

ISS NATIONAL LABORATORY

A Researcher's Guide to:

INTERNATIONAL SPACE STATION

Microgravity Materials Research



This International Space Station (ISS) Researcher's Guide is published by the NASA ISS Program Research Integration Office and was written in collaboration with the ISS National Laboratory[®], which is managed by the Center for the Advancement of Science in Space[™] (CASIS[™]).

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Published: September 2015 Revised: May 2025

Cover:

Scanning Electron Microscope micrographs taken at DLR-Köln of Cannon-Muskegon CMSX-4 Plus (SLS): (a) superalloy droplets solidified after sample preparation on the ground and before levitation testing, (b) after processing in space using the JAXA ELF (Electrostatic Levitation Furnace) on the ISS, and (c) on the ground using the NASA-MSFC Electrostatic Levitation Facility. (Image credit: Douglas Matson, Ph.D., Tufts University)

Back Cover:

Sections of ZBLAN (zirconium barium lanthanum aluminum sodium fluoride) fibers pulled in a conventional 1g process (right) and in experiments onboard NASA's KC-135 lowgravity aircraft (left). The rough surface of the 1g fiber indicates surface defects that would scatter an optical signal and greatly degrade its quality. ZBLAN is part of the family of heavy-metal fluoride glasses. NASA is conducting research on pulling ZBLAN fibers in the low-g environment of space to prevent crystallization that limits ZBLAN's usefulness in optical fiber-based communications. ZBLAN is a heavy-metal fluoride glass that shows exceptional promise for high-throughput communications with infrared lasers. (Image credit: NASA/Marshall Space Flight Center)

The Lab is Open

Orbiting 250 miles above the Earth, the ISS provides a platform for research to improve life on Earth, enable space exploration, understand the universe, and contribute to the development of a low Earth orbit economy. This researcher's guide is intended to help potential ISS materials science researchers plan experiments using the microgravity environment to understand how heat and mass transfer affects materials processing. It covers facilities available for conducting materials science research, provides examples of previous and current microgravity materials research, and discusses promising areas for future materials science research on the ISS.



NASA astronaut and Expedition 69 Flight Engineer Frank Rubio works in the Microgravity Science Glovebox (MSG) swapping graphene aerogel samples for a space manufacturing study. The physics investigation seeks to produce a superior, uniform material structure benefitting power storage, environmental protection, and chemical sensing. Photo courtesy of NASA.



Unique Features of the ISS Research Environment

- Sustained microgravity, or near-weightlessness, alters many observable phenomena within the physical and life sciences. Systems and processes affected by microgravity include surface wetting and interfacial tension, multiphase flow and heat transfer, multiphase system dynamics, melting and solidification, and combustion phenomena. Microgravity induces a vast array of changes in organisms ranging from bacteria to humans, including global alterations in gene expression and 3D aggregation of cells into tissue-like architecture.
- 2. Extreme conditions in the ISS environment include exposure to extreme heat and cold cycling, ultrahigh vacuum, atomic oxygen, and high-energy radiation. Testing and qualification of materials exposed to these extreme conditions have provided data to enable the manufacturing of long-life, reliable components used on Earth as well as in the world's most sophisticated satellite and spacecraft components.
- 3. Unique vantage point in low Earth orbit at 51 degrees inclination, a 90-minute orbit at an altitude of approximately 250 miles (400 kilometers) and an orbital path over 90 percent of the Earth's population. This perspective can provide improved spatial resolution and variable lighting conditions compared with the Sunsynchronous orbits of typical Earth remote sensing satellites.

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Why Use ISS as a Laboratory for Materials Research

The purpose of this *Researcher's Guide to Materials Science Research in Microgravity* is to inform researchers of the benefits of conducting materials science research in microgravity, illustrate some of the impressive scientific investigations being conducted onboard the International Space Station (ISS), and inspire researchers to use the unique microgravity environment to achieve breakthrough science. The guide reflects on some key lessons learned from past materials science investigations, briefly describes the facilities on the ISS that can support materials research, highlights some past and ongoing materials science investigations on the ISS, and discusses research areas critical for the future of materials research in space. Additional important research areas can be found in <u>Thriving in Space: Ensuring the Future of Biological and Physical</u> <u>Sciences Research: A Decadal Survey for 2023-2032</u>, the recent decadal survey released by the National Academies of Sciences, Engineering, and Medicine.

Most materials are formed from a partially or totally fluid sample, and the transport of heat and mass from the fluid into the solid during solidification inherently influences the formation of the material and its resultant properties. The ISS provides a long-duration microgravity environment for conducting experiments that enables researchers to examine the effects of heat and mass transport on materials processes in the near-absence of gravity-driven forces. The microgravity environment greatly reduces buoyancy-driven convection, hydrostatic pressure, and sedimentation. It can also be advantageous for designing experiments with reduced container interactions. The reduction in these gravity-related sources of heat and mass transport may be taken advantage of to determine how material processes and microstructure formation are affected by gravity-driven and gravity independent sources of heat and mass transfer.

Materials science experiments on the ISS have yielded broad and significant scientific advancements, including contributing to the development of improved mathematical models for predicting material properties during processing on Earth and enabling a better understanding of microstructure formation during solidification towards controlling the material properties of various alloys. As the world's premier orbiting laboratory continues operations through the end of the decade (2030), the quantity and quality of materials research in space and the resulting benefits back to humanity are expected to continue to increase.

Materials Science Research in Microgravity: Lessons Learned

Experiments have been conducted on the ISS for more than two decades. Materials science research has been at the forefront of these efforts. As research on the ISS continues over the next several years and as microgravity research transitions to new space station platforms, it is important to reflect on the lessons learned from past experiments and research efforts.

Materials science research on the ISS has been fruitful in understanding key issues in working with materials in microgravity. A major focus has been on the investigation of fluids where the effects of sedimentation, buoyancy-driven convection, and density-driven motion can be effectively decoupled from diffusion-controlled processes. These conditions enable the exploration of novel non-equilibrium effects as well as the investigation of practical issues related to processing materials in reduced gravity. Listed below are some of the key lessons learned from past materials science investigations:

- Experiment results have shown the need for replicate measurements. Are observed phenomena merely isolated effects, or do they demonstrate a fundamental difference between Earth and microgravity-based processes?
- Performing experiments in reduced gravity is complex. Crew time and launch vehicle transportation are scarce resources that must be managed effectively.
- The ability to perform at least some sample characterization in orbit would accelerate research; for example, by guiding decisions on sample reuse without needing to transport them to and from Earth. *In situ* analyses can enable iterative science.
- Complementary ground-based experiments are essential. Many characterization tools are easier to use or are only available on Earth, and these tools help maximize the value of experiment data collected in microgravity.
- Modeling can assist in optimizing experiments and in interpreting results.
- Due to the absence of buoyancy forces, surface forces tend to dominate. This can become problematic for bubble formation and handling. Therefore, bubble mitigation strategies should be developed in advance.

ISS Facilities for Materials Science Research

The following sections cover accommodations and facilities used to support microgravity materials science experiments on the ISS.

Logistics, Operations, and Accommodation of Flight Experiments

Launch vehicles deliver the required experiment equipment and samples to the ISS. The experiments are conducted on the ISS using a combination of crew control, autonomous pre-programmed operations, and/or ground command of the hardware. Most of the internal payloads carried by launch vehicles operate in an ambient environment, but refrigerators, freezers, and incubators are available for temperature-sensitive payloads.

Onboard the ISS, a host of research facilities are available for materials science research. Below is a summary of many of the facilities that may be used for materials science research. Additional updated information on ISS research facilities and past investigations on the ISS are available using <u>NASA's Space</u> <u>Station Research Explorer</u>.

