Experiments using the ISS Electrostatic Levitation Furnace (ELF) and Solution Crystallization Observation Facility (SCOF)

Materials Science for the ISS Workshop

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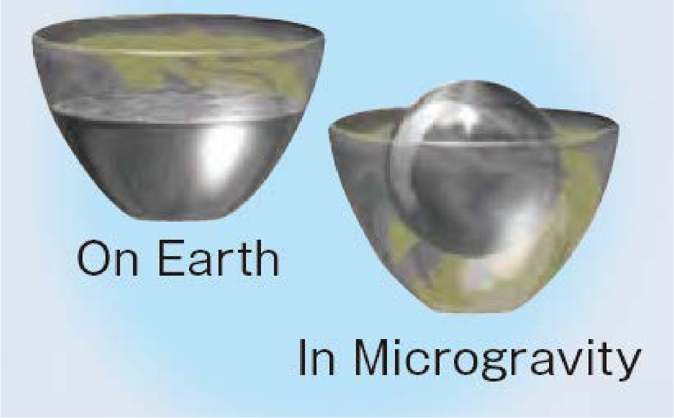
1. Electrostatic Levitation Furnace (ELF)

1. Overview of ELF
1.1. Advantage of ELF

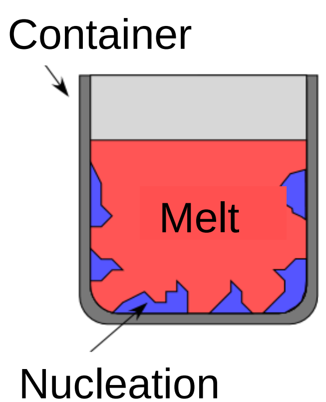
ELF can conduct material processing (melting, solidification, etc.) without container.

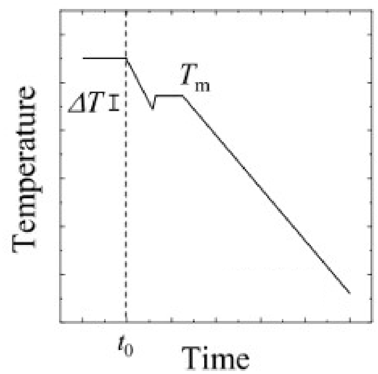


✓ No contamination from container.
→ ELF can obtain high temperature material thermophysical properties.

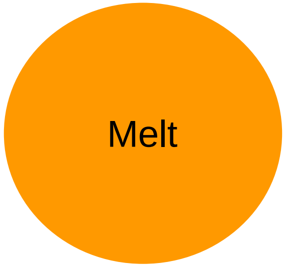
✓ Can prevent heterogeneous nucleation from container at solidification.
→ ELF can achieve large super cooling.

