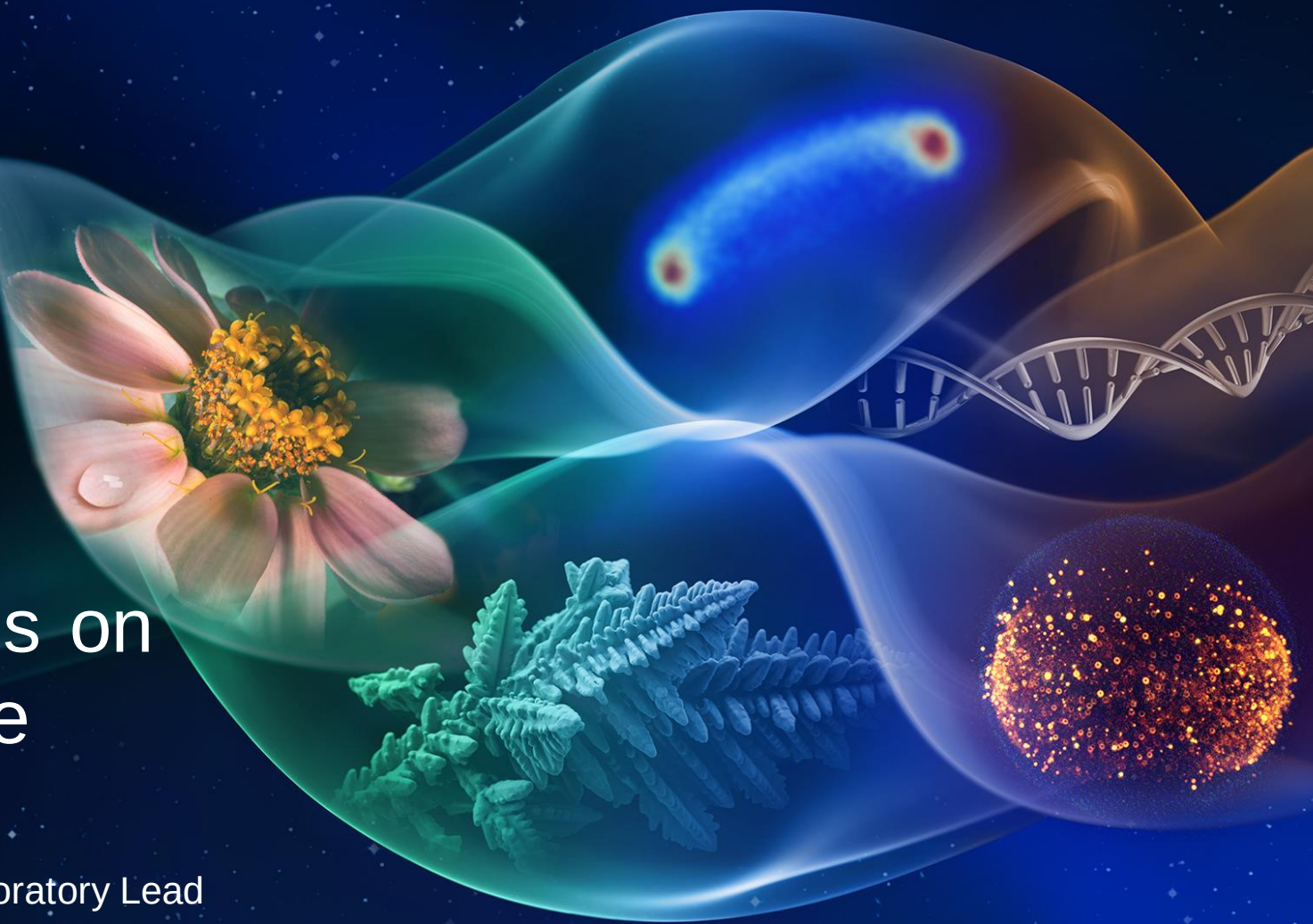




Biological and Physical Sciences

Levitation Capabilities on Ground and In Space

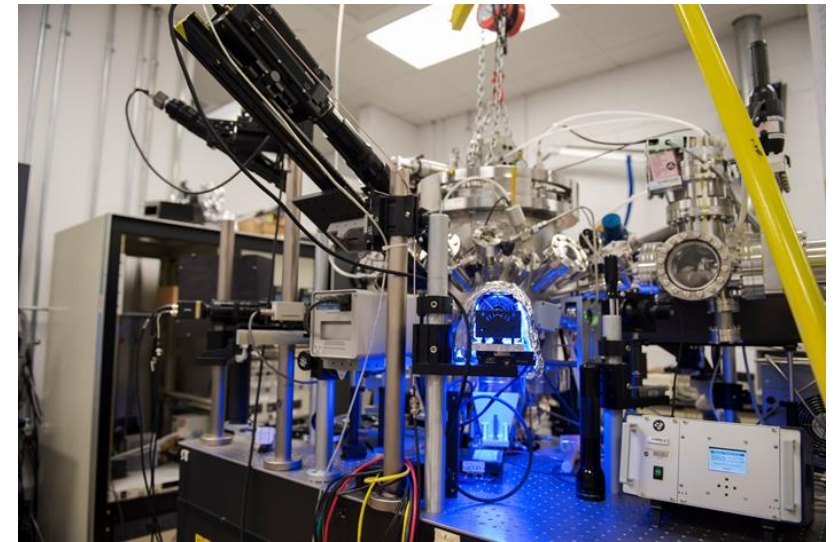
Michael SanSoucie
NASA Marshall Space Flight Center
MSFC Electrostatic Levitation (ESL) Laboratory Lead



MSFC ESL Lab

The MSFC ESL Lab is a resource for researchers developing advanced materials for new technologies

- Electrostatic levitation
 - Containerless process
 - Eliminates any container-sample interaction
 - Allows for deep undercooled of samples
- Can process elements, alloys, refractory metals, superalloys, ceramics, oxides, and glasses
- The lab typically measures thermophysical properties
 - Density, Surface Tension, and Viscosity
- The lab hosts government, academic, and commercial investigators
- Provides ground-based support for US investigators with levitation experiments on the International Space Station (ISS)
 - ESA ISS Electromagnetic Levitator (ISS-EML)
 - JAXA Electrostatic Levitation Furnace (ELF)