Additive Manufacturing

BioFabrication Facility (BFF)

The BFF is operated by Redwire Space Technologies and was developed by Techshot to print organ-like tissues in microgravity, acting as a steppingstone in a long-term plan to manufacture whole human organs in space. The BFF prints a fluid bioink through an extrusion-based printing process. After the tissues are printed, they are placed in bioreactor cassettes for several weeks, allowing the tissues to cohesively form on a cellular level.



Figure 1. A photo of the commercially developed and operated BioFabrication Facility (BFF). Image credit: NASA

Furnaces

Several furnaces that cover a variety of temperature ranges and ambient conditions are available onboard the ISS. These furnaces have allowed researchers to study the microstructural features of materials during melting/ solidification for applications such as casting, welding, soldering, and additive manufacturing (Heider and Pesquet, 2022). The furnaces are described briefly below.

Solidification Using a Baffle in Sealed Ampoules (SUBSA) Hardware

The SUBSA furnace was developed for operation in the ISS Microgravity Science Glovebox (MSG). The furnace provides for solidification of samples via the gradient freeze technique. The SUBSA furnace can be used for semiconductor or low melting point metal crystal growth with a maximum heater temperature of 850°C and maximum thermal gradient of 110°C/cm, depending on the sample. Samples are contained in sealed glass ampoules with a maximum sample length of 30 cm and outer diameter of 12 mm. The transparent furnace zone of 8 cm allows imaging of the crystallization process. The "baffle" in SUBSA allows for additional control and suppression of the melt motions near the growing crystal-melt interface beyond what is accessible through reduced gravity alone. An annotated cross-sectional view of the SUBSA hardware is seen in Figure 2.



Figure 2. Cross-section of the SUBSA furnace. The sample is immediately to the left of the quartz plug. The baffle assembly is to the left of the sample. Image credit: NASA

Pore Formation and Mobility Investigation (PFMI) Hardware

The PFMI apparatus was also developed for operation in the ISS MSG. PFMI hardware allows the observation of directional solidification of transparent samples and allows the injection of bubbles into the melt. Examples of solidification physics that may be studied include bubble dynamics, tracer particle movement induced by gravity-independent thermocapillary forces, and planar front to dendrite transitions.

The solid/liquid interface, or some other region of interest, is observed and recorded by two solid state cameras offset by 90 degrees. The temperature range of the hardware is approximately 5 to 120°C. Gradient freeze and isothermal experiments are also possible. Figure 3 shows an image of the ground PFMI hardware.



Figure 3. Labeled photo of the PFMI ground hardware. Image credit: NASA

The sample ampoule is constructed of Dow Corning 7052 borosilicate glass. A stainless-steel spring with a Kovar piston allows for thermal expansion and contraction of the sample material. A thin, transparent layer of Indium Tin Oxide (ITO) is deposited on the exterior surface of the Sample Ampoule Assemblies (SAAs). The ITO coating is electrically conductive and acts as a resistance heater when current is applied. To process the sample, a cold zone using four Thermal Electric Devices (TEDs) is translated along the sample together with the forward electrode ring. As the cold zone and electrode ring are translated, the hot zone shortens, and heat is removed from the sample to the radiators by the cold zone. Additional thermal capabilities are possible by using an independently heated zone melting ring that is placed near the cold end.

Coarsening of Solid-Liquid Mixtures (CSLM) Hardware

CSLM hardware is also operated in the MSG. CSLM consists of an Electronics Control Unit (ECU), which functions as the Data Acquisition and Controller and the Sample Processing Units (SPUs) that house the samples to be processed. The function of the ECU is to control the heaters in the SPU and to maintain the required temperature up to 185°C for a specific period of time, at which point, the ECU commands the SPU to quench cool the samples using ambienttemperature water. The SPU houses the four samples for processing. To reduce the convective heat transfer, the SPU can be evacuated to approximately 10-6 Torr on the ground prior to launch as well as drawing down the pressure just before processing using the ISS Vacuum Exhaust System. The SPU consists of resistive heaters and RTDs to monitor the temperature and provide closed-loop control signals back to the ECU. Each location is measured exactly to provide investigators with the information required to size their samples such that when heated to maximum temperature, the samples encompass no more than the available volume.

Levitation Furnaces

Traditional techniques for the study of molten materials involve the use of containers such as crucibles. Containerless levitation techniques eliminate the need for containers during the test while reducing the risk of sample contamination. This capability provides the ability to process a wide range of metals, alloys, oxides, and semiconductors, both above and below their melting temperatures.

Electromagnetic and electrostatic levitation are the two most widely used containerless levitation techniques for materials science research in microgravity. Electromagnetic levitation uses an alternating electromagnetic field within a water-cooled copper coil to levitate a diamagnetic sample. The Lorentz force is induced by the eddy current, and the magnetic field helps position the sample to the center of the coil. The Ohmic loss of the eddy currents simultaneously heats the sample and creates internal liquid convection. Electrostatic levitation (ESL) technology involves positioning a charged sample between two horizontal electrodes that produce a large electric field gradient. The size of the sample is limited by a threshold value of charges applied to the sample surface in normal gravity conditions to minimize electrode arcing. However, in microgravity, the Coulombic forces are mainly derived from induced charges, and the applied electric field allows a relatively larger sample to be processed at lower field strength with reduced potential for arcing compared with terrestrial levitators.

The ISS currently hosts two main levitation facilities that can cater to metals and alloy synthesis: ESA's ISS-EML and JAXA's (Japan Aerospace Exploration Agency's) Electrostatic Levitation Furnace (ELF). The MSL-EML facility uses electromagnetic levitation to process conductive samples in high vacuum or gas environments with a wide range of applied convective conditions spanning the laminar to turbulent regimes, while the ISS-ELF facility uses electrostatic levitation to process materials including high-temperature oxides with limited (to zero) stirring in a gas environment. Both facilities offer the unique capability of extended reduced gravity experiment duration to allow for completion of thermophysical property measurement and phase transformation kinetics observation objectives.

Electrostatic Levitation Furnace (ELF)

ISS-ELF is an Electrostatic Levitation Furnace developed by JAXA for materials science investigations on the ISS. The ELF facility is currently installed in the Multi-Purpose Small Payload Rack 2 (MSPR-2) within the KIBO module. This facility accommodates advanced investigations in the microgravity environment of the ISS for basic materials science research in areas such as property measurement or solidification of metals, alloys, semiconductors, and oxides. The main body of ISS-ELF consists of an experiment controller, a positioner controller, and a processing chamber that houses the optical devices, heating lasers, cameras, and pyrometer. The sample cartridge is shown in Figure 4(b) along with the sample holder, which can contain up to 20 test samples.



Figure 4. (a) Molten metal oxide sample levitated in the ELF and (b) Sample cartridge and sample holder for material processing. Image credit: NASA

The sample cartridge also contains a cubic array of six electrodes between which a sample is levitated. Because there is no strong g-vector in microgravity, the size of these six electrodes is identical and the applied field is low compared with ground testing. The facility also has two pushing rods: the upper rod helps with sample insertion in the beginning of the experiment, and the lower rod carries the sample back to the holder after the experiment. The sample cartridge is inserted into the chamber by crew members, and operations are conducted from the ground through commands issued from the JAXA Tsukuba Space Center. The sample is heated using four identical lasers, each positioned at a 90-degree angle from each other in a tetrahedral array to ensure homogeneous heating and to minimize the potential for developing significant Marangoni flow. Table 1 provides an overview of the operating parameters of this facility.

