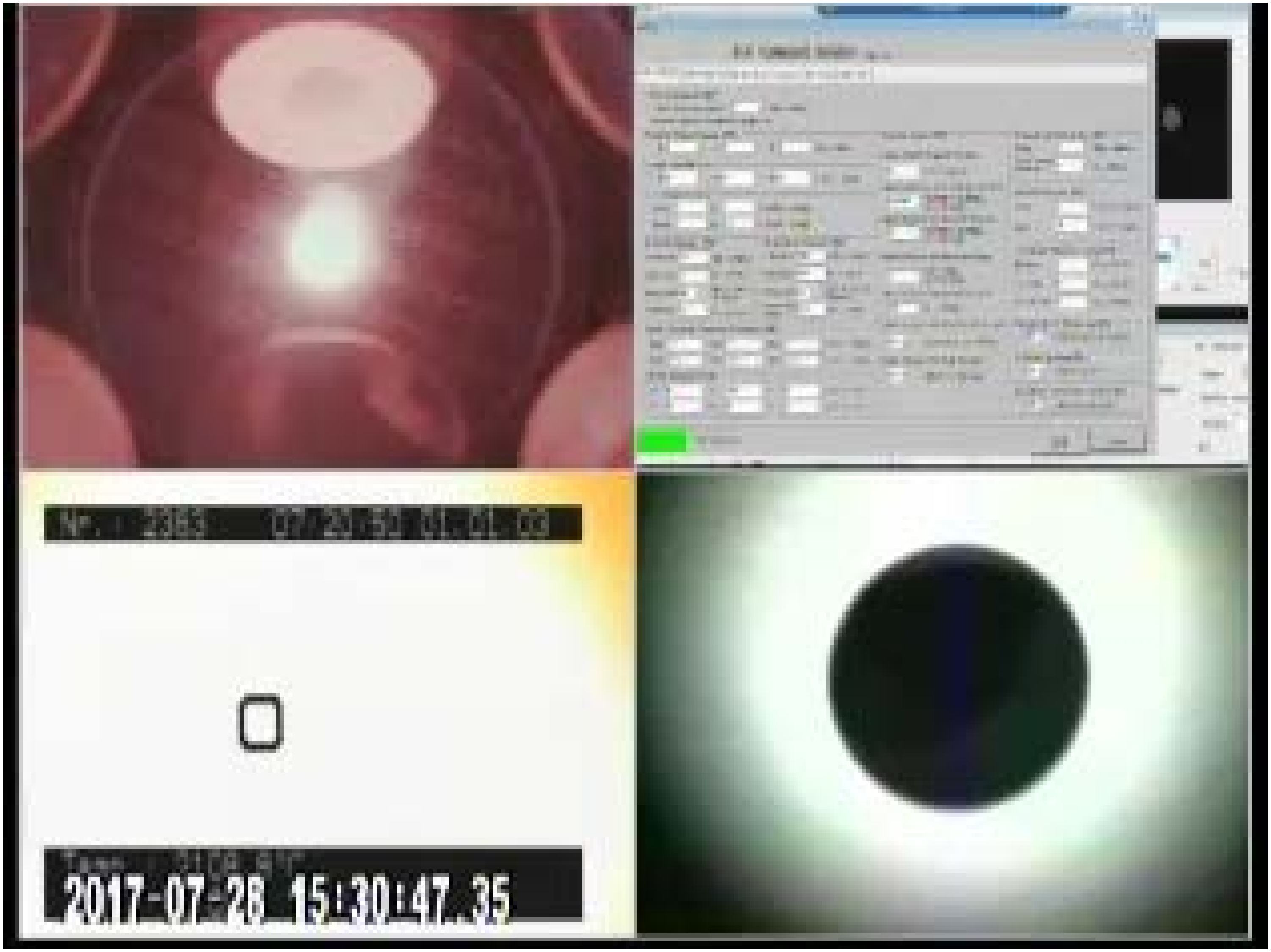
in Space



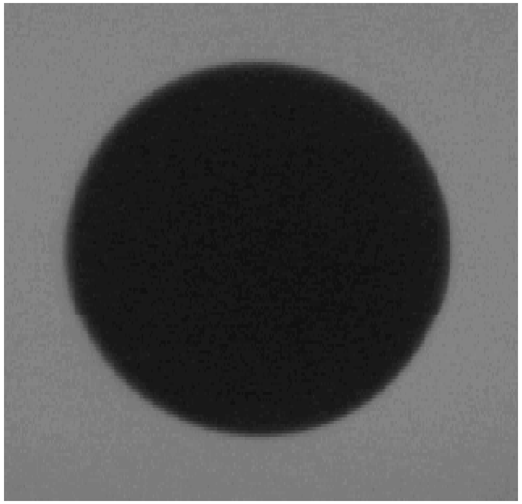
Operation cycle



Levitation and solidification of molten Al2O3

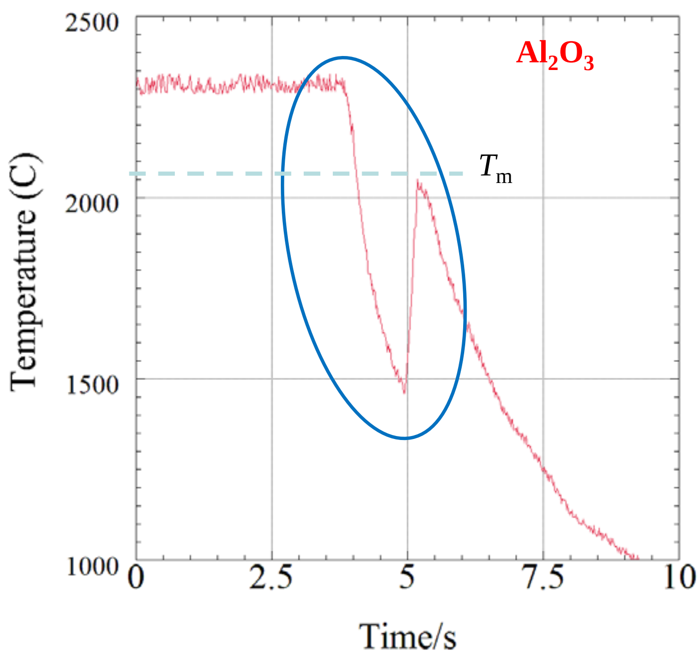