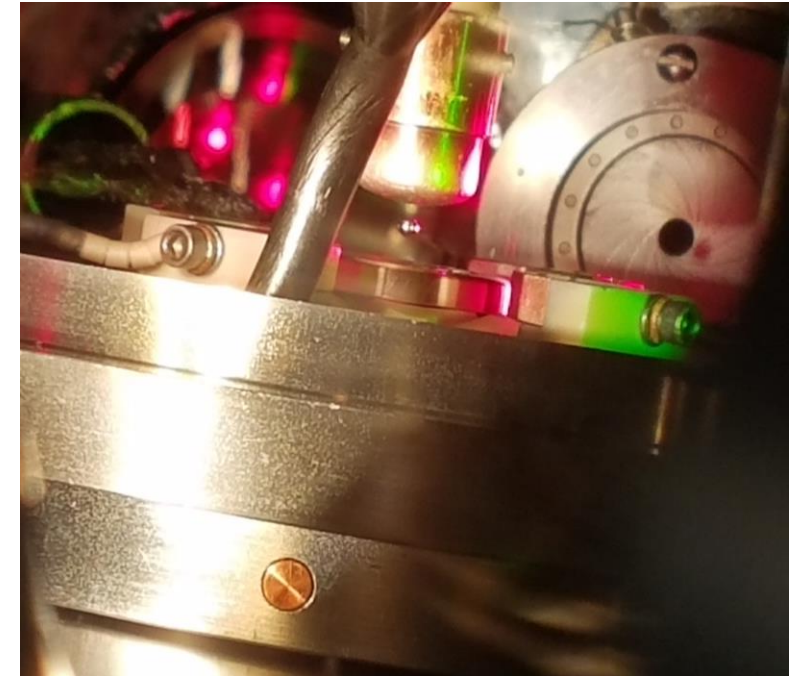


Electrostatic Levitation (ESL)

- ESL uses an electrostatic field to levitate charged specimens
 - Typically 2-3mm spheres
- The field is created between 6 electrodes
 - 3 are grounded
 - 3 are connected to high-voltage DC amplifiers
 - Top electrode is typically held at a negative potential
- Positive charge is maintained by irradiating the sample with UV light
 - Deuterium arc lamp
- As samples are heated volatile components tend to evaporate as positively charged ions
 - Surface ionization
 - Requires recharging
 - Photoelectric emission of electrons
- Samples are heated and melted with a laser
 - Either fiber optic (1064nm) or CO₂ (10600nm)
- Temperature is measured with a pyrometer
 - InGaS (~1.5 μ m) typically

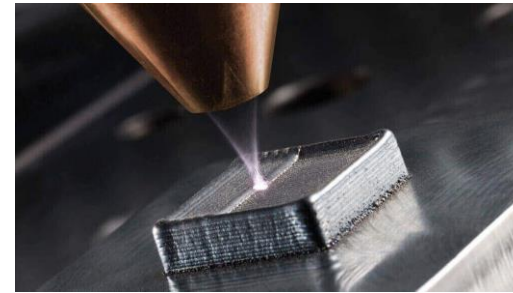


~2mm sample

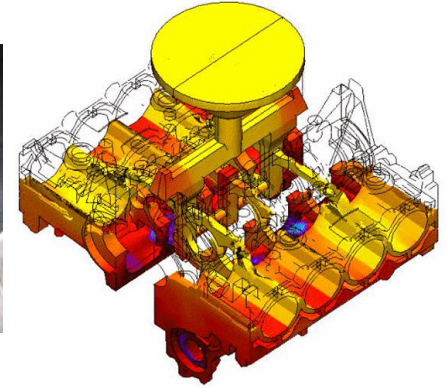


Why Levitation?

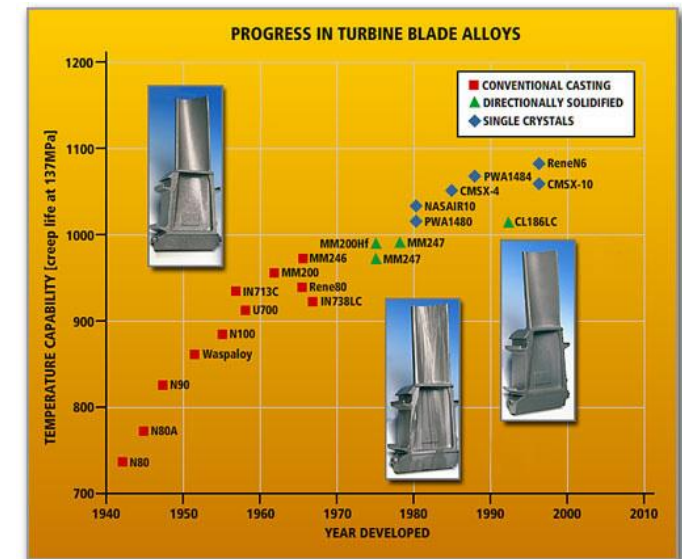
- In order to expand the boundaries of space exploration, its necessary for spacecraft hardware to operate in extreme environments.
 - To meet these demands new materials must be developed
- Developing new materials (e.g. Vitreloy106 bulk metallic glass and rare earth aluminum oxide glasses) can and has been done with levitation experiments in the past.
 - SMD used bulk metallic glass as a detector material in the Genesis mission
- Spacecraft and manufacturing techniques are now almost always modeled.
 - Modeling is used to understand and control high-temperature processes, such as:
 - Welding
 - Casting
 - Additive manufacturing
- Accurate models require accurate material properties
 - “Garbage in, garbage out”