ISS-ELF Capabilities and Critical Performance Parameters				
Sample Materials	High-temperature ceramics, oxides, semiconductors, and metallic alloys Samples returned to ground for post-mission analyses			
Sample Size	Spherical 1.5~2.3 mm DIA			
Ambient atmosphere	400 mbar Argon gas (maximum chamber pressure 2 atm) or vacuum through vent line (Pvac better than 1 mbar)			
Positioning Control	Three-axis control frequency: max 1 kHz, Absolute positioning accuracy: ± 300 μm			
Heater Control	Semiconductor laser: 980nm wavelength / 40W power x 4 directions Laser assembly arranged in tetrahedral alignment to minimize convection			
Observation	Temperature measurement: IR pyrometer InGaAs 1.45 – 1.80 μm with Tmax = 3000°C Field of view: 0.35 mm DIA spot size at 100 Hz			
	Observation of overview (for position relative to linear array): CCD camera Camera 1: 19x14 mm (zoom 2.4x1.8 mm) 640x480 pixels at 60 Hz Camera 2: 24x18 mm (zoom 3.0x2.3 mm) 640x480 pixels at 100 Hz			
	Linear array 4 mm length with 1x256 pixels at 1 MHz (laser shadow mode)			
Techniques for Measurement of Properties	Density: Image Analysis of CCD cameras and linear array			
	Surface Tension: Oscillating droplet method using linear array High Speed/High Voltage Amplifier voltage: ± 0.6 kV to ± 3 kV sinusoidal pulses applied to positioning field to excite oscillations 50-500 Hz, with resulting desired deformation amplitude of 2%, 5%, and 10%			
	Viscosity: Oscillating droplet decay using linear array			

Table 1. ISS ELF Facility Attributes

Because the heating lasers are arranged in a tetrahedral arrangement, induced Marangoni convection can be suppressed, and sample stirring during melting and subsequent cooling is effectively eliminated.

ElectroMagnetic Levitator (EML)

The ISS-EML is a facility jointly developed by ESA and the German Space Agency (DLR) and installed in the European Laboratory, Columbus. This facility accommodates investigations focusing on solidification and undercooling of metallic materials such as pure elements, compounds, and commercially important alloys. Figure 5(a) provides a view of the front panel of the ISS-EML facility. Figure 5(b) shows the inside of the processing chamber with the copper levitation and heating coils located at the center and viewing ports for instrumentation access around the outer periphery to allow video imaging and temperature measurement using optical pyrometry. In Figure 5(c), the ceramic holder is raised into the coils for sample processing: the holder contains a wire cage that limits the sample motion during periods when the electromagnetic field is turned off.



Figure 5. (a) ESA astronaut Alexander Gerst is shown installing the ISS-EML facility (photo courtesy of ESA), (b) levitation coils within the processing chamber (photo courtesy of Airbus), and (c) wire cage sample holder used to keep the sample within the coil center. Photo courtesy of Airbus.

Below the sample processing chamber is a removable sample cartridge that contains a rotating carousel that holds 18 individual samples within their respective ceramic holders. Any sample motion is dampened by slightly raising and lowering the holder. Because the sample stays at coil-center, this positioning moves the bottom of the holder until it contacts the sample to stop any unwanted translation or rotation. The sample can be heated by applying a superimposed heating field to the coil. Cooling is accomplished by turning this heating field off. During testing, if stirring or if a thermal hold is required, the heating field may be turned down only partially to achieve the desired experiment conditions. Because heating and positioning of the sample are not independent, it is not possible to eliminate all convection in the sample during processing. This limitation can be seen as a significantly important asset since convection can be controlled to the desired level, and the influence of stirring on transformation kinetics may be studied. In addition, the inductive response of the sample may be monitored throughout the test using the Sample Coupling Electronics (SCE) hardware to track changes in resistivity as a function of temperature or phase composition.

ISS-EML Capabilities & Critical Performance Parameters				
Sample Materials	Pure metallic elements and metallic alloys Samples returned to ground for post-mission analyses			
Sample Size	Spherical 6-10 mm DIA			
Ambient atmosphere	350 mbar 6N purity Helium or Argon gas or UHV turbopump vacuum (P_{vac} better than 10 ⁻⁸ mbar)			
Positioning Control	150 kHz quadropole electromagnetic field from water-cooled Cu coil			
Heater Control	350 kHz dipole electromagnetic field from water-cooled Cu coil			
Observation	Temperature measurement: IR pyrometer InGaAs $1.45 - 1.80 \mu m$ with $T_{max} = 2,100^{\circ}C$ Field of view 0.8 mm DIA spot size at 100 Hz			
	Axial Camera 1: 10x10 mm 352x352 pixels at 150 Hz Radial Camera 2: 10x10 mm 256x256 pixels up to 60 kHz			
Techniques for Measurement of Properties	Density: Image Analysis of radial or axial camera digital images			
	<i>Surface Tension</i> : Oscillating droplet method through application of a single heater pulse to excite oscillations 20-100 Hz with deformation to 10%			
	Viscosity: Oscillating droplet decay following single heater excitation pulse Specific heat: Modulation calorimetry Electrical conductivity: Sample Coupling Electronics (SCE) module Sample quench plate for directional chill casting			

Table 2.	ISS-EML	Facility	Attributes
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General Purpose Facilities

Microgravity Science Glovebox (MSG)

The Microgravity Science Glovebox (MSG), shown in Figure 6, is a rack-level payload facility that provides a safe, contained environment for crew members to conduct research. The MSG has power, data, video, heat rejection, vacuum, and nitrogen available for investigations. It can be used to facilitate complete investigations or to test small parts of larger investigations. For example, the MSG is used to house experiments that use the SUBSA, PFMI, or CSLM hardware.



Figure 6. NASA astronaut Nick Hague conducting research operations in the Microgravity Science Glovebox (MSG). Image credit: NASA

KEyence Research MIcroscope Testbed (KERMIT)

KERMIT is a commercial off-the-shelf fluorescence microscope (Keyence model number BZ-X800E) available aboard the ISS and currently housed in the MSRR-1. Imaging can be conducted through fluorescence, brightfield, phase contrast, or oblique illumination. Objective lenses of 2x, 4x, 10x, 20x, 40x, and 60x are available, and multiple filters are also available. KERMIT observation modes include image cytometry, Z-stack imaging, image stitching, sectioning, time-lapse, and video recording. KERMIT can be operated by space station crew members or remotely by the ground team or PI. The ground analog of the KERMIT is located at NASA's Marshall Space Flight Center (MSFC) in Huntsville, Alabama.

Mochii Scanning Electron Microscope (SEM)

Mochii is a miniature Scanning Electron Microscope (SEM) with spectroscopy aboard the ISS that is operated by Voxa and can conduct real-time imaging and compositional measurements of particles through energy-dispersive X-ray spectroscopy (EDS). Mochii serves two purposes: 1) supporting crew and vehicle safety by rapidly identifying crew- and vehicle-threatening particles and 2) conducting novel microgravity science tasks by serving as a unique commercial platform for microgravity research in low Earth orbit.



Figure 7. NASA astronaut Kayla Barron setting up the Mochii Scanning Electron Microscope (SEM). Image credit: NASA

Modular Multiuse Facilities

The ISS is home to many general-purpose research facilities that make it possible to conduct a wide range of materials research investigations. These facilities are often modular and flexible, yielding a wide range of options for experiment setups.

ADvanced Space Experiment Processor (ADSEP)

ADSEP, operated by Redwire Space Technologies and designed by Techshot, is a thermally controlled single-locker processing facility that interfaces with an ISS middeck EXPRESS Rack. ADSEP provides power and data and can house up to three cassette-based experiments that can be independently operated. The temperature can be independently controlled for each cassette between 4°C and 40°C. ADSEP cassettes have been used for materials research such as microencapsulation and crystal growth.



Figure 8. Photo of a Techshot fluid processing cassette designed for installation in the ADvanced Space Experiment Processor (ADSEP). Image credit: NASA

TangoLab

The Space Tango MultiLab Locker (TangoLab) is a general research facility aboard the ISS that provides a standardized platform and open architecture for experimental modules called CubeLabs. This standard platform reduces the development cycle and cost for research and development (R&D) payloads. TangoLab provides power and near-real-time data streaming. The CubeLabs are installed on orbit and are easily reconfigurable. They are designed to be autonomous after being installed by the crew.



