

Coarsening in Solid-Liquid Mixtures (CSLM)

The Coarsening in Solid-Liquid Mixtures (CSLM) experiment is a materials science space flight experiment whose purpose is to investigate the kinetics of competitive particle growth within a liquid matrix. During coarsening, small particles shrink by losing atoms to larger particles, causing the larger particles to grow. In this experiment solid particles of tin will grow (coarsen) within a liquid lead-tin eutectic matrix. By conducting this experiment in a microgravity environment, a greater range of solid volume fractions can be studied, and the effects of convection present in terrestrial experiments will be negligible. The flight hardware consists of two separable pieces of equipment, the sample processing unit (SPU) and the electronic control unit (ECU).



Contact Information

PI: Professor Peter W. Voorhees, Northwestern University

PS: Dr. Walter Duval, NASA GRC

PM: Robert Hawersaat, NASA GRC

Engineering Team: ZIN Technologies, Inc.

Coarsening in Solid Liquid Mixtures-2 (CSLM-2)

In an effort to reduce the total interfacial area per volume, two-phase mixtures undergo coarsening wherein large particles grow and small particles shrink. The process occurs via a diffusive mass transfer from small particles to large particles; thus, it is important for the particles to remain stationary in the matrix phase.

Materials containing a few large particles rather than many small particles can be structurally weaker; thus, the coarsening process affects the properties of many materials, such as aluminum alloys. To study this process in a system that can be directly compared to theory, investigators employ a two-phase solid-liquid mixture. Unfortunately, on Earth, the solid Sn-particles sediment to the top of the sample. Such a non-uniform distribution of particles is not observed in solid alloys or assumed in theory. Performing the experiments in space will allow investigators to study the coarsening process in a manner that can be directly compared to theory and other two-phase alloys.

CSLM-2 samples are processed inside the Sample Processing Unit (SPU), which has a large cylindrical sample chamber. After a sample is processed, pressurized water is pumped into the chamber to quench the sample, cooling it for removal. This system can quench the sample from 185°C (the temperature required to form the solid-liquid mixture and initiate coarsening in tin-lead (Sn-Pb) samples) to 120°C in only 6 seconds.



The Electronics Control Unit (ECU) provides power and the software that controls all stages of processing. Parameters and status are displayed on the ECU's LCD screen. The ECU controls the temperature inside the SPU sample chamber and monitors and records the sample's temperature. The quenching stage can be initiated automatically or controlled manually by the crew. A base plate attaches the SPU and ECU to the Microgravity Science Glovebox (MSG) work volume floor.

Previous Missions

CSLM-1, a precursor to CSLM-2, was conducted on STS-83 and STS-94. CSLM-2 was conducted during ISS Expedition 7. CSLM-2 was conducted during ISS Increments 16 and 17. CSLM-2R with low volume fraction samples was conducted during Increments 23 and 24.

Science Objectives

Science Objectives for Everyone

Coarsening in Solid Liquid Mixtures-2 (CSLM-2) investigates the rates of coarsening of solid particles embedded in a liquid matrix. During this process, small particles shrink by losing atoms to larger particles, causing the larger particles to grow (coarsen) within a liquid lead/tin matrix. This study defines the mechanisms and rates of coarsening that govern similar processes that occur in materials such as turbine blades, dental amalgam fillings, aluminum alloys, etc.

Science Results for Everyone

Lumps in your hot chocolate are annoying; lumps in alloys can spell disaster. This experiment studied the rate at which particles of tin suspended in molten alloy increased in size. In this process, called coarsening, small particles shrink by losing atoms to larger particles, which then grow. Large particles can result in structural weakness, affecting materials such as dental fillings and aluminum alloys. Samples from an earlier low volume test were not returned to Earth in time for evaluation, but data was collected on equipment function. Samples from a later high-volume run were successfully returned and are currently being analyzed. Other results show that particle size distributions in microgravity were very close to that predicted by theory. Analysis is ongoing.