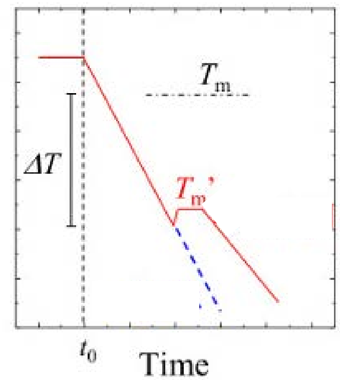
Container





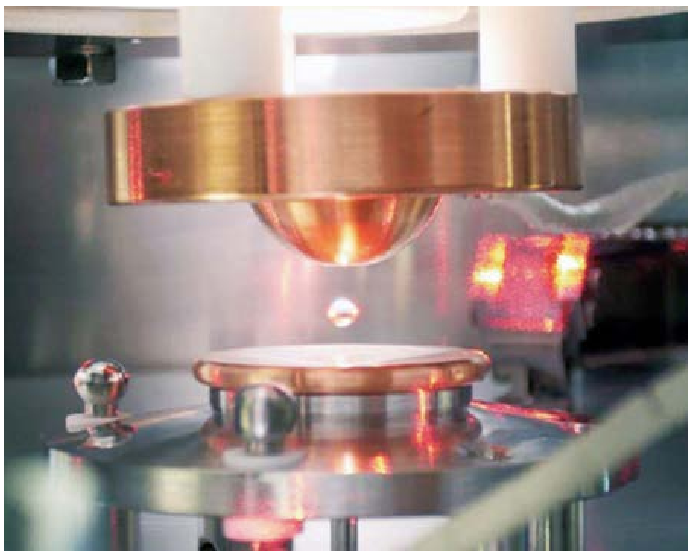
Container



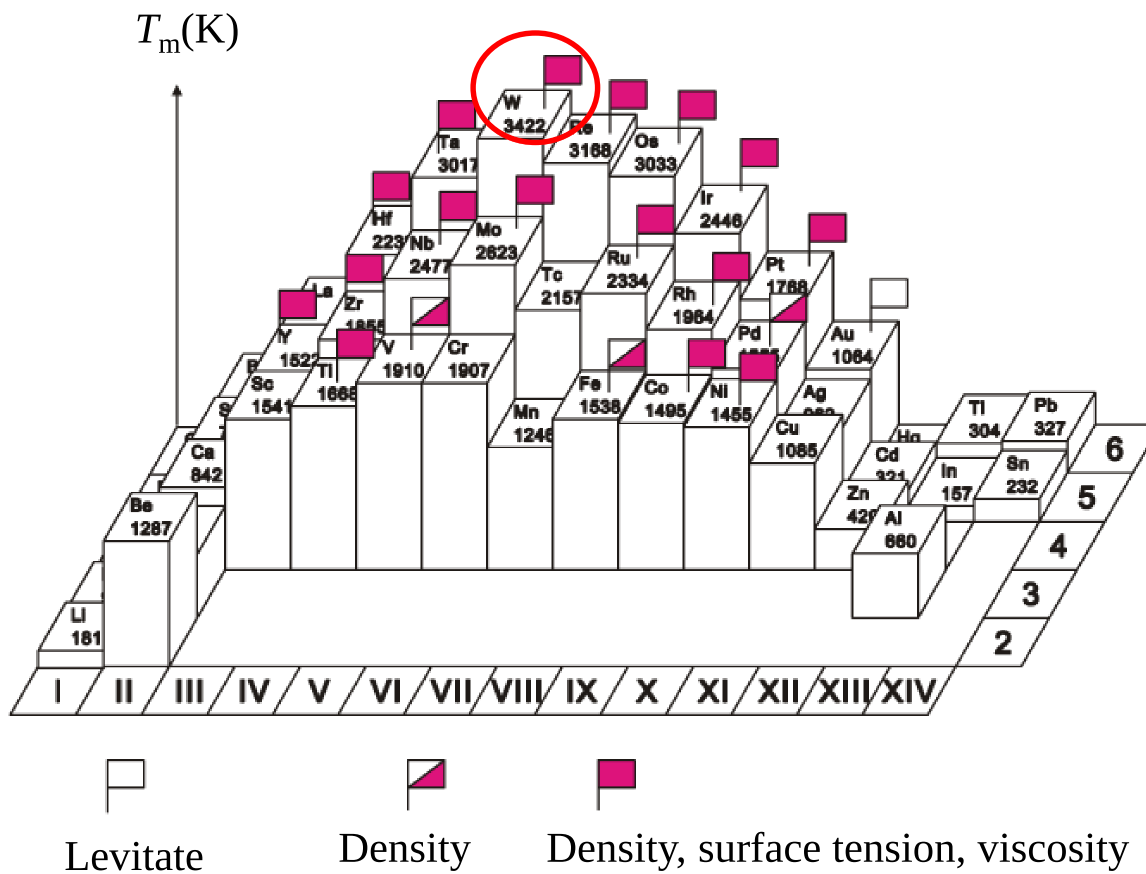


1. Overview of ELF
1.2. Results of ground experiment

ELF can obtain thermophysical properties (density, surface tension, viscosity) of tungsten which have highest melting temperature of metal.



Sample is levitated by ELF



1. Overview of ELF
1.3. Why in space ?

	Ground (1G)	Space（microgravity）
Electric field	• Need large electric field which can overcome gravity (8kVcm^{-1}<)[1]	• Need small electric field because no need to overcome gravity (<3kVcm^{-1})
Feasibility of levitation	• Metals, alloys: easy for levitation because they have much charging on surface. • Oxides: difficult for levitation because they do not have enough charging on surface.	Easy for levitation both metals, alloys, and oxides.
Atmosphere	• high vacuum (to prevent electrical discharge by high electric field) • can not use inactive gas (will occur electrical discharge)	Can use inactive gas, vacuum (because small electric field)
Effect of sample evaporation	• Metals: little effect • Alloys, oxides: sample composition will change by evaporation	Can suppress sample evaporation by using inactive gas

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

1. Overview of ELF
1.4. Comparison with other facility

- **ESA: Electromagnetic Furnace (launched on ATV5 in 2014) :**

  Sample is limited for conductors (metal, alloys), because sample is levitated by Lorentz force.
  Can heat 5–8 mm diameter sample max. 2,000 degree Celsius.

- **JAXA:ELF (launched in 2015) :**

  Can accommodate various materials, and wide temperature range, because utilize coulomb force between charged sample and electrodes.

	Low temperature (~500℃)	High temperature (500-2,000℃)	Very high temperature (>2,000℃)
Conductor (metal, alloy)	Electromagnetic Furnace(ESA)	Electromagnetic Furnace(ESA)	ELF(JAXA)
Insulator (oxide)	ELF(JAXA)	ELF(JAXA)	ELF(JAXA)

Comparison of IP facility cover range on ISS

1.5. ELF target

(1) Measurement of unknown thermophysical properties

Acquisition of unknown oxides thermophysical properties.