Figure 9. NASA astronaut Mark Vande Hei swapping out a payload card from a TangoLab facility. Image credit: NASA

International Commercial Experiments (ICE) Cubes

The ICE Cubes facility is a general research facility that provides flexibility for R&D experiments onboard the ISS. During flight, users have access to nearreal-time telemetry and tele-commanding capabilities with the experiments from any location with an internet connection. The ICE Cubes facility framework can accommodate up to 20 experiment cubes and provides power and data. Each cube can be either a standard (1U) cube size (10 cm x 10 cm x 10 cm) or modular combinations of that size (e.g., up to 4U x 3U, which is 45 cm x 35 cm x 11 cm). The cubes provide forced air cooling.



Figure 10. A photo of the ICE Cubes facility onboard the ISS. Image credit: NASA

Nanoracks Platforms

Nanoracks Platforms is a multipurpose research facility that supports Nanoracks Modules by supplying power and data to operate investigations onboard the ISS. Nanoracks Platforms are 17 in x 9 in x 20 in and are installed into a single locker EXPRESS Rack. Each Nanoracks Platform can accommodate 20 Nanoracks Modules (4 in x 4 in x 4 in), each of which hosts a different experiment. Crew interaction with Nanoracks Platforms is limited to installation and activation into the EXPRESS Rack locker.



Figure 11. A photo of a NanoLab in the Cupola window onboard the ISS. Image credit: NASA

Microgravity Research for Versatile Investigations (MaRVIn)

The MaRVIn hardware, operated and designed by Tec-Masters, Inc., is another general research processing system for operating investigations aboard the ISS. MaRVIn uses interchangeable experiment modules that offer heating, cooling, internal fluid and chemical management, power, data, and video. The hardware supports sample processing from 0°C to 1,000°C. MaRVIn can be operated in the MSG, the MWA, or the Life Science Glovebox (LSG).



Figure 12. A preflight image of the Microgravity Research for Versatile Investigations - Phase Change in Mixtures (MaRVIn-PCIM) system as it will be housed in the MSG. Image credit: NASA

Additional multiuse facilities on ISS with capabilities to support materials science research include DECLIC (Device for the Study of Critical Liquids and Crystallization), developed by the French Space Agency (CNES), and SABL (Space Automated Bioproduct Laboratory), developed by BioServe Space Technologies.

Past/Ongoing Microgravity Materials Science Research

Microgravity materials science research began during the Apollo Program with directional solidification experiments involving semiconductor materials. During Apollo 14, a small, low-temperature furnace was used to cast or directionally solidify a number of two- and three-phase systems. It was found that the dispersion of the phases was much more uniform because of the lack of sedimentation. Prior to this, the first welding experiment in space was conducted during the Soyuz 6 mission using the Vulkan system to test different techniques (low pressure compressed arc, electron beam, and arc with a consumable electrode). Electron beam was the most successful.

More sophisticated experiments were conducted on Skylab involving directional solidification of semiconductor crystals and eutectic materials, brazing, containerless processing to form spheres, and more.

This research continued during the Space Shuttle Program. One notable experiment from the early shuttle flights involved the creation of the first commercial material formed in space. Spherical standards were created from an emulsion of styrene monomers. This emulsion was unstable under normal 1g conditions because of sedimentation but was quite stable in microgravity. The emulsified styrene monomers were polymerized in orbit to form highly uniform spheres (Vanderhoff et al., 1984).

Materials science research in microgravity is now in the ISS era. Initial materials science research experiments conducted on the ISS included Coarsening in Solid-Liquid Mixtures (CSLM), Solidification Using a Baffle in Sealed Ampoules (SUBSA), Toward Understanding Pore Formation and Mobility During Controlled Directional Solidification in a Microgravity Environment, In-Space Soldering Investigation (ISSI), Comparison of Structure and Segregation (CSS) in Alloys Directionally Solidified in Terrestrial and Microgravity Environments, and Dynamic Selection of 3D Interface Patterns in Directional Solidification. Descriptions of these experiments, and all since then, can be found using NASA's Space Station Research Explorer. Additionally, there are several resources available for accessing microgravity research data, including full experimental data on many materials science investigations through the <u>NASA</u> Physical Science Informatics (PSI) database and microgravity crystal growth data through the <u>Microgravity Crystal Database</u> created by Dr. Anne Wilson at Butler University (Wright et al., 2022).

Over the past two decades, many more materials science experiments have been conducted on the ISS. Examples of past and ongoing microgravity materials science research aboard the ISS are briefly described below for glasses and ceramics, metals, multiphase materials, nanomaterials, polymers, semiconductors, and thermophysical properties.

Glasses & Ceramics

ZBLAN Optical Fiber Production

There has been a significant amount of research in the production of ZBLAN (zirconium barium lanthanum aluminum sodium fluoride) optical fibers in microgravity. Studies have shown that the lack of gravity-induced forces in microgravity significantly reduces the quantity of defects in ZBLAN fibers manufactured in space, significantly improving the resulting properties. ZBLAN optical fibers are used for optical fiber-based communications and show great promise for high-throughput communications with infrared lasers. Figures 13 and 14 show results of ZBLAN fibers manufactured in microgravity versus those manufactured on Earth under similar conditions.



Figure 13. Sections of ZBLAN fiber pulled during a microgravity parabolic flight (left) and during ground-based 1g manufacturing process (right). The rough surface/defects seen in the 1g manufactured fiber would greatly degrade the quality of the fiber by scattering the optical signal. Image credit: NASA/Marshall Space Flight Center ZBLAN is part of the heavy metal fluoride glass group. Its composition is typically 53% \underline{Z} rF4, 20% \underline{B} aF2, 4% \underline{L} aF3, 3% \underline{A} IF3, and 20% \underline{N} aF.



Figure 14. Scanning electron microscope (SEM) images of the surface of ZBLAN fibers pulled in microgravity (bottom) and in 1g during a typical ground-based manufacturing process (top). The crystallization and defects that occur during ground-based manufacturing result in scattering of the optical signal, significantly degrading the quality of the fiber. Image credit: NASA

Metals

Probing Phenomena Hidden by Gravity

For the investigation of metals, microgravity testing is used to provide enhanced fidelity of results purportedly due to three main effects. First, sedimentation and buoyancy-induced segregation are eliminated in reduced gravity. Second, without strong gravitational accelerations and with reduced levitation forces, containerless processing produces a more spherical sample, which allows for better analysis of experimental behavior. Third, better control of convection in space results in higher measurement precision. Experiments conducted on the ISS complement both ground-based experiments and other microgravity platforms such as parabolic flight and sounding rocket experiments. Figure 15 shows how a steel sample solidifies when intentionally put into contact with a chill plate affixed to the top of the sample holder. On Earth, gravity-related phenomena dominate with buoyancy-driven flow and severe thermomechanical deformation. During directional solidification in orbit, the deformation shrinkage is dominated by surface tension forces, allowing investigation of behavior relevant to modeling optimization of additive manufacturing processes.



Figure 15. Evolution of structure during chill casting showing (a) a sequence of images of a Fe-0.9C-0.2Si steel solidifying in microgravity and (b) modeling of the dynamics and thermal history of the resulting solidification front. Figure reproduced from Matson et al., 2023.

In microgravity, buoyancy and sedimentation may be minimized, enabling researchers to investigate de-mixing of phases. In Figure 16, the miscibility gap in a Co-Cu alloy can clearly be seen as the two liquid phases separate in a manner similar to what is observed when mixing oil and water. On Earth, the difference in density between the two phases causes severe sedimentation that makes analysis difficult.



Figure 16. Miscibility gap in the Co-Cu system. (a) The binary phase diagram with thermodynamic predictions on phase composition (lines) and experimental verification (points). (b) Backscatter scanning electron microscope image of a levitated sample showing solidification following phase separation of two liquids with the arrow showing small, entrained droplets of Cu-rich liquid within the darker Co-rich second phase. Figure reproduced from Matson et al., 2023.