Metal additive manufacturing

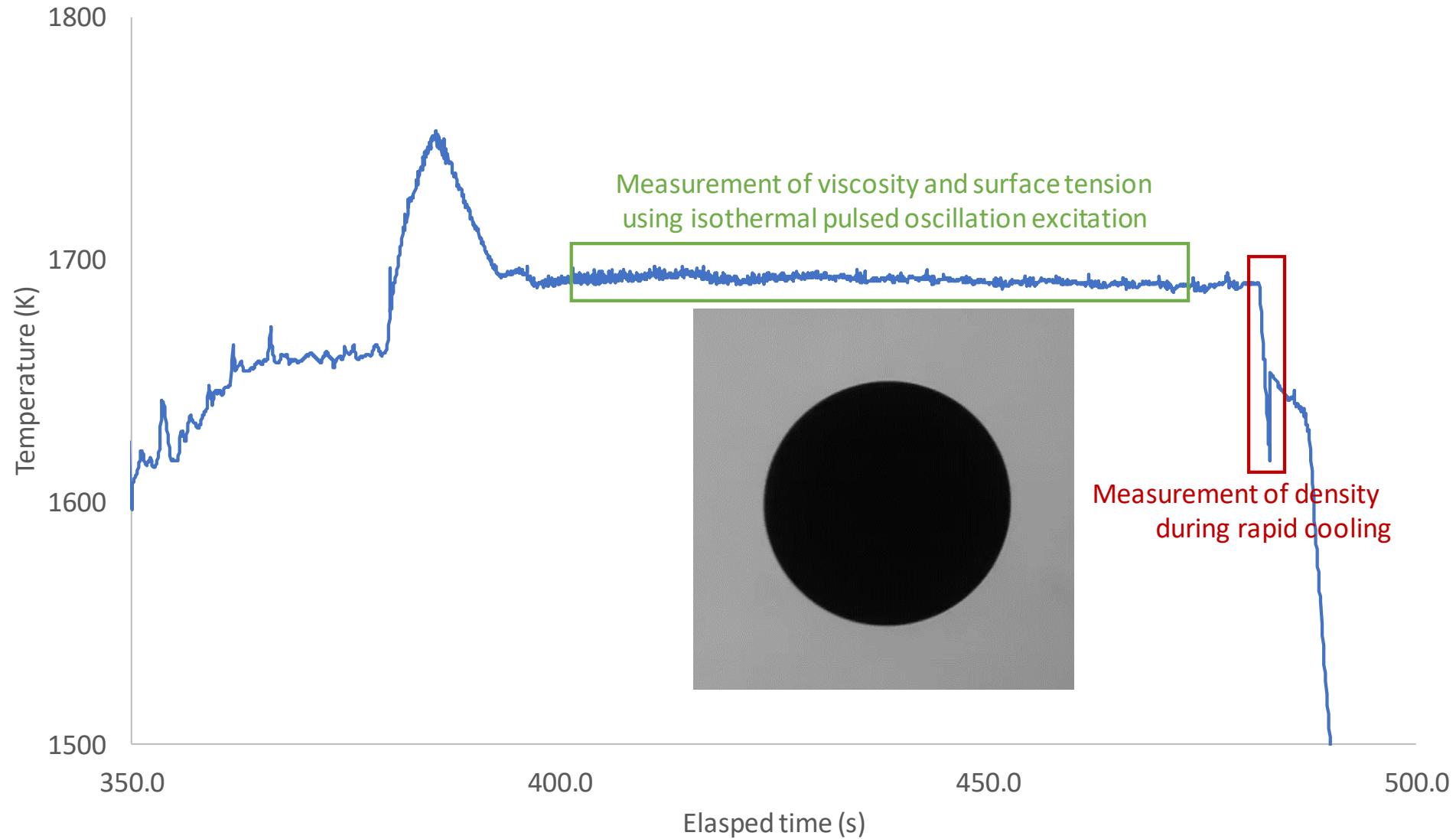


A model of a casting process



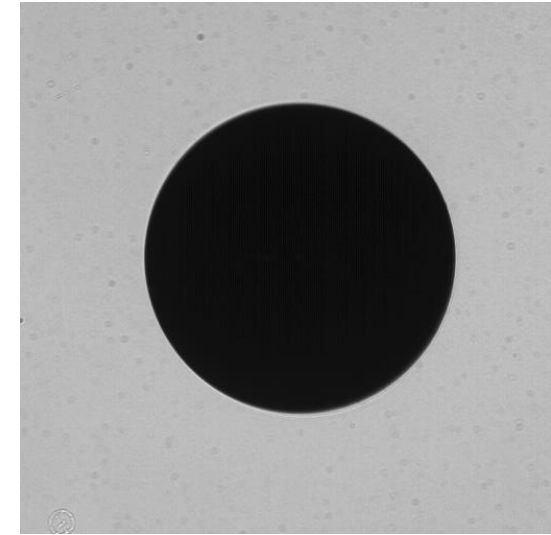
https://www.grc.nasa.gov/WWW/StructuresMaterials/AdvMet/research/turbine_blades.html

Thermophysical Properties Measurement

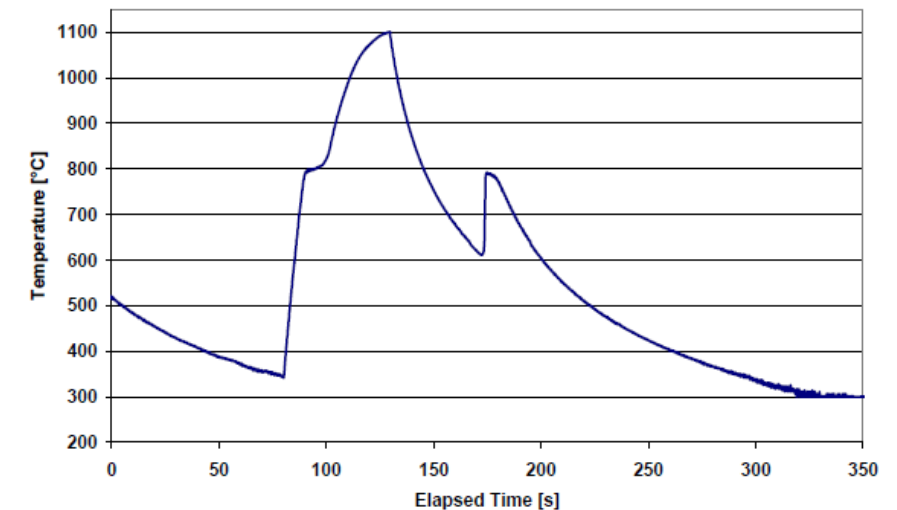


Density Measurements

- Uses a high-speed camera
- Samples are backlit to create dark silhouettes of the sample on a gray background
- Video is taken at 30fps
- Edge detection and a polynomial fit is done in order to calculate the volume of the sample
- Mass is taken both before and after processing
 - $D = M/V$



Backlighting image of a sample for a density measurement



Typical thermal profile for a density measurement

Surface Tension & Viscosity Measurements

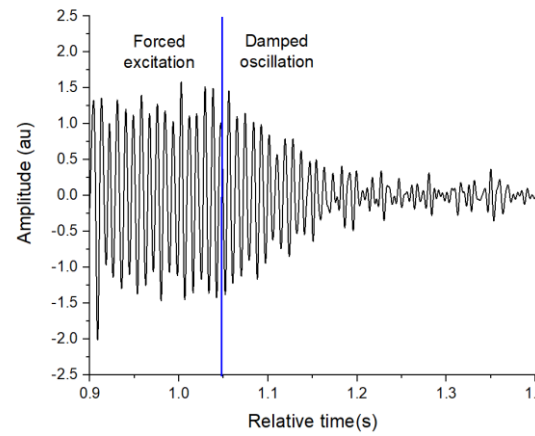
- Oscillating drop method
- Surface oscillations are induced in the molten sample by modulating the electrostatic field near the resonant frequency
- Once stable mode 2 oscillations have developed
 - Modulation is turned off
 - Natural frequency of the resulting oscillations and the damping are recorded with high-speed video
 - Typically at least 1000fps
- Surface tension calculated from natural frequency
- Viscosity calculated from damping coefficient

$$\omega_l = \sqrt{\frac{l(l-1)(l+2)\gamma}{\rho R_o^3}}$$

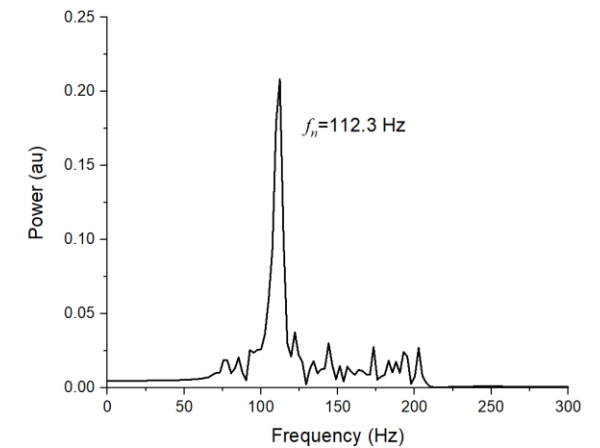
Rayleigh
(1879)

$$\tau_l = \frac{\rho R_o^2}{(l-1)(2l+1)\mu}$$

Lamb
(1881)