Density measurement



$$\rho = M / V$$

M: mass (measured before heating)
V: volume (from image analysis)



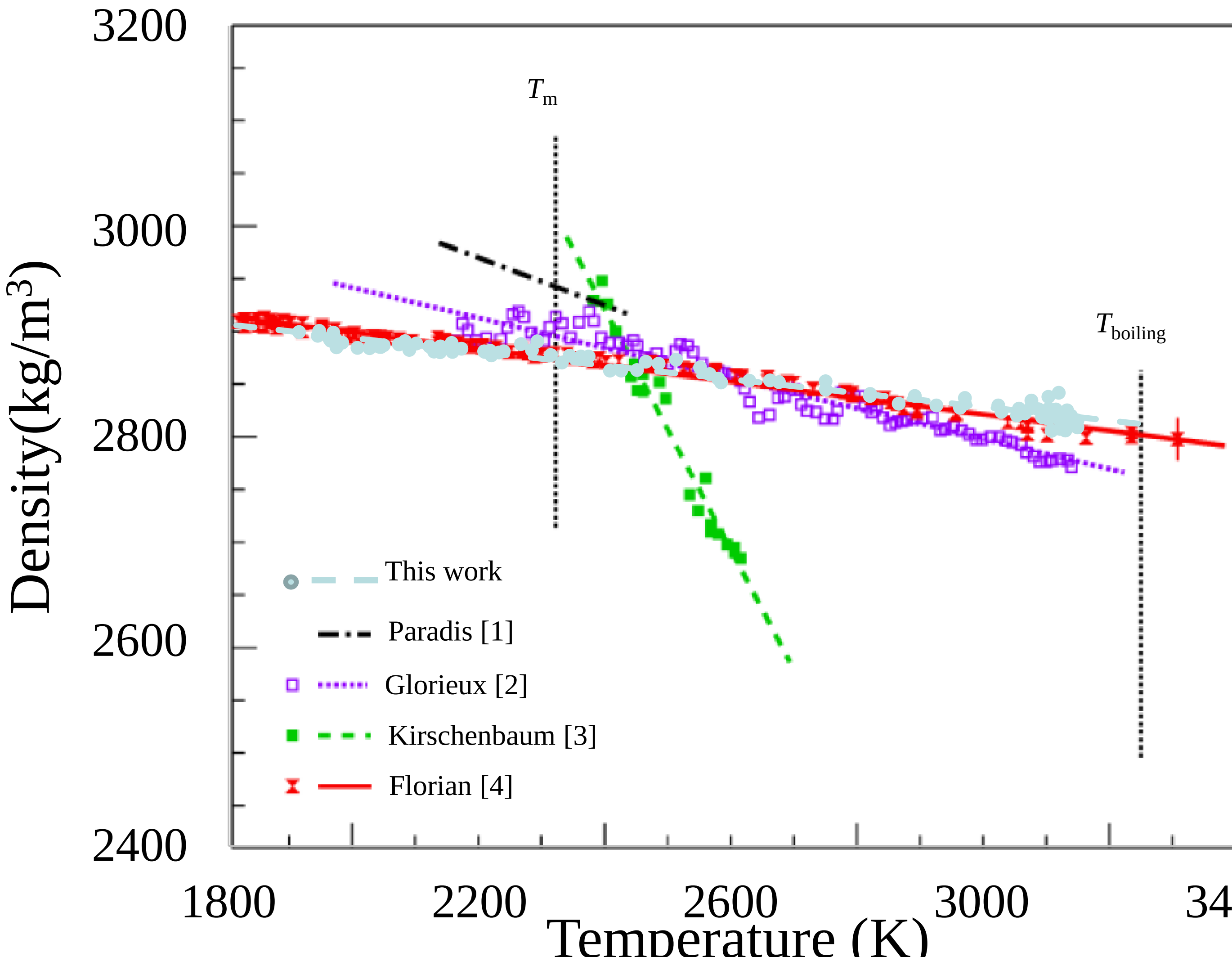
$$V = \frac{2\pi}{3}\int_0^{\pi} r(\theta)^3 \sin\theta d\theta$$