SUBSA Brazing of Aluminum alloys IN Space (SUBSA-BRAINS)

The SUBSA-BRAINS investigation examined the differences in capillary flow, interface reactions, and bubble formation during the solidification of brazing alloys in microgravity. Brazing is a mechanism to bond similar or dissimilar materials by the inclusion of a molten braze interlayer. Bonding occurs when the liquid phase solidifies, and the formation of a metallurgical bond occurs at the interface. Thus, both the solid state of the mating surfaces and the liquid state of the molten interlayer metal have dominant roles in the process. The data obtained from the SUBSA-BRAINS investigation led to a better understanding of the capillary flow features of the brazing alloy and the extent of weakening of the brazing joint due to voids. The investigation studied three types of aluminum brazing samples named HOLE, PIN, and HOLLOW PIN. The samples were integrated into ampoules and processed in the SUBSA facility. The results of this work have been published (Gruzd et al., 2022).

Effect of Convection on the Columnar-to-Equiaxed Transition in Alloy Solidification (SUBSA-CETSOL)

The SUBSA-CETSOL investigation examined the effects of gravity on the columnar-to-equiaxed transition (CET) in the grain structure of metals. The grain structure forms while molten metal that is poured into a mold solidifies. CET occurs during metal alloy solidification when equiaxed crystals block the growth of elongated columnar crystals. The lack of buoyancy-driven convection and sedimentation in microgravity prevents any movement of the melt and unattached solid during solidification, allowing researchers to gain an improved understanding of CET and improve predictions of the grain structure in industrial metal alloys. A range of aluminum alloys were investigated. The samples were contained in ceramic ampoules. For operations, the samples were melted and then cooled at a pre-determined rate to solidify the alloy. The results of this investigation have been published (Williams and Beckermann, 2023).

Multiphase Materials

Advanced Colloids Experiment-Temperature Control and Gradient Sample-11 (ACE-T-11)

ACE-T-11 investigated the assembly of complex 3D structures from colloids. New functional materials were created using colloids that self-organize into crystalline structures or amorphous glass phases due to entropic forces or the control of a non-equilibrium driving force such as a temperature gradient. The phase behavior of the micron-sized colloidal spheres, ellipsoids, and mixtures was studied using tunable density and depletion interaction gradients. Particle number density and interaction change with distance due to the temperaturecontrolled sample cell. Under certain conditions, the colloids should selfassemble into crystals or dense amorphous glass phases, determined by size, colloid number density, and the concentration of the depletant. Particle interactions are observed using confocal microscopy. Studying these phenomena in microgravity allowed researchers to eliminate disturbances from buoyancyinduced convection and sedimentation. The results of this investigation have led to an improved understanding of the formation of colloidal crystals, which is important for advanced optics, additive manufacturing, communication technologies, and photonic materials (Lei et al., 2024).

Advanced Colloids Experiment-Heated-2 (ACE-H-2)

ACE-H-2 was similar to ACE-T-11; however, in ACE-H-2, the driving force for self-assembly of the colloids was the nanoparticle haloing phenomena (NPH). NPH is the interaction of small nanoparticles with larger colloids. NPH is significantly hindered on Earth due to sedimentation and the density disparity of the particles. Microgravity alleviates these concerns, and the unobstructed NPH interactions should result in self-assembly of the colloids into colloidal crystal structures. The effect of particle concentration was studied. The results of ACE-H-2 have been published (Cecil et al., 2022) and have improved our understanding of self-assembling structures for applications such as photonic crystals.

The microgravity environment of ISS enables new opportunities for materials science research in the emerging field of colloidal engineering of new materials.

Nanomaterials

Plasmonic Bubble Enabled Nanoparticle Deposition Under Microgravity

One of NASA's academic partners is conducting a synergistic program of theoretical modeling, microgravity experiments on the ISS, terrestrial experiments, and a technology demonstration in order to understand the fundamental mechanism of the deposition of suspended nanoparticles (NPs) due to multi-scale plasmonic bubble behavior and the surrounding flow phenomena. Findings promise to guide the development of techniques for fabricating high fidelity sensors by leveraging plasmonic hotspots of closely packed plasmonic NPs.

It is expected that the suspended NPs in solution are driven to the surface due to optical forces, which then work as seeds for the surface bubble nucleation (Fig. 17a); the fluid flow surrounding the surface bubble in the suspension drives the suspended particles (e.g., NPs and analytes) to the three-phase contact line (TPCL) of the bubble (Figure 17b). Turning off the laser to allow the bubble to shrink until it vanishes enables the deposited NPs at the TPCL to contract along with the contact line to form a highly concentrated deposit on the surface (Figure 17c-d). Researchers can leverage this shrinking surface bubble deposition (SSBD) process to reduce the sensing limit of detection (LOD) by using spots of highly concentrated deposits and the surface-enhanced Raman spectroscopy effect that is enabled by plasmonic NPs. In this project, the microgravity environment on the ISS provides a unique platform for the research team to unlock the fundamental mechanism of the SSBD process due to the following reasons: (1) the lack of thermal convection allows the team to better observe whether it is the optical force that drives NPs to the surface and then nucleates the surface bubble; (2) the lack of buoyancy allows the surface bubble to grow larger without detachment so that the bubble surface collects more NPs from the suspension, leading to denser NP deposition and potentially interesting packing orders; (3) the lack of thermal convection enables the team to focus on the thermal Marangoni effect, which results in flow against the thermal convective flow, to understand the respective roles in driving NPs to the bubble surface and the TPCL.



Figure 17. Schematics of (a) optical pressure force driving suspended NPs to the surface;
(b) laser-generated photothermal bubble and the flow surrounding it drives suspended NPs to the three-phase contact line (TPCL); (c) laser turned off to allow bubble shrinking, which leads to TPCL contraction; and (d) concentrated NP island deposited by SSBD due to the complete contraction of TPCL as the bubble vanishes. Inset in (d) is a representative scanning electron microscopy image of the SSBD spot. Image credit: Dr. Tengfei Luo

Results of these ISS experiments are compared with their terrestrial counterparts to elucidate the thermofluid mechanisms involved in the surface bubble formation and the SSBD process.

Biomimetic Fabrication of Multi-Functional DNA-Inspired Nanomaterials via Controlled Self-assembly in Space

This investigation evaluates the in-space manufacturing of Janus base nanomaterials that mimic DNA. Janus base units are small molecules that selfassemble into nanotubes (by hydrogen bonding and base stacking) and mimic DNA base pairs. The Janus base nanotubes (JBNt) are modified with a positively charged amino acid side chain and further assemble to incorporate RNA between their bundles to form Janus base nano pieces (JBNp). Current nanomaterial technologies (e.g., lipid nanoparticles, carbon nanotubes, polymers) are limited and not ideal for biomedical applications due to high toxicity, low bioavailability, low stability, and low biocompatibility. Janus base nanomaterials overcome these challenges; however, manufacturing them on Earth is difficult. This investigation aims to study the in-space manufacturing of Janus base nanomaterials for drug delivery of RNA therapeutics.

Polymers

Coalescence of Water-Based Latex Polymers

This investigation tested the way polymers form on a molecular level in microgravity compared with their formation in 1g. Specifically, the investigation tested the coalescence of polymers at different glass transition temperatures. The coalescence process involves evaporating water from a solution, resulting in closely packed layers of polymer particles that intertangle and form a strong film. The microgravity-formed and Earth-formed polymer films were compared using SEM images. Polymers are used in everyday life on Earth, from plastics to clothing to insulation. A better understanding of polymer formation can lead to more durable and resilient polymers for both Earth and space applications.