Develop database of thermophysical properties which is important for industry.

Acquisition of high accuracy thermophysical properties corresponded for request from company.

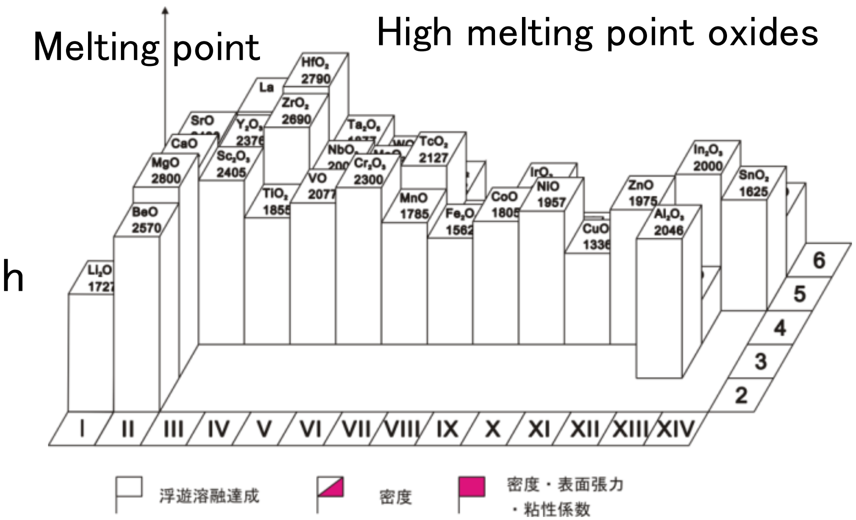
(e.g.) practical alloy (TiVAl alloy, etc.), heat resistance material (ZrO2, SiO2, etc.)

JAXA try to obtain thermophysical properties of binary oxides which have high melting point.

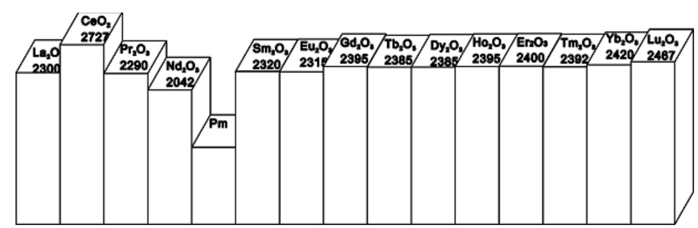
(2) Search for new high performance materials

ELF has a possibility to develop new material using super cooling.

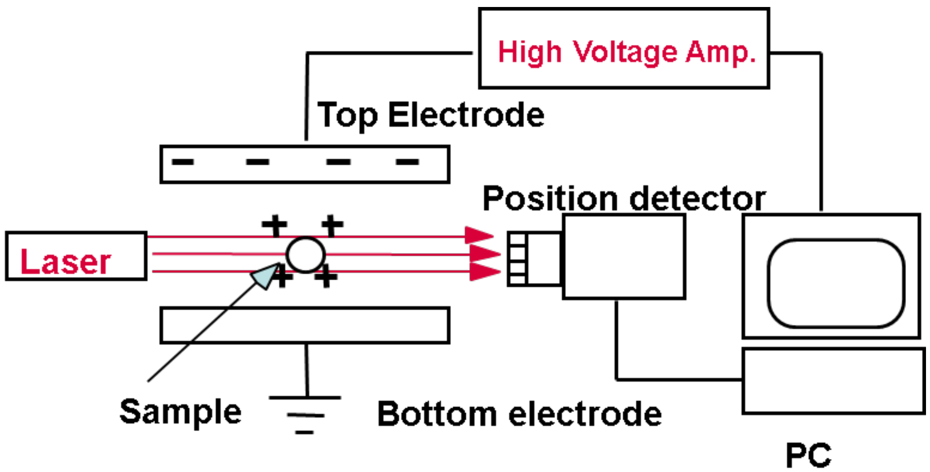
Sample is retrieved to the ground and be analyzed its fine structure.