Oscillation data



Fast Fourier Transformation

Electromagnetic Levitation in Space

International Space Station Electromagnetic Levitator (ISS-EML)

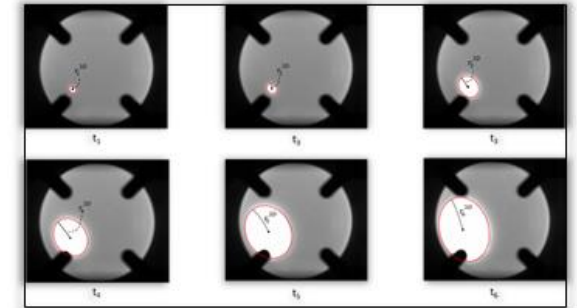
- A multi-user facility for the melting and solidification of conductive metals, alloys, or semiconductors in ultra-high vacuum, or in high-purity gaseous atmospheres.
- The heating and positioning of the sample are accomplished using electromagnetic fields generated by a coil system.
- Installed in the European space laboratory Columbus.

There are currently 3 US PIs with experiments on the ISS-EML

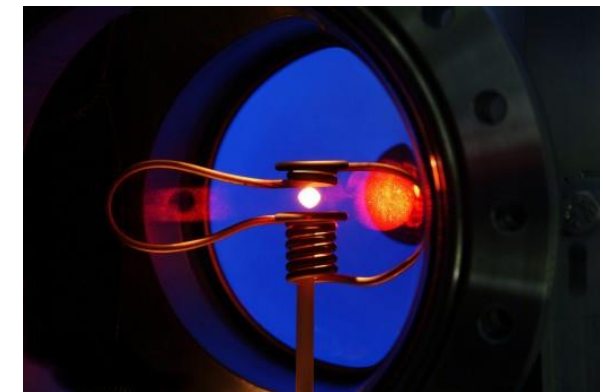
- Robert Hyers, University of Massachusetts
 - Objective: Investigate the thermophysical properties of high-temperature materials used for modeling of material production processes, e.g. additive manufacturing, welding, and casting.
- Kenneth Kelton, Washington University in St. Louis
 - Objective: Correlate the nucleation kinetics with the local structure of a liquid alloy by measuring its thermophysical properties. Applications include bulk metallic glass materials.
- Douglas Matson, Tufts University
 - Objective: Investigate the effect of fluid flow on the solidification path of aerospace alloys, and to measure the thermophysical properties of high-temperature materials. Applications include turbine blades, industrial welding, automobile components.



~8mm



Growth velocity analysis of a ZrNi ISS-EML sample. Liquid is gray, solid is white.



Sample levitated in an EML at DLR.

Electrostatic Levitation in Space

JAXA Electrostatic Levitation Furnace (ELF)

- Materials science facility for thermophysical properties (e.g. density, surface tension, and viscosity) measurement of molten metals, alloys, glasses, and oxides.
- Samples are levitated with an electrostatic field and heated with 4 970nm 40W lasers.
- ELF is installed in the MSPR-2 (Multi-purpose Small Payload Rack #2) in Japanese Experimental Module “KIBO” in the ISS.

There are 4 US investigators with experiment on the JAXA ELF:

- Robert Hyers, University of Massachusetts
 - Objective: Advance the understanding of photorefractivity, which has applications in holographic storage, adaptive optics, and phase-conjugate mirrors, using Bi₁₂SiO₂₀ and Bi₁₂GeO₂₀.
- Douglas Matson, Tufts University
 - Objective: Understand and control the sources of measurement error and to provide a baseline dataset for quantifying uncertainty in measurements (both space- and ground-based).
- Ranga Narayanan, University of Florida
 - Objective: Investigate a novel method to determine the interfacial tension, which is a property that impacts industrial processes such as and additive manufacturing.
- Richard Weber, Materials Development, Inc
 - Objective: Develop new understanding of the behavior of oxide liquid, glass and ceramic materials. Applications include photonics, lasers, and piezoelectrics.



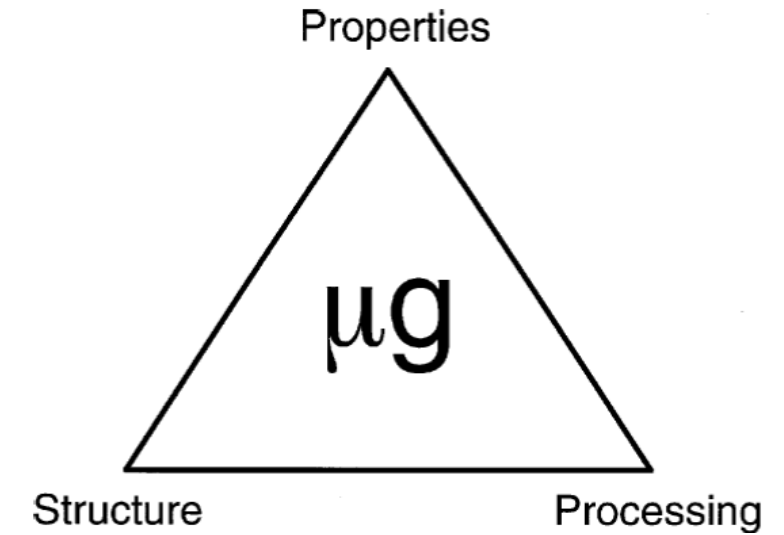
Sample levitated in ELF on ISS during Dr. Matson's ops.



ELF Sample cartridge.

Why Levitation in Microgravity?

- The International Space Station (ISS) provides a long-duration spaceflight environment for conducting microgravity experiments.
- The microgravity environment greatly reduces buoyancy-driven convection, pressure head and sedimentation in fluids.
- Many thermophysical properties can be measured in a levitator on Earth, but with convective contamination.
 - This contamination plays a significant role in the formation of the intermediate phases.
 - In particular nucleation and viscosity measurements demand quiescent conditions.