Determining the Efficacy of Bacteria-Resistant Polymers in Microgravity

This investigation examines the effects of microgravity on bacteria-resistant polymer materials. Biofouling is a significant contributor to the degradation of materials in an isolated environment. Bacteria-resistant, or non-fouling, materials are tested in this investigation to determine their efficacy in space. Two non-fouling materials are studied: [2-(aryloyloxy)ethyl] trimethylammonium chloride and 2-carboxyethyl acrylate (TMA/CAA) and [2-(aryloyloxy)ethyl] trimethylammonium chloride and 3-sulfopropyl methacrylate (TMA/SA). Aluminum coupons were coated with the polymer chemistries and stored in a broth. Bacteria were then injected into the broth to observe bacteria adhesion to the coupons. Microgravity results are compared with results from Earthbased experiments. The results of this work could result in more effective bacteria-resistant polymer materials that could help reduce the risk of disease transmission and ensure material integrity for future long-term space missions.

Semiconductors

Manufacturing of Semiconductors and Thin-Film Integrated Coatings (MSTIC)

This investigation deposits thin film layers on a substrate to create layers of different materials that would be necessary for semiconductors. It is hypothesized that manufacturing these thin films in microgravity will reduce defects and improve their quality (due to the reduction in stresses that result from gravity-induced forces), and ultimately reduce the materials, equipment, and labor required to manufacture semiconductors. Microgravity-deposited films will be compared with Earth-deposited films.

Crystal Growth of Alloy Semiconductor Under Microgravity

This experiment grew InGaSb ternary alloy semiconductors in microgravity. The investigation studied solute transport in liquid and surface orientation dependence of growth conditions. Microgravity-grown InGaSb crystals were compared with Earth-grown counterparts. Ultimately, the results of this work improved understanding of how semiconductor materials grow and crystallize in microgravity and how higher quality crystals may be derived.

Thermophysical Properties

Thermophysical Property Measurement via Electro-Magnetic Levitation Experiments

Since its activation in 2015, the ISS-EML facility has successfully processed more than 50 samples, and the majority of these samples shared objectives from multiple research groups, particularly measuring thermophysical properties. The study of thermophysical properties of high-temperature liquid phases has recently become important in materials science research, especially in support of the growing field of additive manufacturing, where process control requires an understanding of the accuracy and the precision of molten metal properties.

Key thermophysical properties being measured using ISS facilities include the following:

- surface tension and viscosity using the oscillating droplet method
- specific heat
- thermal conductivity and total hemispherical emissivity using alternating current modulation calorimetry
- · density and thermal expansion using optical and inductive methods
- resistivity using sample coupling electronics

Using the oscillating droplet technique involves excitation of sample deformation inductively, either using a magnetic or electrical field, then allowing the sample motion to dampen through viscous decay. The natural frequency of this oscillation can be monitored using high-speed video imaging, as shown in Figure 18, which allows for the evaluation of surface tension while the deformation decay time enables measurement of sample viscosity. In the EML, this effect is accomplished through pulse excitation, where a single heater pulse is applied to cause squeezing of the sample.



Figure 18. A mosaic of frames showing selected side-view images at 30 kHz to demonstrate how a levitated sample deforms after pulse oscillation surface excitation. Figure reproduced from Matson et al., 2016.

Several key thermophysical properties were studied using the ISS-ELF during the 2020-2022 mission, consisting of density, thermal expansion coefficient, surface tension, viscosity and ratio of specific heat, and emissivity. The orthogonal CCD cameras recorded sample position during transfer operations, and once the sample was melted, one camera was set to a zoom position to monitor the sample size (in pixels) as a function of time during the cooling phase. By assuming axial symmetry for a known calibration of pixels to mm, the volume of the sample was monitored with temperature, allowing calculation of density and thermal expansion coefficient from the slope of the density curve, as shown in Figure 19. Tracking of sample mass loss was accomplished post mission. Surface tension and viscosity were measured after sample melting by setting the heating laser at a constant temperature.



Figure 19. A molten Au sample levitated on the ISS-ELF (top left), induced surface oscillation signals (top right), backlit sample (bottom left), and pyrometer (top right). Image Credit: NASA

A sinusoidal modulation of the positioning voltage was applied to both electrodes to excite surface oscillations without changing the sample position. Maximum deformation at a given amplitude defines the natural frequency for oscillations and is used to evaluate the surface tension. Once the natural frequency is identified, the surface excitation modulation is turned off, and the surface motion dampens to zero. The characteristic exponential damping time was used to evaluate the viscosity.

Dynamic mass is important for tracking density, surface tension, and viscosity measurement. Integration of the time-temperature profile allowed calculation of evaporation, assuming a known relationship for loss as a function of temperature. Pre- and post- measurement of sample mass anchored these predictions.



Figure 20. Data from microgravity Electrostatic Levitation at ISS-ESL processing; (a) A typical processing thermal cycle, (b) Density of Liquid Platinum, (c) Surface tension of liquid Au, and (d) Viscosity of liquid Au. Image credit:Dr. Douglas Matson

A typical thermal cycle is shown in Figure 20(a), which shows readings from a sample that was held at a constant temperature for surface tension and viscosity measurement while the cooling curve was used to measure density and the thermal expansion coefficient. Liquid density of all these samples has been measured successfully in this facility. An example of the measured density of liquid platinum is shown in Figure 20(b) over a wide range of temperatures. Surface tension and viscosities of the pure metals have been measured successfully in this facility. Figure 20(c) and 20(d) show results from the Au experiments. For Earth-based measurements, care must be taken to limit evaporation of volatile components during processing of complex industrial alloys. All ELF measurements showed excellent agreement with the terrestrial measurements.

Phase Selection and Transformation Kinetics Using ISS EML

One of the key capabilities of space electromagnetic levitation is the ability to select and control convection to allow investigation from laminar to turbulent flow during containerless processing. This capability is shown in Figure 21 where the first part of the figure illustrates the process for obtaining laminar flow, and the second part illustrates the process for obtaining turbulent flow. In both, the initial application of a high heater setting causes the temperature to rise until it reaches 1,450°C, where the solid sample melts and then becomes a superheated liquid. After the applied heater field is reduced, the sample cools. While cooling, a heater pulse can be used to cause squeezing of the sample to allow for thermophysical property evaluation using pulse oscillation surface excitation. During cooling, the temperature can go below the melting point to allow observation of a metastable liquid in a condition called undercooling. Once nucleation occurs, the temperature rapidly rises in a process called recalescence. During this spike, the solid that forms is hotter than the surrounding liquid, and thus it becomes possible to track the growth of any solid phases that form and observe any subsequent metastable to stable phase transformations.



Figure 21. Thermal profile from a ternary stainless steel alloy showing temperature (blue) resulting from applied positioner (green) and heater (red) EM fields from two different runs: (a) heater voltage goes to 0V to access laminar flow and (b) heater voltage is set to 3V to access turbulent flow. The heater pulse during the liquid cooling process is used to obtain thermophysical properties. Figure reproduced from Matson et al., 2016.

An example of how the transformation can be observed using high speed digital video imaging is shown in Figure 22, where the first part of the figure shows an undercooled molten steel sample as it transforms to metastable ferrite and then to stable austenite. The second part of the figure shows a graph of the transformation delay during conversion of ferrite to austenite as a function of applied convection. Ground-based ESL provides no stirring (red) and laminar stirring (dotted red) while ground-based EML provides turbulent stirring (blue). Only EML in space can provide laminar (green) transitional flow (yellow) and turbulent flow (red) conditions. The star represents a baseline condition where stirring and undercooling are minimized for nucleation model normalization. The third part shows a scanning electron microscope (SEM) image of dendrites that formed during EML processing in space.



Figure 22. EML space processing (a) Molten 6.5mm DIA FeCrNi stainless steel sample showing growth of moderately bright metastable ferrite dendrites into dark undercooled liquid and the simultaneous transformation to bright austenite – dark lines are the cage wires. Figure reproduced from Matson et al., 2016. (b) Comparing transformation delay observed on ground and in space. Figure reproduced from Matson et al., 2023. (c) Postsolidification Ni-Sn dendrites on a sample surface. Photo courtesy of Doug Matson, Ph.D., Tufts University.