Applications

Space Applications

In any mixture that contains particles of different sizes, the large particles tend to grow while the smaller particles shrink in a process called coarsening. Tiny oil droplets coalescing into a large blob are one illustration, but the process occurs in solids as well. Coarsening occurs on Earth during the processing of any metal alloy and thus the coarsening process affects products from dental fillings to turbine blades. Since the properties of an alloy are linked to the size of the particles within the solid, coarsening can be used to strengthen materials. This is the case with the majority of aluminum alloys used commercially today. Conversely, if the coarsening process proceeds too long the material can weaken. This occurs in jet turbine blades and is one of the reasons why turbine blades must be replaced after a certain number of hours of service. Thus, developing accurate models of the coarsening process is central to creating a wide range of new materials from those used in automobiles to those used in space applications. The results of previous experiments performed on the Shuttle have done just that. These models have been incorporated into a

computer code that is being used to design many new materials, including materials of importance to NASA's spaceflight program. Solid-liquid systems are ideal systems to study this coarsening process. However, gravity can induce particle sedimentation and thus hamper the studies of coarsening in these mixtures on Earth. The microgravity environment of the Space Station allows scientists to study the process of coarsening with reduced interference from the sedimentation that occurs on Earth.

Earth Applications

On Earth, materials that contain pores created and trapped during solidification degrade properties and cause a distinct weakening in the overall structure of the cast product. Determining what causes these problems will lead to the development of improved manufacturing processes for materials.

Operations

Operational Requirements and Protocols

CSLM-2 will be conducted inside the sealed MSG work volume. The crew must load and initiate each run. Quenching can be initiated manually. Data captured by the ECU is transferred to the MSG laptop for storage and downloading to the ground-based researchers. The samples are a mixture consisting of Sn (tin)-rich particles in a Pb-Sn liquid, a mixture that has a low sintering temperature and a high coarsening rate, making it perfect for studying Ostwald ripening.

The crew will set up the CSLM-2 hardware and test it before running the first sample. The sample runs are initiated using toggles on the ECU. Once started, the experiments run autonomously. When the sample is completed, the crew will download data from the ECU to the MSG laptop and switch samples by removing the SPU sample chamber and replacing it with a new one. The processed sample chambers are stored until they can be returned to Earth by Shuttle. The hardware is removed from the MSG work volume and stowed after all the runs have been completed. On Earth, the researchers will study each sample for particle size distribution, particle morphology, matrix structure, and particle crystallographic orientation.

Publications

Samples from CSLM-2 that were processed during Increment 7 were not able to be returned to Earth in time for evaluating the results. Although the data was lost, engineering data collected on equipment function can benefit subsequent experiments. The CSLM-2 high volume fraction samples from Increment 16 and 17 were successfully processed and returned for analysis during 2008. The CSLM-2R low volume fraction samples were successfully processed during Increment 23 and 24 and returned on Shuttle flight STS-133/Flight ULF-5 in March of 2011 and are currently under analysis at Northwestern University. Preliminary analysis of the low volume fraction samples indicates that the furnaces performed as planned. We are continuing to section the other samples to determine if this is case for all the volume fractions processed. Recent results show that the particle size distributions for a 30% volume fraction of coarsening phase is very close to that predicted by theory. The particle spatial distribution functions appear different, perhaps due to the nonspherical shape of the particles that are present at this high-volume fraction. Analysis of both the low and high-volume fraction samples is continuing.

Results Publications

- Physical Review Materials; Title: [Coarsening of solid \$\beta\$ -Sn particles in liquid Pb-Sn Alloys: Reinterpretation of experimental data in the framework of trans-interface-diffusion-controlled coarsening](#); Published: April 2021
- Cool T, Voorhees PW. The evolution of dendrites during coarsening: Fragmentation and morphology. Acta Materialia. 2017 April 1; 127359-367.
DOI: [10.1016/j.actamat.2017.01.029](#). DOI: [10.1016/j.actamat.2017.01.029](#)
- [Duval WM, Hawersaat RW, Lorik T, Thompson J, Gulsoy EB, Voorhees PW. Coarsening in Solid-liquid Mixtures: Overview of Experiments on Shuttle and ISS. 2013 Materials Science and Technology Conference and Exhibition, Montreal, Quebec, Canada. 2013 Oct 27;](#)