Establishing quantitative and predictive relationships between the way a material is produced (processing), its structure (how the atoms are arranged), and its properties is fundamental to the study of materials.

TEMPUS Experiments

- Bulk metallic glass (BMG) alloys are metallic alloys that form a non-crystalline (or glass-like) structure when solidified.
 - First produced by CalTech in 1960
- Spacelab missions
 - IML-2 on STS-65 (1994)
 - MSL-1 on STS-83 (1997)
 - MSL-1R on STS-94 (1997)
- NASA PI, Dr. Bill Johnson of Caltech, studied the metallic glass forming alloys in TEMPUS to gain fundamental insight into the nature of metallic glass formation.
- Dr. Johnson's NASA support and containerless processing was critical to the developments of BMGs.
- The fundamental mechanisms behind glass formation are still being studied today.



Zr₆₄Ni₃₆ BMG sample for
IML-2 TEMPUS
experiments

Bulk Metallic Glass Development at MSFC

- The MSFC ESL lab was utilized in 1998-2001 to show that a slight variant of Vitreloy 106, $\text{Zr}_{57}\text{Nb}_5\text{Cu}_{15.4}\text{Ni}_{12.6}\text{Al}_{10}$ (named Vitreloy 106a), exhibited a critical cooling rate much lower than Vitreloy 1 and also lower than Vitreloy 106.
- Liquidmetal Technologies and Liquidmetal Golf was spun off from the research done at MSFC



Golf club heads made from BMG by Liquidmetal Golf.

Ref: <https://spinoff.nasa.gov/spinoff2001/ch3.html>

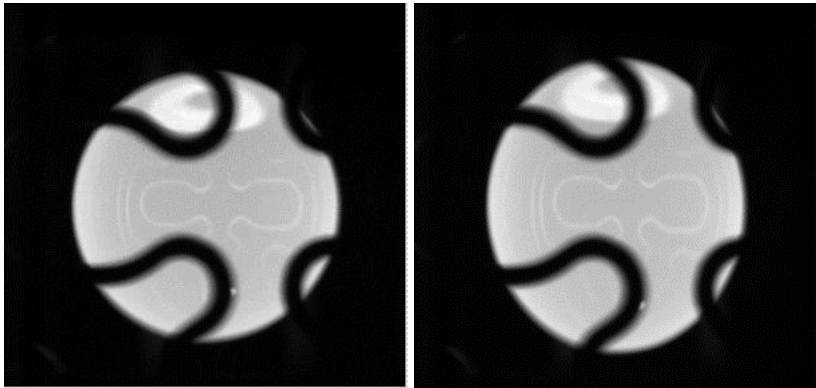


Liquidmetal engineers and Rawlings Sporting Goods designers developed a baseball bat that offers superior durability while dramatically reducing the energy loss experienced with other materials upon impact with the ball.

Ref: <https://www.nasa.gov/vision/earth/technologies/liquidmetal.html>

Current ISS Experiments with BMGs

- PI: Dr. Ken Kelton, Washington University in St. Louis
- Dr. Kelton's research shows that metallic glass characteristics can be determined while the metal is still molten liquid.
 - The liquid's viscosity can indicate whether a glass will form, what kind of glass will form as well as some of its properties.
 - This is a new development in metallic glass research and liquids in general.



Left: An undisturbed, spherical Vitreloy106a liquid in the ISS-EML.
Right: Image of the Vitreloy106a liquid elongated along its vertical axis during a viscosity measurement.

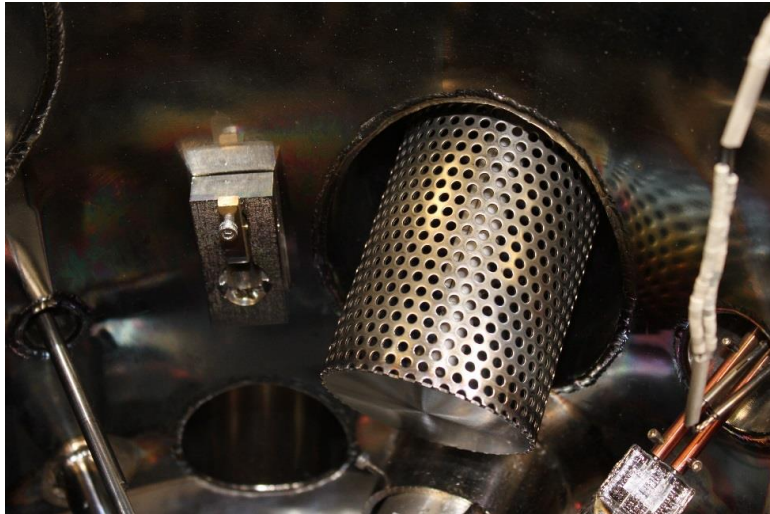
- PI: Dr. Doug Matson, Tufts University
- Dr. Matson's research uses the JAXA ELF and will provide baseline thermophysical property measurement experiments.
- One of his samples is CuZrAl
 - BMG-forming material that JPL provided