The Future of Materials Science Research in Microgravity

Research onboard the ISS will continue until the end of station. As the end of the ISS era nears, microgravity materials research will begin to transition to other microgravity research platforms in space such as commercial low Earth orbit destinations, free flyers, and rocket-powered suborbital platforms. Until then, the ISS continues to serve as an ideal platform for microgravity materials research. It is imperative that the research capabilities of the ISS be used to their full potential to continue to enable groundbreaking scientific discoveries and a sustainable low Earth orbit economy before the end of station. Below are brief descriptions of some research areas that are important to the future of materials science research in space.

Metals

In the last few decades, advances in high-performance computing, new materials characterization methods, and, more recently, an emphasis on Integrated Computational Materials Engineering (ICME) coupled with additive manufacturing has been a catalyst for multiscale modeling and simulation-based design of materials and structures across all industries. Through ongoing experimental and computational projects on the ISS, containerless processing supports the vision of the 2023-2032 Biological and Physical Sciences Decadal Survey priorities through greater attention to the potential benefits of domestic and international partnerships along with the growing capability of commercial service providers.

The future of materials science is to develop new materials for highperformance applications for NASA and the space economy. Novel metal alloys are a significant focus area for ICME, including shape memory alloys (actuators), Ni superalloys (high temperature applications), high entropy alloys (additive manufacturing), and magnetic systems. Density-function theory computations, interatomic potential development, and molecular dynamic simulations of thermodynamic and mechanical processes are also being pursued. All these advanced techniques require a baseline database on which models may be trained. Collaboration with researchers at other NASA centers, other government agencies, and universities, as well as in industry and international partnerships, is also a key focus to further progress the future of microgravity research. Various collaborative teams currently work to generate a database for ICME validation of solidification models through experimental work. Microgravity levitation processing supports this work by providing experimental validation of behavior and thermophysical properties for the prediction of a wide range of phenomena hidden by gravity, phase selection and solidification behavior, and quantification of uncertainty in the measurement of thermophysical properties. Uniting these three key threads through process modeling will allow future designers to improve material performance, provide better process control, and mitigate the deleterious effects of defects in the structure by predicting microstructural evolution.

Brazing, Soldering, & Joining

In-space servicing, assembly, and manufacturing (ISAM), as well as repair processes, require further development and maturation of alloy solidification processes in space environments such as brazing, soldering, and joining (including welding and additive manufacturing processes such as wirefeed directed energy deposition). The physical mechanisms governing the solidification and microstructural evolution of these key terrestrial metalworking and manufacturing processes are poorly understood in space environments, especially with respect to microgravity or reduced gravity such as the lunar surface. This unresolved understanding of fundamental processes during solidification in space led the most recent 2023-2032 Biological and Physical Sciences (BPS) Decadal Survey to ask, "How does the space environment impact the joining of materials—for example, by welding...?"

NASA has collated datasets from historical and recent flight experiments under microgravity materials science. NASA-managed open data repositories, such as Physical Sciences Informatics (PSI), provide publicly available datasets that are useful for conducting new scientific investigations using existing experiment data. Relevant datasets include both the In-Space Soldering Investigation (ISSI) and the Brazing of Aluminum alloys IN Space via SUBSA (SUBSA BRAINS) experiments. For example, a recent re-investigation of several ISSI specimens was able to provide copious characterization data using advanced nanomechanical techniques. The research also employed coupled solidification-fluid dynamics models of the soldered specimens to predict resultant microstructure and mechanical properties. This experience demonstrates the value of retaining physical specimens and datasets in an open science manner for use beyond the original experimental period. Future works can readily leverage these open science assets. Researchers studying microgravity and other space environmental factors affecting brazing, soldering, and joining processes can greatly contribute to the development of integrated computational materials engineering (ICME) tools and processes that promise to revolutionize design and qualification of ISAM components and systems. Presently, ICME models do not fully capture the unique physical mechanisms underlying alloy solidification in space such as microgravity and reduced gravity, reduced pressure, and extreme temperatures that significantly affect brazing, soldering, and joining.

During the remaining ISS availability, additional brazing and soldering and new welding experiments are of interest both to enhance fundamental understanding of processes that are highly dependent on metal solidification mechanisms and to support emerging ISAM efforts. Follow-on brazing experiments may investigate additional alloys and joint geometries, while soldering experiments could further explore the relationship between molten solder morphology, thermophysical properties, and internal fluid dynamics. A joining experiment on the ISS, especially employing non-contact welding methods such as electron or laser beam, would represent the first humantended experiment of its kind since Skylab and would benefit from modern characterization methods. Ultimately, brazing, soldering, and joining investigations will continue, with initial experiments conducted on the ISS as a springboard for those on future commercial low Earth orbit destinations.

Glasses & Ceramics

It can be said that glasses and ceramics are the materials of the future. This statement underestimates the transformational importance and value that these materials already represent in optical communications, advanced energy applications, aerospace, and medicine. However, it does highlight the continued potential for glasses and ceramics to transform technologies both in space and on Earth.

Glasses can be formed from all classes of materials. Changes in thermophysical and thermochemical properties (e.g., viscosity, density, heat capacity), structural evolution, and energetics differ significantly among materials polymerized and unpolymerized ionic liquids, semiconductors, metals, and organic molecules. A unified model of glass formation encompassing these diverse materials will be a major step forward in understanding the vitrification process. Toward this goal, obtaining measurements on supercooled liquids as they cool and form glass is important to answer questions about the mechanisms of vitrification.

- Understanding the extent to which glass-forming behavior can be predicted from liquid properties and structure will help to guide the focus of experimental research efforts to improve functional glass properties.
- In soft materials, molecular conformality can be affected by fluid flowinduced stresses.
- Understanding how this effect influences the stability of glasses and their properties can help to improve pharmaceutical and food processing and the development of functional soft materials.
- Studying the effects and behavior of radiation (e.g., cosmic rays) on glasses is important for the development of reliable, long-range flight hardware.
- Natural glasses are abundant and could potentially be harvested to aid in space exploration and habitat construction and could serve as feedstock to produce new materials.

Supercooling and supersaturation are frequently means by which a system enters non-equilibrium. The degree to which this occurs depends on avoiding nucleation of new phases. Supercooling/ supersaturation is essential to form glass from liquid precursors. Containerless processing can enable deep supercooling/super-saturation of liquids by eliminating heterogenous nucleation. This process enables investigation of supercooled liquids and, often, the formation of glass or amorphous materials that cannot be made by other methods. Levitation techniques can help to reveal the structural pathways through a variety of cooling routes. These "extreme" glasses can serve as benchmarks for glass discovery. Understanding the ways that thermophysical properties and the structure of liquids. For example, correlation of supercooling contributes to models of liquids. For example, correlation of melt and solution properties and structure with process parameters can provide needed data for machine-learning-based design of functional materials.

Semiconductors

Semiconductors have been an active area of investigation in low Earth orbit going back to Skylab. A meta-analysis of publicly available data showed that more than 160 semiconductor crystals had been grown in microgravity. Of that dataset, more than 80% showed improvement in one or more characteristics, including structure, uniformity, and defect reduction (Wright et al., 2022). These results come at a time of change within the semiconductor industry as it approaches the limits of Moore's Law. In order to continue the acceleration of computing power, researchers are increasingly seeking out new architecture, materials, and processes beyond traditional silicon and complementary metaloxide semiconductor (CMOS) technology in today's integrated circuits. Building on these foundational investigations, it is expected that microgravity crystal growth, processing, and even manufacturing may lead to improved technologies. The database of semiconductor crystal growth (Wright et al., 2022) indicates it may be possible to improve the crystal size and purity for semiconductor crystal boule production via growth in a microgravity environment. This possibility is particularly intriguing for next-generation wide-bandgap semiconductors for power converters and high-power electronics. Semiconductor crystal growth may also be applied to applications for sensors and radiation detectors.