Related Publications

- Thomson JR, Casademunt J, Drolet F, Vinals J. Coarsening of solid-liquid mixtures in a random acceleration field. Physics of Fluids. 1997 9(5): 1336-1343. | Abstract
- Snyder V, Alkemper J, Voorhees PW. The development of spatial correlations during Ostwald ripening: a test of theory. Acta Materialia. 2000 482689. DOI: [10.1016/S1359-6454\(00\)00036-7](#). DOI: [10.1016/S1359-6454\(00\)00036-7](#)
- Alkemper J, Snyder V, Akaiwa N, Voorhees PW. The Dynamics of Late-Stage Phase Separation: A Test of Theory. Physical Review Letters. 1999 822725. | Abstract
- Kammer D, Genau A, Voorhees PW, Duval WM, Hawersaat RW, Hickman JM, Lorik T, Hall DG, Frey CA. Coarsening In Solid-Liquid Mixtures: A Reflight. 46th Aerospace Sciences Meeting and Exhibit, Reno, NV. 2008 AIAA2008-813. | Abstract
- Kammer D, Genau A, Voorhees PW, Duval WM, Howersatt R, Hickman JM, Lorik T, Hall DG, Frey CA. Results from the International Space Station: Coarsening in Solid-Liquid Mixtures. 47th Aerospace Sciences Meeting and Exhibit, Orlando, FL. 2009 AIAA2009-0616. | Abstract
- Seyhan I, Ratke L, Bender W, Voorhees PW. Ostwald Ripening of Solid-Liquid Pb-Sn Dispersions. Metallurgical and Materials Transactions A. 1996 27(9): 2470 – 2478. | Abstract
- Rowenhurst DJ, Kuang JP, Thorton K, Voorhees PW. Three-dimensional analysis of particle coarsening in high volume fraction solid-liquid mixtures. Acta Materialia. 2006 54(8): 2027-2039. | Abstract
- [Gulsoy EB, Wittman K, Thompson J, Voorhees PW. Coarsening in Solid-Liquid Mixtures: Effect of Microgravity Accelerations on Particle Sedimentation. 49th AIAA Aerospace Sciences Meeting including the New Horizons Forum and Aerospace Exposition, Orlando, FL. 2011 Jan 4-7; AIAA 2011-1346-323](#)

Contact Information

PI: Professor Peter W. Voorhees, Northwestern University

PS: Dr. Walter Duval, NASA GRC

PM: Robert Hawersaat, NASA GRC

Engineering Team: ZIN Technologies, Inc.

Coarsening in Solid Liquid Mixtures-3 (CSLM-3)

The CSLM-3 investigation expects to grow and examine metal dendrites (i.e., tree-like structures) that form during the solidification of all metals. Dendrites are also observed when water freezes and are called snowflakes. The spacing between the branches of the dendrite controls the mechanical properties of the solidified metals, such as engine blocks used in car engines. During the casting process the dendrites undergo a process called coarsening. During coarsening the dendrites change their shape, with a change in the spacing between the branches of the dendrites. Since the spacing alters the mechanical properties of the alloy, the coarsening of dendrites has a major effect on the properties of metal alloys. The objective of this experiment is to investigate this coarsening process without the complicating effects of convection of the liquid or sedimentation (e.g., gravity induce effects) of the dendrites.

In the predecessor CSLM-1 and 2 studies, spherical particles of the high-volume liquid lead-tin fraction samples were observed during coarsening. The CSLM-1 and 2 experiments also used two-phase solid alloys as analogs that contained precipitates in a solid. The compositions of those alloys are very similar to those that are planned for use in the CSLM-3 experiments, the major difference being that the CSLM-3 alloy consists of solid dendrites rather than spherical particles. Research has shown that spherical particles coarsen in a very different fashion than dendrites.

The CSLM-3 samples are a mixture consisting of Sn (tin)-rich particles in lead-tin liquid, a mixture that has a low sintering temperature and a high coarsening rate, making it perfect for studying the process of Oswald ripening. Sample runs are conducted inside the sealed MSG work volume using existing CSLM-2 hardware. Samples are processed inside a Sample Processing Unit (SPU), which has a cylindrical sample chamber. Each SPU contains 4 samples and crewmembers must load each SPU, and initiate runs individually. Samples are heated to 185 degrees Celsius to enable dendrite growth. This temperature is maintained for various, predetermined intervals. Each isothermal heat soak time is unique for each SPU. After an SPU is processed, pressurized water is released into the chamber to quench the sample, cooling it down to lock in the structures. During a normal sequence the quench cycle is initiated automatically by the Electronic Control Unit (ECU). Quenching can be initiated manually if needed. Data captured by the ECU is transferred to the MSG laptop for storage and downloading to the ground-based researchers. The ECU provides power and controls all stages of the sample processing and experimental parameters, and status are displayed on the ECU's LCD display screen. The ECU also controls the temperature inside the SPU sample chamber and monitors and records the sample's temperature. A baseplate is used to attach the SPU and ECU to the Microgravity Science Glovebox (MSG) work volume floor.