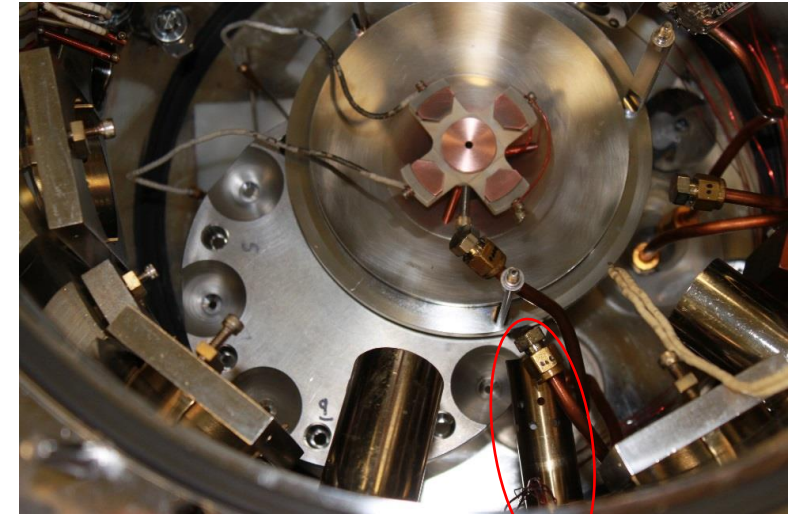


Interesting Capabilities of the MSFC ESL Lab

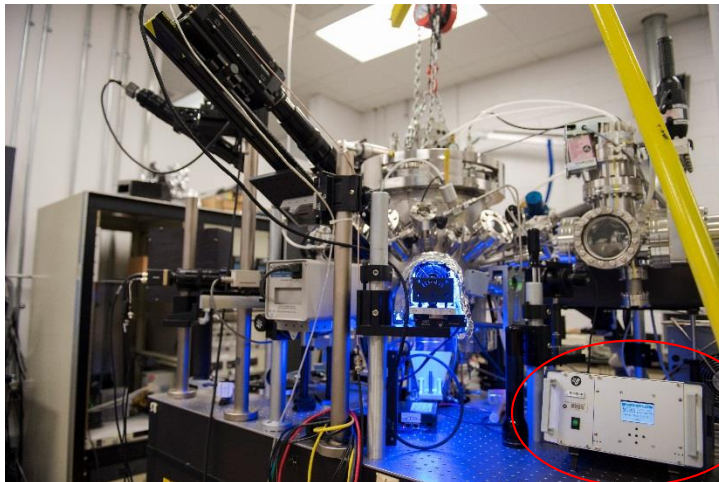
Oxygen Partial Pressure Control



Oxygen Pump



Oxygen Sensor

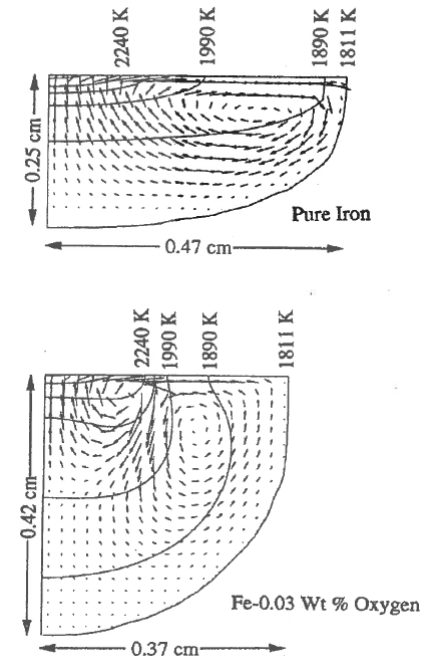


Controller

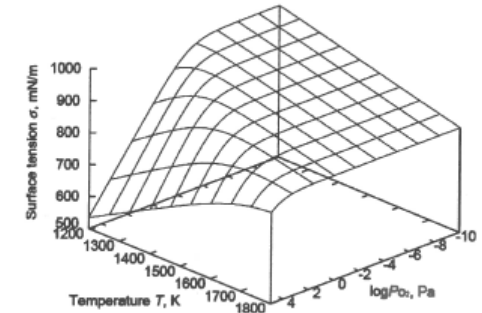
- Developed by Astrium North America
- Fabricated by Clausthal University of Technology (TU Clausthal)

Need for Oxygen Partial Pressure Control

- Surface tension of molten metals is affected by even a small amount of adsorption of oxygen
 - Oxidation may have an impact of 10-30% on surface tension measurements³.
 - Causes a decrease in surface tension
- Oxidation can occur at very low p_{O_2}
 - Occurs in the ESL as low as $\sim 1 \times 10^{-24}$ bar p_{O_2}
- Supports microgravity principal investigators
 - An oxygen control system is planned for the European Space Agency (ESA) Electromagnetic Levitator on the International Space Station (ISS)



Calculated velocity and temperature fields for gas tungsten arc welding of pure iron and iron with 0.03 wt% oxygen¹



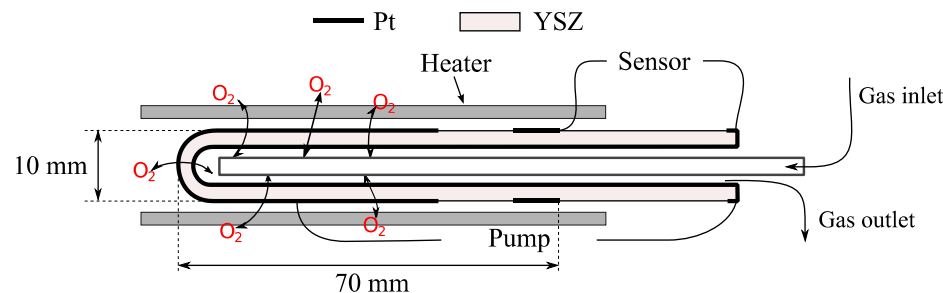
Measured relationship between surface tension, temperature, and p_{O_2} for molten silver²

References:

1. DebRoy, T. and S.A. David, *Physical processes in fusion welding*, Reviews of Modern Physics, 1995, 67(1), p. 85-112
2. Ozawa, S., et al., *Influence of oxygen partial pressure on surface tension of molten silver*, Journal of Applied Physics, 2010, 107.
3. Ozawa, S., et al., *Influence of oxygen partial pressure on surface tension and its temperature coefficient of molten iron*, Journal of Applied Physics, 2011, 109.

Oxygen Sensing

- Potentiometric sensor
 - Determines the difference in oxygen activity in 2 gas compartments separated by an electrolyte
 - Yttria-stabilized zirconia (YSZ)
- Activity of gaseous compounds corresponds closely with their partial pressures
- p_{O_2} is calculated by using the Nernst equation: $E = \frac{RT}{4F} \ln \left(\frac{p_{O_2}}{p_{O_2}^{ref}} \right)$
 - E is the electromotive force, R is the universal gas constant, F is the Faraday constant
 - $p_{O_2}^{ref}$ is the oxygen partial pressure of the reference gas (the lab atmosphere, in this case)
 - p_{O_2} is the oxygen partial pressure of the gas in question
- Must be operated above 500°C to enable sufficient ionic conductivity

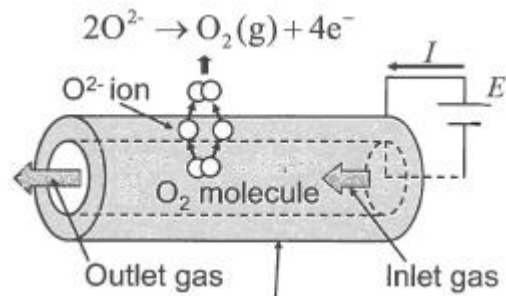


Schematic of oxygen ion sensor

Ref: Schulz, M., et al., *Oxygen partial pressure control for microgravity experiments*, Solid State Ionics, 2012, 225, p. 332-336.