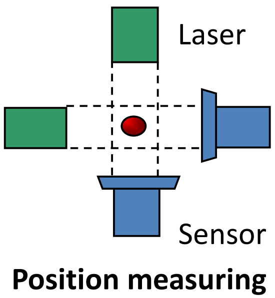
Rare earth oxide

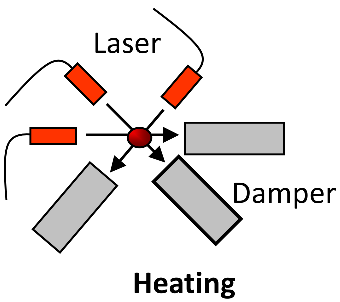


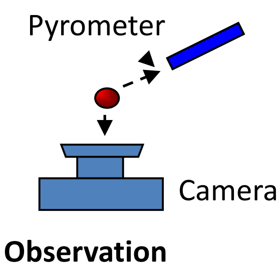
1.6. How it works



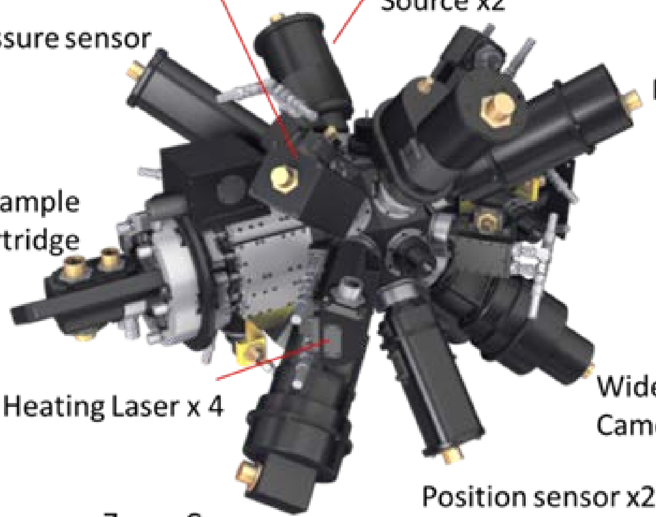
- ✓ ELF detects sample position in high accuracy, and control sample position by feedback to electrodes.
- ✓ Once sample is controlled in right position, then sample is heated by laser.
- ✓ Sample temperature is measured by pyrometer.







1. Overview of ELF
1.7. ELF specification



ELF components
- ✓ chamber
- ✓ sample cartridge
- ✓ laser (x4)
- ✓ pyrometer
- ✓ position sensor (x2)
- ✓ observation camera (x2)
- ✓ pressure sensor
- ✓ oxygen sensor

item	specification
sample type	oxide (main target), insulator, metal, alloy
sample size	2mm diameter
atmosphere	Ar, N_2, Air (max 2atm), vacuum
heat	semiconductor laser (980nm), max 40W x4
measurement temperature	299～3,000℃

1. Overview of ELF
1.8. How to operate

space

Transfer sample holder to ISS by visiting vehicle.

Astronaut install sample holder to ELF.

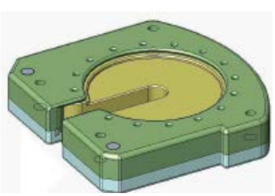
ELF experiment in KIBO

Retrieve sample holder to the ground.

ground

Launch sample holder by rocket

Bi-directional communication via satellite.

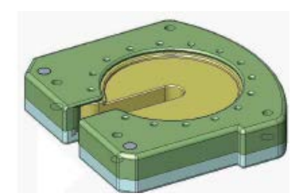


Sample holder
Size: 86 x 53 x 24 mm
mass : 70g

Samples are loaded to sample holder.
Sample holder can storage 15 samples.

Ground operation in Tsukuba

ELF experiment is remotely operated from ground station.



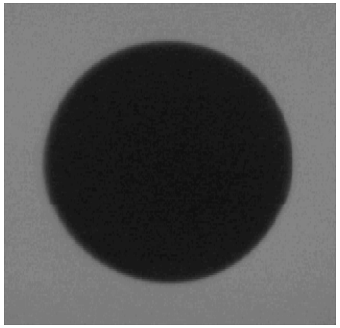
Investigator analyze retrieved samples.

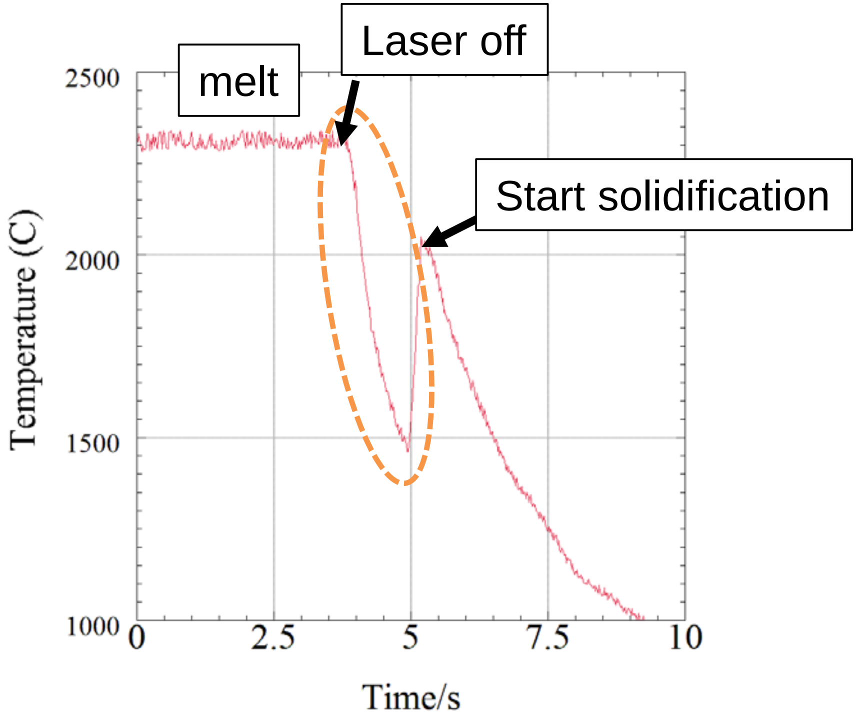
2. Measurement of thermophysical properties
2.1. Density