$$r(\theta) = \sum_{n=0}^{5} a_n P_n(\cos\theta)$$

P_n $\cos\theta$: Legendre polynomial

Chung et al., *Rev. Sci. Instrum.* 67 (1996), 3517

Density of molten Al_2O_3

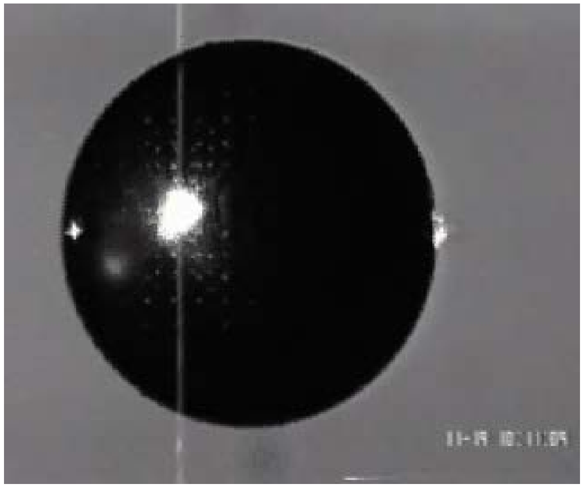


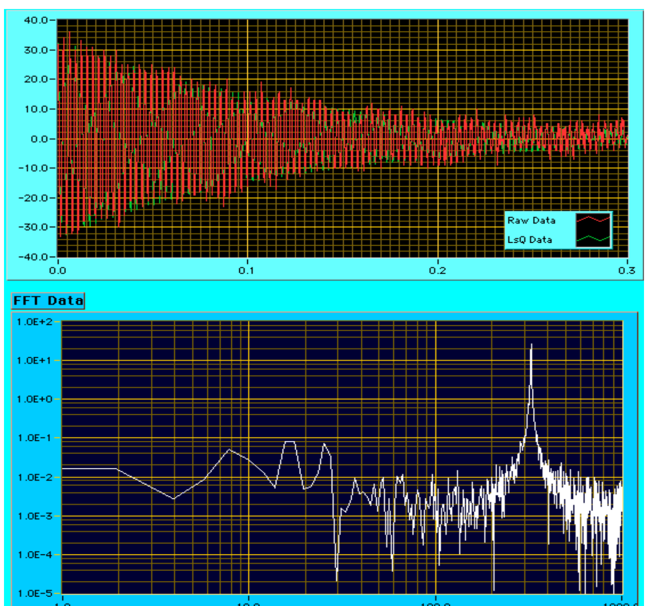
Measured correctly in the wide range of temperature including undercooling.

Estimated with the weight measured before heating.