Oxygen Pumping

- Electric current is applied to the electrodes (Pt)
- Charge moved across the electrolyte in the form of oxygen ions, O^{2-}
- Negative electrode
 - Oxygen is incorporated into vacancies of the electrolyte, V_O^{00}
- Positive electrode
 - Oxygen leaves crystal lattice to form gaseous oxygen
- Must be operated above 500°C to enable sufficient ionic conductivity



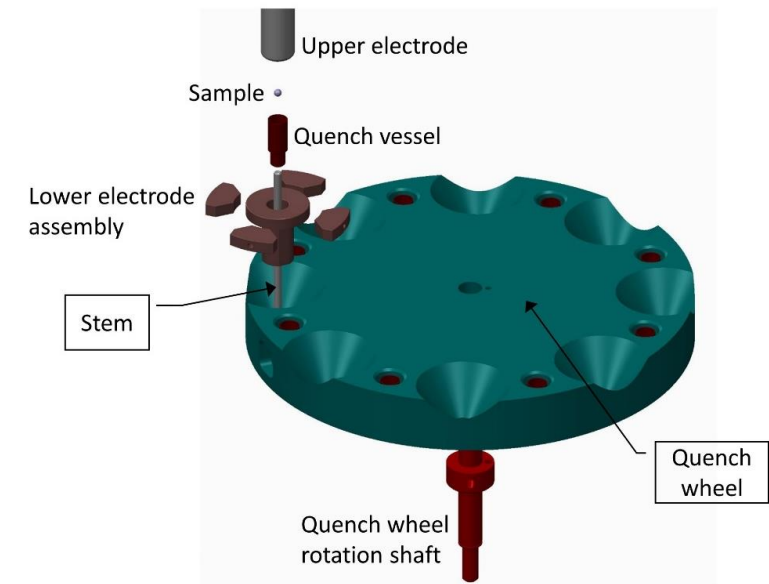
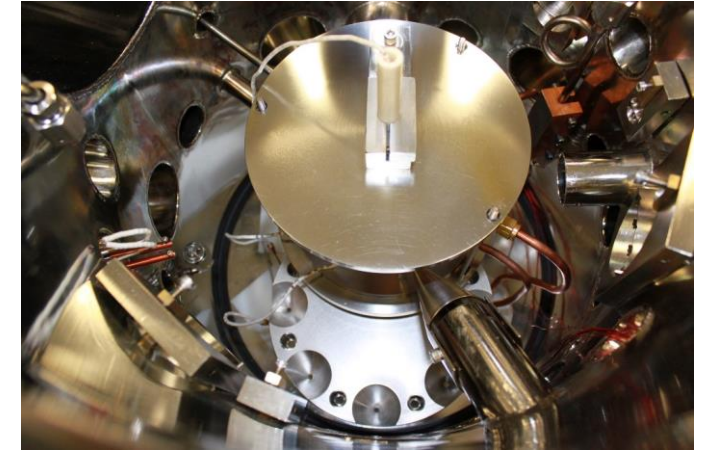
Schematic of oxygen ion pump

Oxygen molecules move through the YSZ tube from inside to outside when a difference in electrical potential is provided between the tube walls.

Ref: Ozawa, S., et al., *Influence of oxygen partial pressure on surface tension of molten silver*, Journal of Applied Physics, 2010, 107.

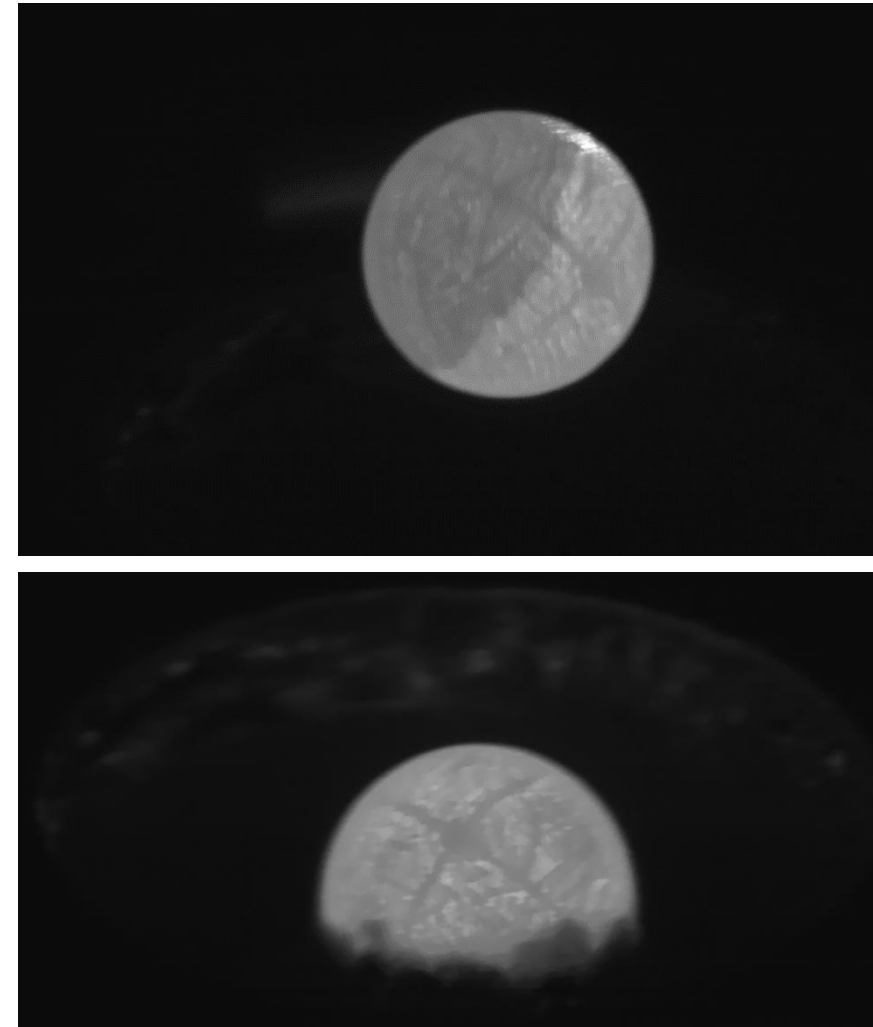
Rapid Quench System

- First submersion quench system inside an electrostatic levitator
- Allows samples to be dropped into a quench vessel that can be filled with a low melting point material, as a quench medium
 - Thereby allowing rapid quenching of undercooled liquid metals
- Quench vessels can be raised or lowered using the same stem that is used to launch samples
- Up to 8 quench vessels can be loaded into the quench wheel
- Wheel is indexed with servo motors that are controlled with LabVIEW software



Motivation

- To preserve transient microstructures for quantitative metallographic analysis
- To freeze-in metastable phases for solidification path determination
- To rapidly solidify reactive melts while minimizing internal fluid flow
- To reduce fragmentation of structures associated with splat quench techniques
- To eliminate coarsening of microstructures to define as-solidified dendrite shape
- To reduce both solid and liquid diffusion processes to observe partitioning in-situ