1. Sample is heated and melt by laser.
2. Sample is cooled by stopping laser.
3. Images of sample in each temperature are obtained. Volume is measured by image analysis.
4. Sample is taken out from ELF, and retrieved to the ground.
5. Mass is measured on the ground.
6. Density is obtained from following equation.

$$\rho = \frac{m}{V}$$

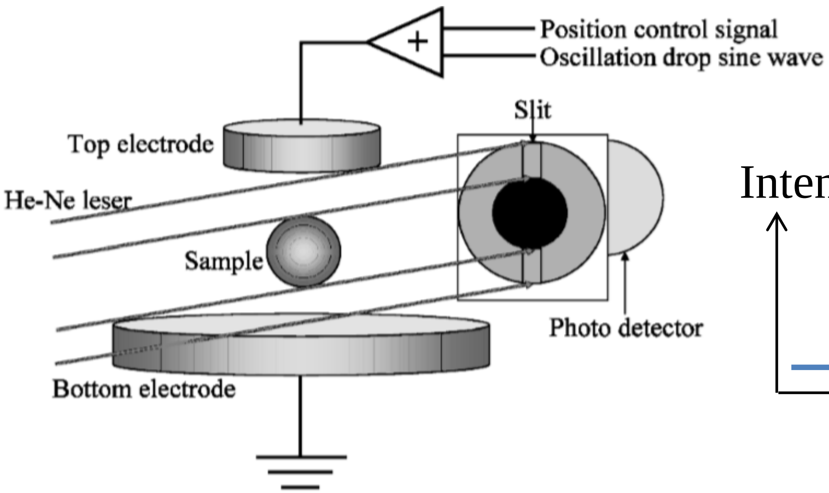
ρ :density
m :mass
V :volume

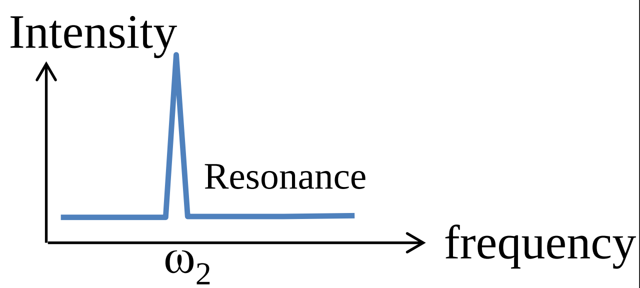


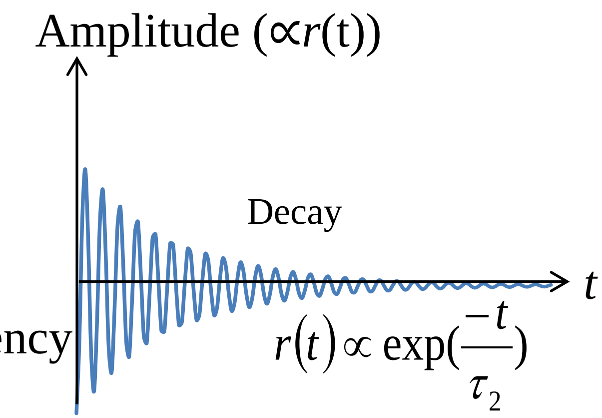


Temperature profile of molten aluminum oxide

Surface tension and viscosity are obtained from droplet oscillation







ω_2 :resonance frequency

τ :decay rate

r_0 :sample radius

η :viscosity

γ :surface tension

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

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

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

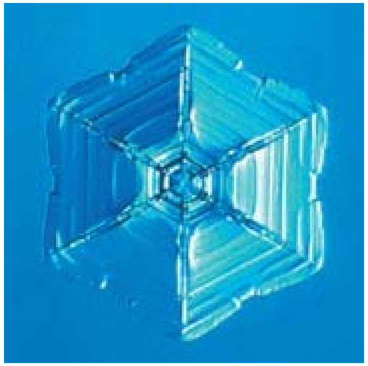
3. Utilization of ELF
3.1. ELF experiments