[1] Paradis, et al., J. Jpn. Appl. Phys. 43 (2004) 1496. [2] Glorieux et al., AIP Conf. Proc. 552 (2001) 316.
[3] Kirshenbaum et al., J. Inorg. Nucl. Chem. 14 (1960) 283. [4] Florian Kargl et al., Int. J. Microgravity Sci. Appl. 32 (2015) 320200 .

Surface tension and viscosity





Oscillating drop method

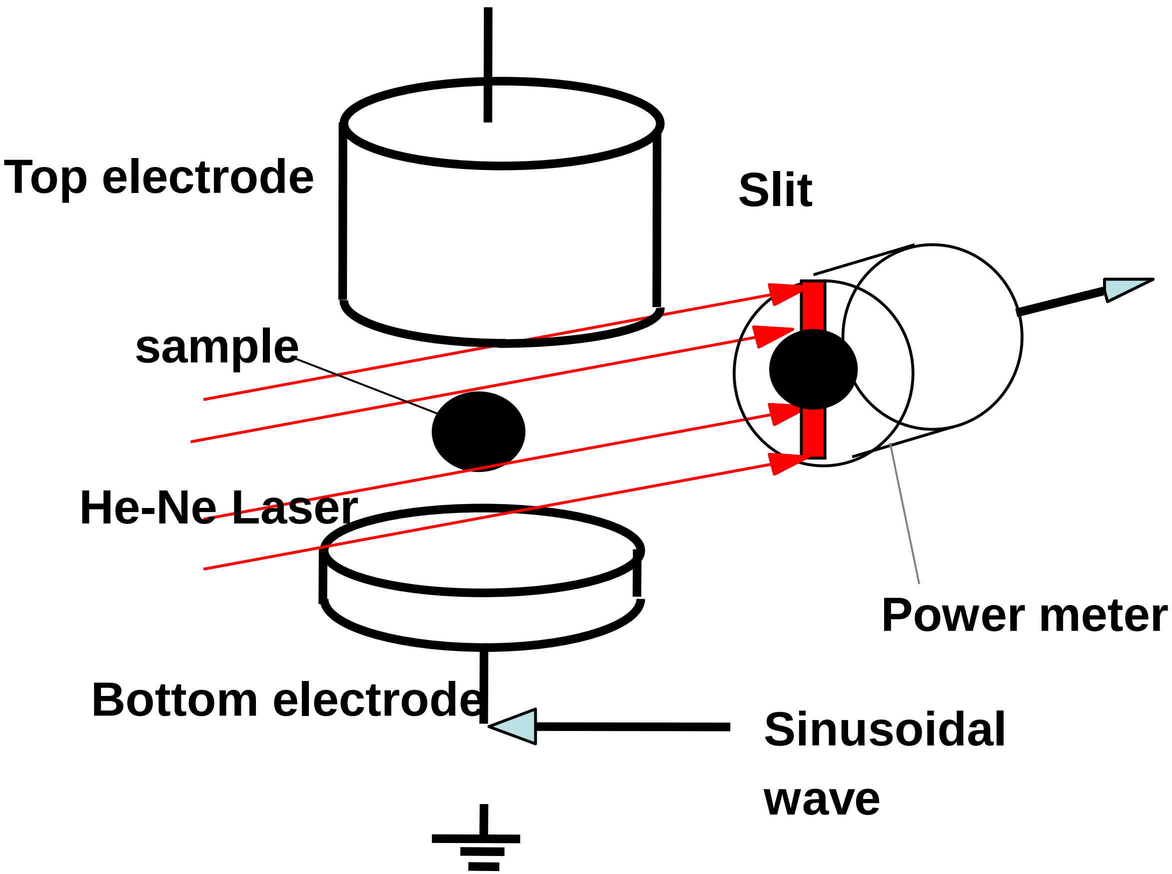
$$\gamma = \frac{{\omega_2}^2 \rho {r_0}^3}{8}$$