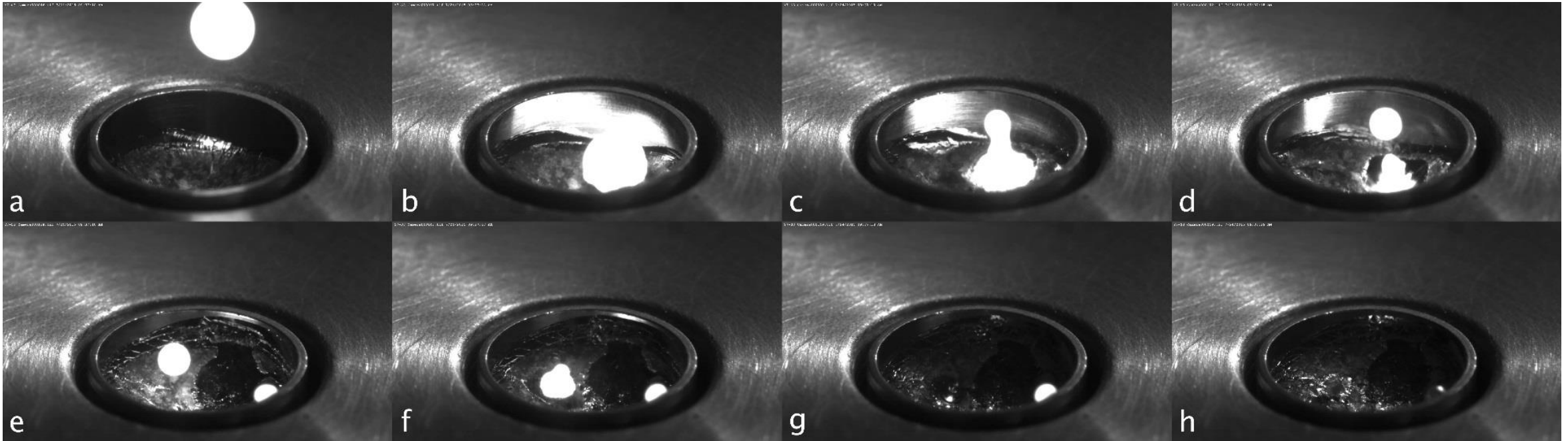
FeCrNi sample quenched after second recalescence

Quench Medium

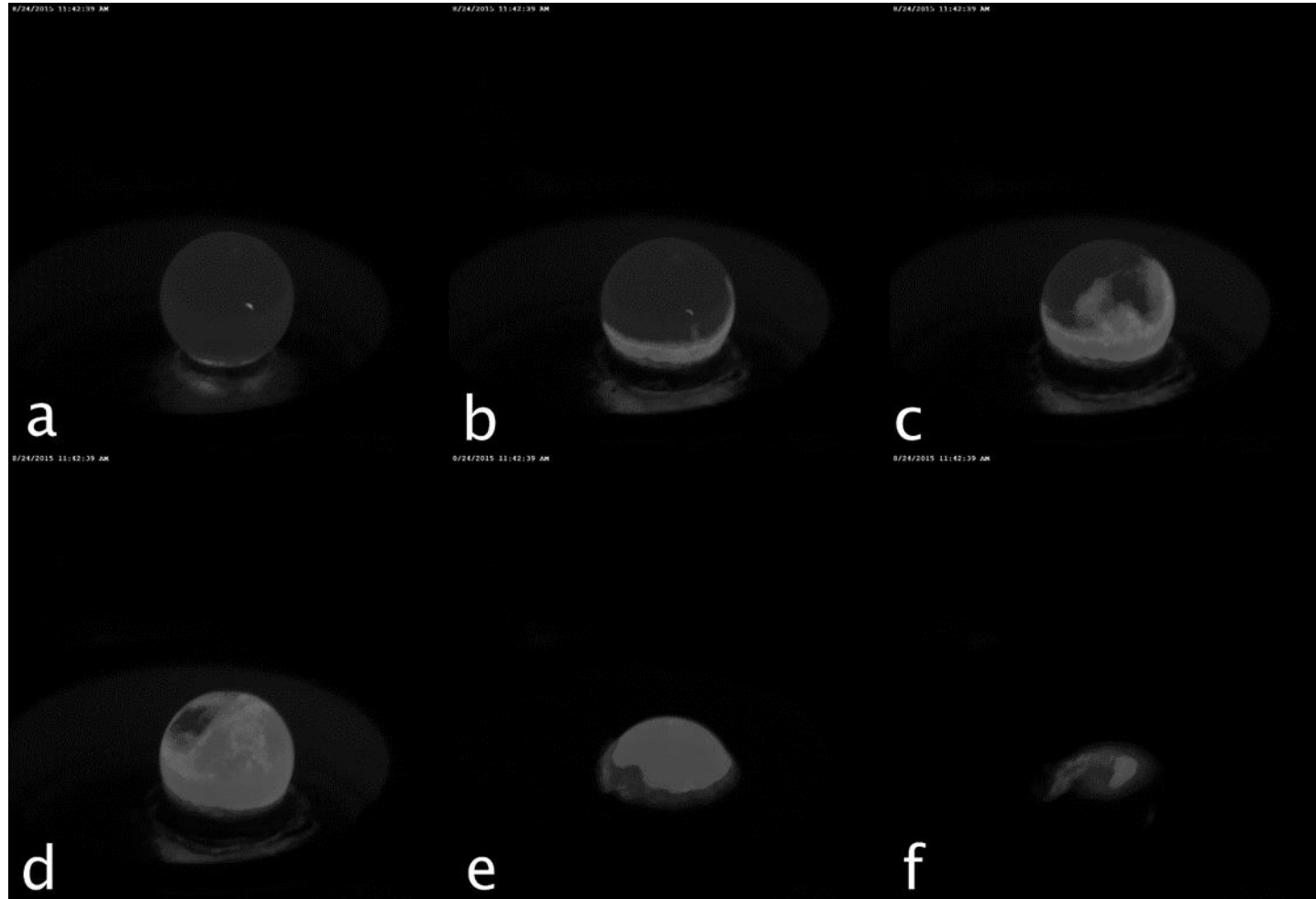
- Indalloy 46L
 - 61.0Ga - 25.0In - 13.0Sn - 1.0Zn
 - Liquidus = 7.6 C
 - Thermal Conductivity = ~15 W/mK (estimated by manufacturer)
- Gallium-Indium alloys have been proposed for similar studies by Koseki and Flemings
 - T. Koseki and M.C. Flemings, "Solidification of Undercooled Fe-Cr-Ni Alloys III: Phase Selection in Chilling", Metallurgical and Materials Transactions A, 28A (11) (1997), 2385-2395.



Quench Sequence – Superheated Zr



Quench Sequence – Undercooled CoSi





Thank you!