In addition, the future of semiconductor technology is expected to move beyond traditional CMOS technology. This may include 2D materials like graphene, boron nitride, and transition metal dichalcogenides; photonic circuits; and non-von Neumann computing including neuromorphic and quantum computing. This may also include advanced manufacturing including additive manufacturing where microgravity can be leveraged to eliminate some processing steps in traditional wafer processing.

One specific example of non-traditional semiconductors for electronics are semimetal-semiconductor composites (SSC), which are based on two-phase material structures with a semimetal (or metallic) phase embedded in a semiconductor bulk matrix. These composites are a novel class of materials with unique characteristics for numerous large-scale emerging applications, including magnetic sensing, quantum computing devices, thermoelectrics, photovoltaic power generation, etc. They are based on two-phase material structures with a semimetal (or metallic) phase embedded in a semiconductor bulk matrix. The field of SSC is still in its infancy, primarily due to the absence of adequate commercial crystal growth technology and, as a result, a lack of appropriate materials for device research and product development.

Crystals grown in terrestrial conditions exhibit morphologies of semimetal wires as depicted in Figure 32(a). The semimetal wires are discontinuous, aligned along different directions, and have varying diameters and spatial distribution across the entire wafer. Figure 32(b) shows the desired morphology for SSC wafers as produced in microgravity to be useful for large-scale device fabrication. Microgravity conditions provide the perfect environment for eliminating the effect of natural convection and buoyancy while keeping the solid-liquid interface shape flat throughout the experiment. In addition, the microgravity environment may enable crystal polymorphism that includes formation of new crystal species not observed under terrestrial conditions with potential novel applications.



Figure 23. (a) Optical micrograph depicting the morphology of NiSb-InSb semimetalsemiconductor composite (SSC) wafers extracted from terrestrial-grown bulk crystals. Discontinuous semimetal needles embedded in semiconductor matrix leads to poor yield of high-performance electromagnetic sensors. (b) Optical micrograph depicting the expected morphology of SSC wafers from space-grown bulk crystals. The continuous semimetal needles embedded in the semiconductor matrix will provide high yield of high-performance electromagnetic sensors. The figure was provided by United Semiconductors LLC.

Examples of other emerging technologies in materials science for potential research in microgravity include programmable matter, quantum dot (semiconductor nanocrystal) applications, and synthetic biology.

Funding, Developing, and Launching Research to the ISS

Every experiment on the ISS needs to be sponsored and funded to be developed, integrated, flown, and operated onboard. Several sources of funding are available to scientists for research, payload development, payload processing at NASA facilities, in-orbit operation, and post data collection analysis and reporting of results.

Funding sources can be broadly divided into:

- International funding sources via non-U.S. space agencies
- U.S.-sponsored funding via NASA and the ISS National Laboratory

International Funding Sources

Unique and integral to the ISS are the partnerships established between the United States, Russia, Japan, Canada, and Europe. All partners share in one of the greatest international collaborations of all time, providing various research platforms and experiment opportunities. These organizations provide potential funding opportunities for international scientists from many diverse disciplines:

- Canadian Space Agency (CSA)
- ESA (European Space Agency)
- Japan Aerospace Exploration Agency (JAXA)
- Russian space agency Roscosmos

NASA

In general, NASA funding for ISS use is obtained through NASA Research Announcements (NRAs). Funding from other government agencies, private, and nonprofit entities to use the ISS is obtained through ISS National Lab Research Announcements (NLRAs) released by the ISS National Laboratory®, managed by the Center for the Advancement of Science in Space[™] (CASIS[™]). ISS international partner funding can be obtained through their respective agencies.

Potential funding for research on the ISS is also available via governmental partnerships with the ISS National Lab and includes, but is not limited to, such government agencies as:

- U.S. National Science Foundation (NSF)
- National Institutes of Health (NIH)
- U.S. Department of Agriculture (USDA)
- Department of Energy (DOE)
- Department of Defense (DOD)

ISS National Laboratory

The ISS was designated a U.S. National Laboratory by Congress in 2005. In 2011, NASA finalized a cooperative agreement with CASIS to manage the ISS National Lab. The independent, nonprofit research management organization ensures the unique capabilities of the ISS are available to scientific, technological, and industrial communities in the United States.

It provides a marketplace for commercial research and funding sources to conduct innovative research on the ISS platform. The goal is to support, promote, and accelerate innovations and new discoveries in science, engineering, and technology that benefit humanity.

More information on the ISS National Lab, including NLRAs, is available at <u>www.issnationallab.org</u>.

In-Space Production Applications

Decades of materials research in microgravity has shown the potential of producing unique and sometimes superior material properties compared to those produced on Earth. Even before Skylab was operational, researchers dreamed about what new materials could be created in the microgravity environment and how these new materials could provide technological advancements for humans on Earth in addition to space-based benefits. One of the first products produced in space and commercialized on Earth was polystyrene beads, later called "space beads". These were produced on the space shuttle Challenger in 1983 in a collaboration between NASA and the U.S. National Institute of Standards and Technology (NIST). Compared to Earth manufactured beads, the "space beads" exhibited superior particle sphericity, narrow size distribution, and particle rigidity.

NASA and the ISS National Lab continue to support new materials development through the In-Space Production Applications (InSPA) portfolio. The tenant of the InSPA program is to support research and development institutions to leverage the microgravity environment to produce new materials and products with the intention of returning the products for use on Earth. InSPA research is poised to bridge the gap between discovery and application, addressing the "valley of death" between lab-based research and the creation of a successful product, medical treatment, manufacturing process, or new and improved material.

One example of this is the active research in ZBLAN optical fiber production discussed previously. Several companies that have been awarded grants through InSPA are attempting to produce optical fibers on the ISS with improved optical properties such as attenuation compared to terrestrial products. Other examples include crystal growth, thin-layer deposition, additive manufacturing, and advanced material manufacturing ranging from 2D materials to semiconductors to nanomaterials.

ISS Commercial

NASA has opened the ISS for business to enable commercial and marketing opportunities on the microgravity laboratory. Since then, there has been a growing demand for commercial and marketing activities from both traditional aerospace companies and novel industries, demonstrating the benefits of the ISS to help catalyze and expand space exploration markets and the low Earth orbit economy.

NASA's Commercial Low Earth Orbit Development Program is supporting the development of commercial owned and operated space stations in low Earth orbit from which the agency, along with other customers, can purchase services and stimulate the growth of commercial activities in microgravity. NASA is using a two-phased approach to, first, support the design and development of commercial space stations and, second, enable the agency to certify the commercial space stations and purchase services as one of many customers. NASA is committed to maintaining a continuous human presence in low Earth orbit as the agency transitions from the ISS to commercial space stations.

ISS operations continue to return benefits to the United States, and to humanity as a whole, while preparing for a successful transition of those capabilities to one or more commercial space stations. NASA awarded a contract to provide at least one habitable commercial module to be attached to the ISS and signed two funded Space Act Agreements for the design of commercial space stations that go directly into low Earth orbit. In addition, the agency signed seven unfunded Space Act Agreements with companies to develop additional commercial space station concepts and other commercial technologies. U.S. industry is developing these commercial space stations to begin operations in the late 2020s for both government and private-sector customers, concurrent with space station operations, to ensure these new capabilities can meet the needs of the United States and its partners.

Working with NASA

Once a payload has been selected for development by the funding program the ISS Research and Integration Program Office will work with payload teams through the design, test, certification, build, and launch phases prior to beginning mission operations on the ISS. More detailed information on this process, and information on current and planned launch vehicles, is available at https://www.nasa.gov/stationopportunities._

Potential proposers to any NASA program announcement should contact the relevant Program Scientist to discuss the appropriateness of their concepts for the specific solicitation and to determine who to contact within the ISS Program Office for discussing expected development costs for their proposal budgets.

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