No.	PI	organization	title	abbreviation	status
1	Prof. Masahito Watanabe	Gakushuin University	Interfacial phenomena and thermophysical properties of high-temperature liquids -Fundamental research of steel processing using electrostatic levitation-	Interfacial Energy	On-orbit experiment
2	Dr. Shinji Kohara	National Institute for Materials Science	The origin of fragility in high-tempe rature oxide liquids - towards fabri cation of novel non-equilibrium oxi de materials	Fragility	Waiting on-orbit experiment
3	Prof. Shinsuke Suzuki	Waseda University	Thermo-physical properties of liquid and heterogeneous solidification behavior of powder metals for 3D printer	Hetero-3D	In preparation
4	Prof. Douglas Matson	Tufts University	Round Robin - Thermophysical Property Measurement	Round Robin	In preparation

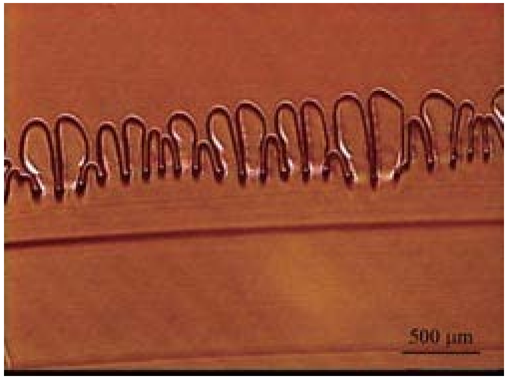
2. Solution Crystallization Observation Facility (SCOF)

SCOF（Solution Crystallization Observation Facility） Overview

SCOF can observe solution crystallization phenomena using various optical equipment.