Previous Missions

The initial CSLM-2 investigations were conducted during ISS Increment 7. CSLM-2 high volume fraction samples were conducted during ISS Increment 16/17. CSLM-2R with low volume fraction samples were conducted during Increment 23/24.

Science Objectives

Science Objectives for Everyone

The Coarsening in Solid Liquid Mixtures-3 (CSLM-3) is a materials science investigation that studies the growth and solidification processes (i.e., coarsening) in lead-tin solid-liquid mixtures that contain a small amount (low volume fraction) of tin branch-like (i.e., dendritic) structures, some of which possess many arms. During sample heating, the growth at the tip of each dendrite continues over time, whereas side branches, behind the tip, develop during constant temperature (i.e., isothermal) conditions. By understanding how temperature and time control the growth of such dendrites, researchers hope to develop more efficient and economical means of producing higher quality products derived from the casting of molten metals.

Applications

Earth Applications

CSLM-3 examines the growth of metal dendrites (i.e., tree-like structures) that form during the solidification of all metals. This process is called coarsening and the growth of these dendritic structures within solid-liquid mixtures has major technological and production implications, since it occurs in virtually all casting processes. During the casting process, the coarsening of dendrites changes their shape and the spacing between branches of the dendrites, which alters the mechanical properties of the solidified metals and alloys, such as a car engine block. In the case where a dendritic structure forms quickly, a so-called “mushy zone” is created. This mushy zone can exist, often for long periods of time, during which virtually every dendrite undergoes coarsening. The length of the dendrites in the mushy zone, and thus those present after complete solidification, are determined by the coarsening process and are intimately related to the mechanical properties of metal ingots. Through a better understanding and control of the formation of dendrites and anomalous structures this research leads to the production of more efficient methods and ultimately better products that are cast from molten metals and alloys.

Operations

Operational Requirements and Protocols

CSLM-3 is required to operate within the MSG. Crew support during setup, monitoring, processing of samples, data recording and stowing of investigation hardware is necessary. During both SPU pre and post processing, the temperature of each SPU is required to remain below 30 degrees Celsius.

- The crew unstows and sets up the CSLM-2 hardware (ECU, first SPU, baseplate, cables, and vacuum hose) in the MSG and runs any necessary vacuum cycles before testing each SPU which contains the lead-tin samples.

- Sample heating runs are initiated using a toggle switched on the ECU. Once started, the investigation runs autonomously.
- Individual SPU heat soak times range from 10 min, 1.6 hours, 5.5 hours, 13.5 hours, 27 hours, and 48 hours, followed by a quench cycle.
- When an individual sample run is completed, the crew downloads data from the ECU to the MSG laptop and switches samples by removing the SPU and replacing it with a new SPU.
- Once all runs have been completed, the hardware is removed from the MSG work volume and stowed.
- The processed SPU's are stored on ISS until they can be returned to Earth. On Earth, the researchers analyze each sample for particle size distribution, particle morphology, matrix structure, and particle crystallographic orientation.

Contact Information

PI: Professor Peter W. Voorhees, Northwestern University

PS: Dr. Walter Duval, NASA GRC

PM: Robert Hawersaat, NASA GRC

Engineering Team: ZIN Technologies, Inc.

Coarsening in Solid Liquid Mixtures-4 (CSLM-4)

In the predecessor CSLM-3 study, samples are a mixture consisting of Sn (tin)-rich particles in lead-tin liquid, a mixture that has a low sintering temperature and a high coarsening rate, making it perfect for studying the process of Oswald ripening. Sample runs are conducted inside the sealed MSG work volume using existing CSLM hardware.

The CSLM-4 investigation further examines metal dendrites (i.e., tree-like structures) that form during the quenching and solidification of molten metals. The spacing between the branches of the dendrite controls the mechanical properties of the solidified metals, such as engine blocks used in car engines. During the casting process the dendrites undergo a process called coarsening. During coarsening the dendrites change their shape, with a change in the spacing between the branches of the dendrites. Since the spacing alters the mechanical properties of the alloy, the coarsening of dendrites has a major effect on the properties of metal alloys. The objective of this experiment is to investigate this coarsening process without the complicating effects of convection of the liquid or sedimentation (e.g., gravity induce effects) of the dendrites.