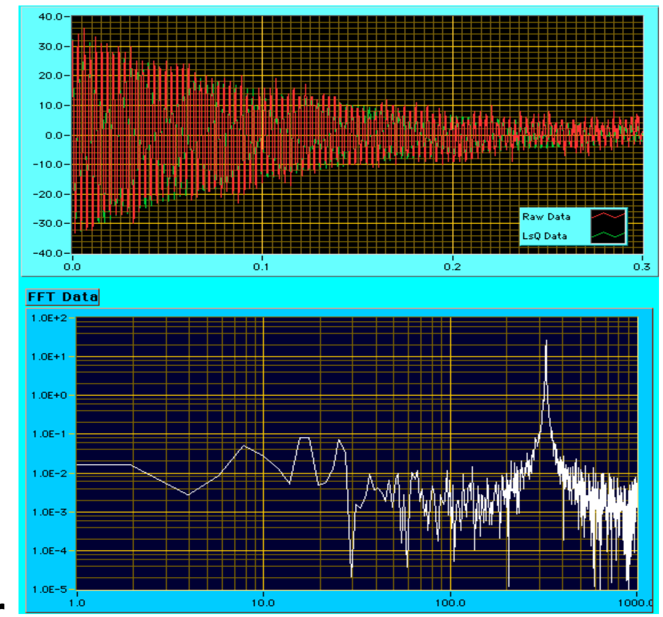
$$\eta = \frac{\rho {r_0}^2}{5\tau}$$

ω_2: Freq. of mode 2 oscillation
τ: Decay constant
ρ: density **r** $_0$:radius of sample
γ: surface tension η: viscosity

Rhim et al., *Rev. Sci. Instrum.* 70 (1999), 2796

Oscillation excitation/ detection



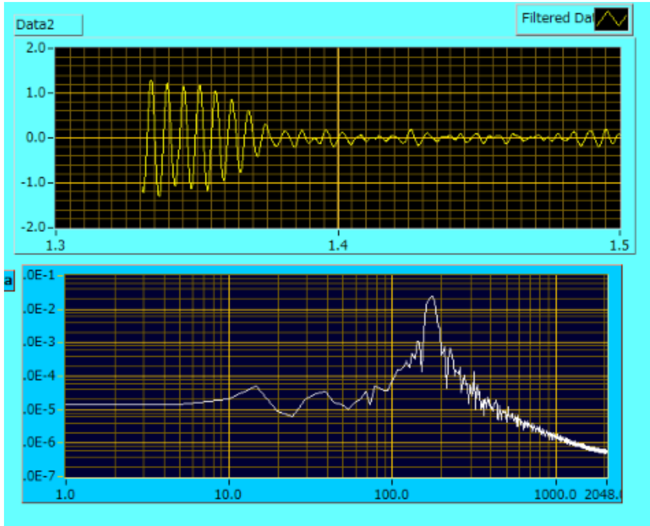


Uncertainty:

γ ±3%

η ±10-15%

Surface tension and viscosity of molten Al_2O_3

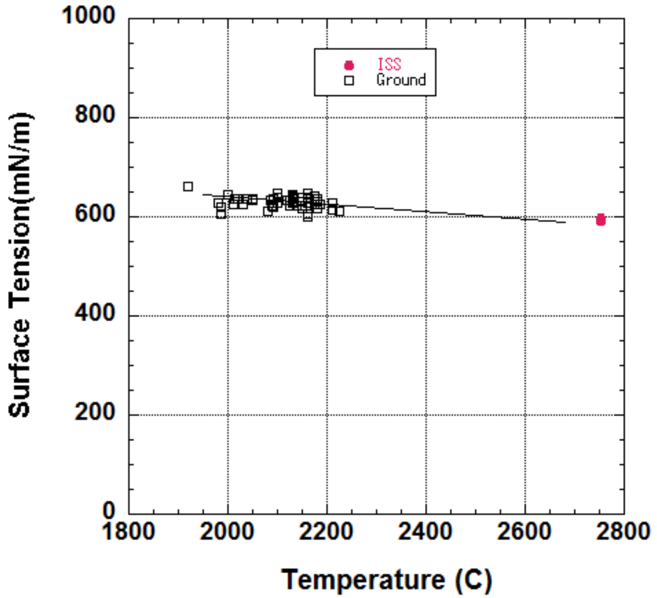


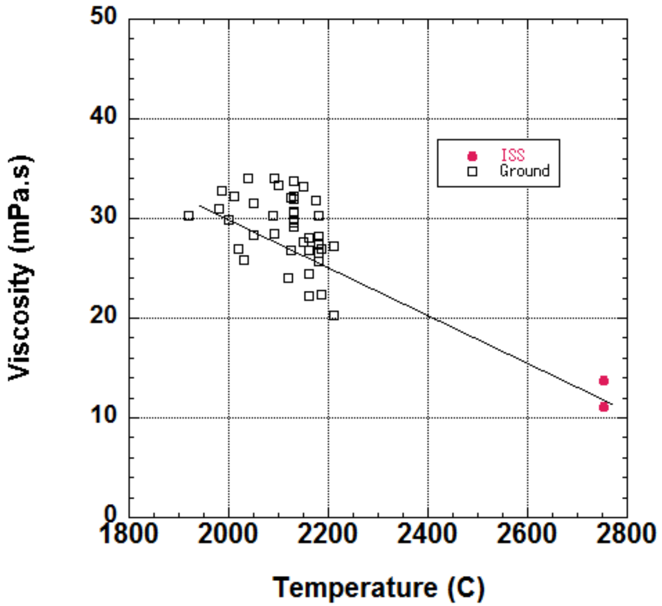
$$\tau = 0.0515 \text{ sec}$$

$$\Rightarrow \eta = 13.8 \text{ mPas}$$

$$\omega_2 = 1090\ rad/s$$

$$\Rightarrow \gamma = 592 \text{ mN/m}$$



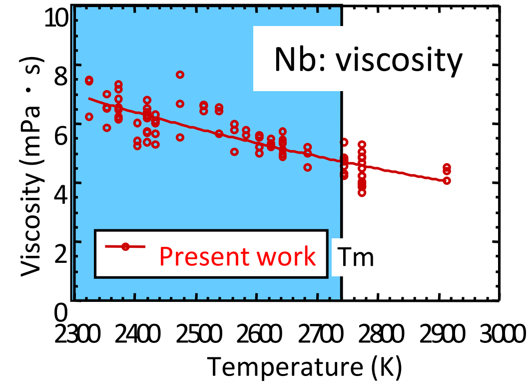


Utilization Plan

- Interfacial Energy (2018-2019)
  - Interfacial tension measurement of molten steel and oxides as basic research on a steel refining process
- Fragility (2018-2019)
  - Density and viscosity measurements of high temperature molten oxides for elucidating the origin of the vitrification
- Technical Demonstration (2017-2019)
  - Thermophyisical property measurement of binary oxides.
- NASA experiments (2020)
  - Under the Japan-U.S. Open Platform Partnership Program (Japan-U.S. OP3), four experiments will be conducted.
  - Establishing framework for JAXA-NASA Material Science research collaboration is on going.

Contribution to Material Science

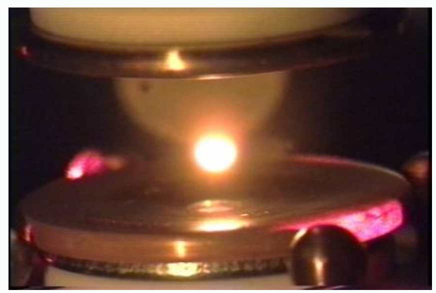
Combined structural analysis of high temperature liquid with X-rays on the ground and ISS-ELF



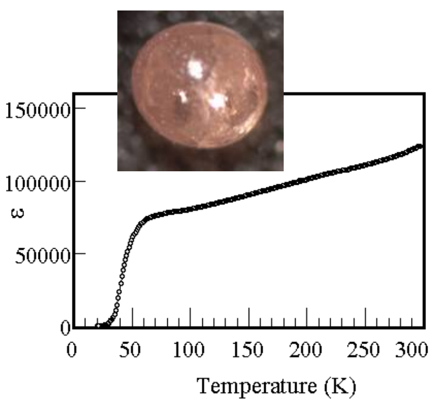
thermophysical properties

Contribute Earth Benefits

Investigation the unknown liquid states of oxidized materials will achieve breakthroughs in science and materials development..

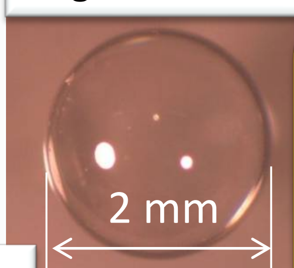


Computer-based casting simulation in order to develop efficient turbines, aircraft, and jet engines.



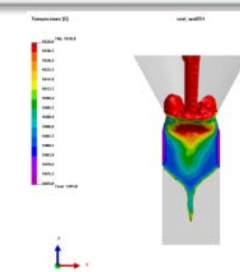
Giant permittivity of $BaTiO_3$

New glass with high reflection index



Novel material synthesis is open the way for future practical application and production on Earth.

Casting Simulation



Summary

- Density measurement of molten Al_2O_3, Fe_2O_3, Er_2O_3 and Gd_2O_3
- Surface tension and viscosity of Al_2O_3, etc.

  → ELF's all functions were verified successfully.
- Establishing International Collaboration framework is on-going.