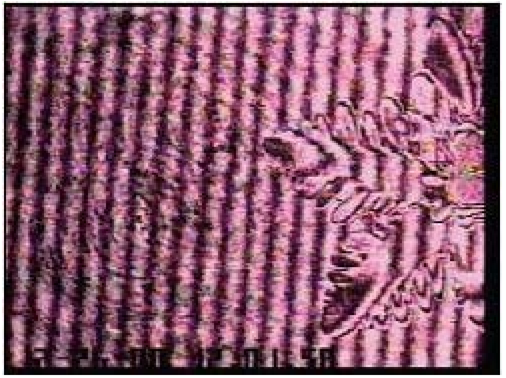


Ice crystal



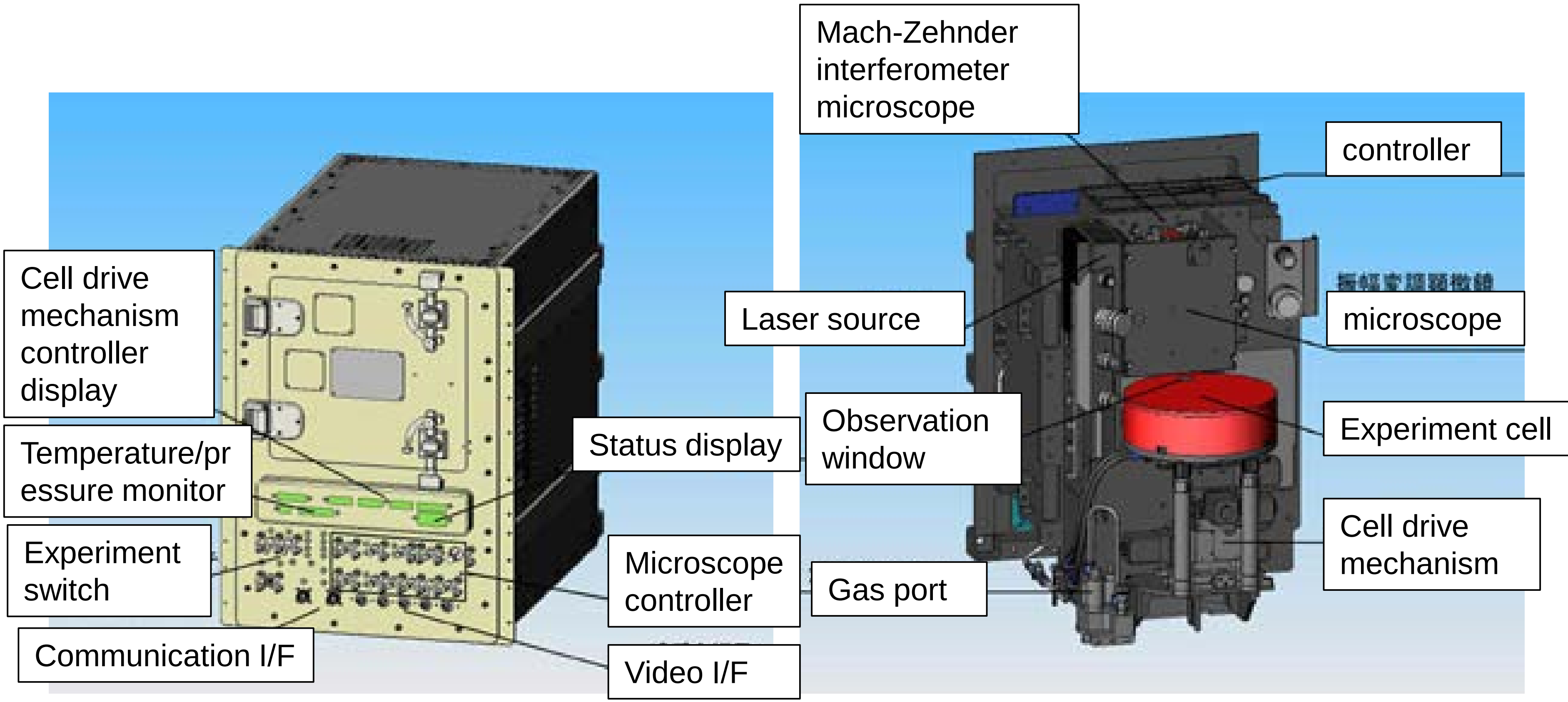
crystallization

Interferometer

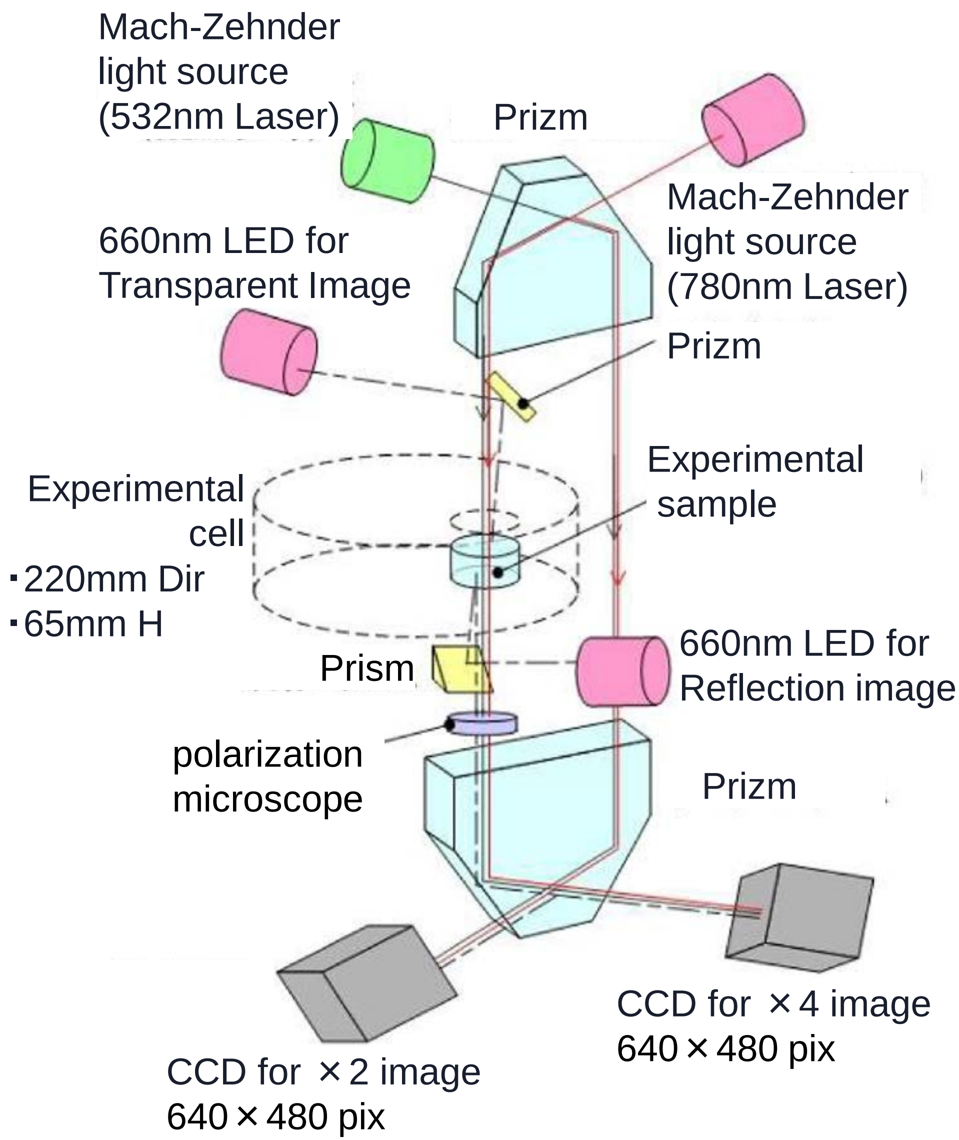


dendrite

SCOF（Solution Crystallization Observation Facility） Facility (1/2)



SCOF（Solution Crystallization Observation Facility） Facility (2/2)