Samples are processed inside a Sample Processing Unit (SPU), which has a cylindrical sample chamber. Each SPU contains 4 samples and crew members must load each SPU and initiate runs

individually. Samples are heated to 185 degrees Celsius to enable dendrite growth. This temperature is maintained for various, predetermined intervals. Each heating time is unique for each SPU. After an SPU is processed, pressurized water is released into the chamber to quench the sample, cooling it down to lock in the structures. During a normal sequence the quench cycle is initiated automatically by the Electronic Control Unit (ECU). Quenching can be initiated manually if needed.

Data captured by the ECU is transferred to the MSG laptop for storage and downloading to the ground-based researchers. The ECU provides power and controls all stages of the sample processing and experimental parameters, and status are displayed on the ECU's LCD display screen. The ECU also controls the temperature inside the SPU sample chamber and monitors and records the sample's temperature. A baseplate is used to attach the SPU and ECU to the Microgravity Science Glovebox (MSG) work volume floor.

Previous Missions

The initial CSLM-2 investigations were conducted during ISS Increment 7. CSLM-2 high volume fraction samples were conducted during ISS Increment 16/17. CSLM-2R with low volume fraction samples were conducted during Increment 23/24. CSLM-3 dendritic growth samples were conducted during ISS Increment 33/34, and 35/36.

Science Objectives for Everyone

The Coarsening in Solid Liquid Mixtures-4 (CSLM-4) investigation studies growth and solidification processes in tin-lead mixtures that contain a small amount of tin dendrites. Some metal alloys form tiny branching structures called dendrites when they crystallize, and the size, spacing, and interlocking of these dendrites play an important role in determining their physical properties such as softness, hardness, or brittleness. The microgravity environment of the International Space Station enables scientists to study dendritic growth without any interference from gravity to improve our knowledge of materials science, potentially leading to making new alloys with enhanced properties.

Applications

Space Applications

Alloys' properties are strongly linked to their dendritic makeup and the spaces between the individual branches. Understanding the controlling factors in the growth of dendrites is crucial for developing accurate computer simulations and manufacturing techniques. On Earth, gravity affects the growth and solidification process (called coarsening), so microgravity-based experiments are necessary to better understand the true nature of dendrite growth. New insights into dendrite formation and alloy behavior are used to design lighter and stronger materials for use in space exploration.

Earth Applications

Understanding how temperature and time affect the growth of dendrites helps researchers to develop more efficient and economical means of producing higher quality products from the casting of molten metals. CSLM series of experiments provide insights into new physics that can be

used by alloy scientists and engineers to enhance the properties of important materials used in the manufacturing jet turbine blades, automobile engine components, and support structures for buildings and bridges.

Operations

Operational Requirements and Protocols

CSLM-4 is required to operate within the MSG. Crew support during setup, monitoring, processing of samples, data recording and stowing of investigation hardware is necessary. During both SPU pre and post processing, the temperature of each SPU is required to remain below 30 degrees Celsius.

The crew unstows and sets up the CSLM-4 hardware (ECU, first SPU, baseplate, cables, and vacuum hose) in the MSG and runs any necessary vacuum cycles before testing each SPU which contains the lead-tin samples. Sample heating runs are initiated using a toggle switched on the ECU. Once started, the investigation runs autonomously. Individual SPU heat soak times range from 10 min, 15 min, 30 min, 1 hour, 5 hours, and 15 hours, followed by a quench cycle. When an individual sample run is completed, the crew downloads data from the ECU to the MSG laptop and switches samples by removing the SPU and replacing it with a new SPU. Once all runs have been completed, the hardware is removed from the MSG work volume and stowed. The processed SPU's are stored on ISS until they can be returned to Earth. On Earth, the researchers analyze each sample for particle size distribution, particle morphology, matrix structure, and particle crystallographic orientation.

Contact Information

PI: Professor Peter W. Voorhees, Northwestern University

PS: Dr. Walter Duval, NASA GRC

PM: Robert Hawersaat, NASA GRC

Engineering Team: ZIN Technologies, Inc.