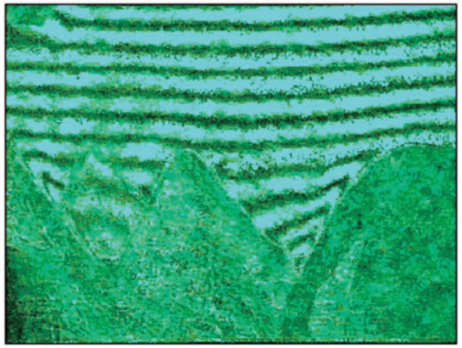


- ✓ SCOF experiment parameters can be changed by sending commands from Earth.
- ✓ Interfaces for experimental-cell temperature control and measurement, pressure control and measurement, cell stage XYZ positions, evacuation, and nitrogen gas supply are available.
- ✓ Schematic of SCOF optical setup is shown in the left figure. A Mach-Zehnder (MZ) interferometer enables interference fringes of the protein solution around the crystal growth interface.
- ✓ The polarization microscope, bright- and dark-field microscopes are available for observing crystal shapes, surfaces, etc.
- ✓ Experimental cells are available with standard dimensions of 220 mm in diameter and 65 mm in height (around MZ light path area).
- ✓ Nano-scale step dynamics on the protein crystal growth surface was detected by a Michelson type interferometer integrated in the experimental cell.

SCOF（Solution Crystallization Observation Facility） Specification

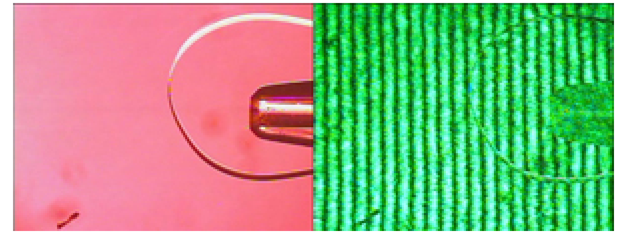
Item		Specification
Mach-Zehnder microscope	Magnification	x2, x4
	Laser source	LD and LD excitation solid laser (532nm, 780nm)
	Phase resolution	0.2 wave length
	Field of view	2.4 x 3.2mm for x2 1.2 x 1.6mm for x4
	CCD size	½ inch
Amplitude modulation microscope	magnification	x2, x4
	Light source	LED (660nm)
	Phase resolution	0. 2 wave length
	Field of view	2.4 x 3.2mm for x2 1.2 x 1.6mm for x4
	CCD size	½ inch
User interface	Temperature control	Perche device
	Temperature measurement	Thermistor, thermo couple
	Pressure control	0 to 147.1 MPa
	N2 supply pressure	0 to 827 kPa

SCOF（Solution Crystallization Observation Facility） Experiments



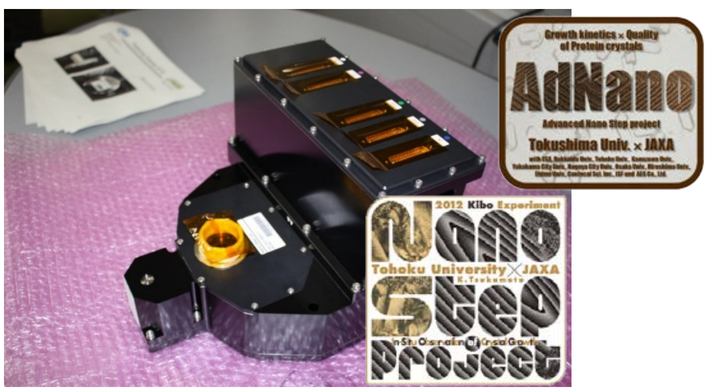
FACET experiment

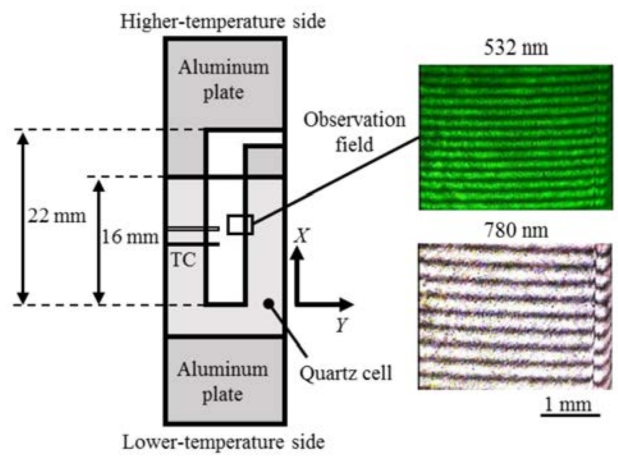
Formation mechanism of the faceted cellular array growth of salol-butanol alloy.



ICE and ICE2 experiment

ICE crystal growth mechanisms associated with the macromolecules adsorbed at a growing interface. And the microgravity effect for self-oscillatory growth.





Nano Step and Advanced Nano Step experiment

In-situ observation of growth interfaces of lysozyme and glucose isomerase crystals and crystal quality investigation.

Soret-Facet experiment

Study on Soret effect (thermal diffusion process) for the mixed solution (salol-butanol alloy) by the in-situ observation