Apr 1st, 8:00 AM

Ground Based Materials Science Experiments At Lewis Research Center

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In order that researchers can take full advantage of the opportunities offered by the space shuttle and space station, NASA has furnished a facility to encourage thorough pre-flight development and post-flight analysis of experiments. The Microgravity Materials Science Laboratory (MMSL) was created to offer immediate and low-cost access to ground-based facilities where industrial, academic, and government researchers can test their ideas. By examining preliminary experiments in the specialized equipment in the MMSL, ideas can be developed and promising concepts can be identified early. Researchers can use the experience gained in the MMSL to select their most promising experiments for more rigorous and more expensive testing, including flight testing. The Microgravity Materials Science Laboratory is a service laboratory, equipped and staffed with the goal of providing hardware and expertise to scientists with ideas.

The equipment in the MMSL falls into three categories: devices which emulate some aspect of low gravitational forces, specialized capabilities for one-g development and refinement of microgravity experiments, and functional duplicates of flight hardware.

**DEVICES WHICH EMULATE LOW-GRAVITY CONDITIONS**

When a material is sensitive to contamination by its crucible, a low-gravity environment can allow the crucible to be eliminated, providing for containerless processing. Another need for microgravity occurs during melting and solidification, when density differences can lead to maldistributions of material. Various levitation and convection-damping devices can simulate different aspects of microgravity. But emulation on earth is imperfect. For example, both types of levitators discussed below introduce forced convection.

The MMSL's electromagnetic levitator suspends conducting samples of up to 30 grams in a magnetic field trap. A radio frequency current is induced in the coil, producing a vertical magnetic field. The vertical field, which changes polarization with the same frequency as the current, induces eddy currents in the sample. These in turn create an oppositely directed vertical magnetic field about the sample, causing it to act like a small bar magnet. The strong field of the outer coil repels the magnetized sample, suspending it against the gravitational force. The eddy currents cause resistive
heating in the sample. As a result, heating and levitation are coupled.

The open-ended induction coil of the electromagnetic levitator is positioned over a one-second drop tube (Figure 1) so that a melted sample can be dropped and solidified during free fall. Both the levitation chamber and the drop tube can be operated either under vacuum (10^-6 Torr) or with an inert gas backfill. Instrumentation on the levitator/drop tube apparatus includes two-color pyrometers to determine sample temperature during melting and free fall and position detectors along the length of the drop tube so that the drag forces caused by backfill gases can be known quantitatively. Currently under development is the capacity for continuous, high speed, non-contact temperature monitoring during levitation and freefall. Since large samples will not solidify during the one second of drop time available, small sample handling facilities are being designed.

Nonconducting samples such as ceramics and glasses can be levitated acoustically. The MMSL currently owns a single axis, 15 kHz acoustic levitator with a 150 decibel sound pressure level (Figure 2). This functional duplicate of the flight-version acoustic levitator uses a reflecting surface to produce standing waves. Low pressure nodes hold the sample in position, thus sample diameters cannot exceed half the wavelength of the acoustic field, which is about 6 mm. The advantage of a reflecting acoustic levitator is that it is self-adjusting. As the temperature rises and the node position moves, the sample moves with it. The acoustic levitator has an independently controlled furnace which can provide temperatures up to 1600°C, and it operates at one atmosphere of gas pressure. The maximum cooling rate is approximately 30°C/sec.

The ability of the acoustic levitator to support samples depends on several parameters. It is proportional to the sample volume, hence density is critical. Reflecting acoustic levitators have been used successfully to support platinum at room temperature, but the levitating force is also proportional to T^-5/2 (ref. 1). Thus, at the high temperatures needed to melt glasses and ceramics, levitation in one-g becomes impossible for most materials. In addition to low density, high surface tension is necessary to confine droplets against the deformations caused by the combined forces from the acoustic and gravitational fields. The MMSL's present equipment can be employed to examine positioning requirements for proposed space experiments or to determine the effects of acoustically-induced vibration of mechanically suspended samples.

Axial restoring forces in the acoustic levitator are strong, but those in the radial direction, which are provided only by the pressure of gas rushing past the sample, are weak (ref. 1). The resulting containment difficulties are being addressed, along with density restrictions, in the MMSL's work with Intersonics, Inc. to design a high-pressure, multi-axis acoustic levitator; the new model should be useful for terrestrial experimentation.
Figure 1. One-second instrumented drop tube showing the positions of the sample position sensors, pyrometers, and electromagnetic levitator vacuum.

Figure 2. Single axis acoustic levitation furnace.
Another low gravity emulating device in the MMSL is the high temperature directional solidification furnace, Figure 3, which acts against gravity by using a five kilogauss magnetic field to damp convection currents in conducting samples. If a molten binary system is subjected only to the earth's gravitational field, density differences caused by the horizontal temperature gradients result in convective flow. However, if the sample is conducting, the convection currents are capable of interacting with a magnetic field. In the high temperature directional solidification furnace, a DC-powered electromagnet is oriented so that its field runs horizontally across the sample. The magnetic field causes the melt to traverse tighter loops, decreasing convective mixing. The high temperature directional solidification furnace is operable up to 1100°C.

The Lewis Metals Science Branch solidification team is using the high temperature directional solidification furnace to examine the magnitude of the effect of magnetic damping on alloys. Lead alloy samples of several hundred grams will be quenched after a stable solid/melt interface is obtained. The morphology and solute distribution of samples processed with the magnetic field will be compared to the characteristics of those processed without it.

ONE-G TESTING AND RESEARCH EQUIPMENT

The Microgravity Materials Science Laboratory is interested in providing quantitative data to aid researchers in maximizing the efficiency and usefulness of their flight apparatus. Crystal growth, long more of an art than a science, is under close examination in the MMSL. A two-zone resistive heater wrapped about a quartz furnace tube provides a transparent crystal growth apparatus which permits observation during the growth process. The samples are encased in quartz ampoules which are pulled through the furnace tube by a guide wire (Figure 4). The crystal growth apparatus has been used for both vapor growth, with mercurous chloride, and growth from the melt, with lead chloride. Ampoules are approximately one centimeter in diameter and ten centimeters in length. Axial temperature gradients are varied between 0 and 40°C per centimeter; growth rates range between 50 nm/sec and 10 microns/sec. Researchers in the MMSL have determined the optimum values of these parameters for both mercurous chloride and lead chloride crystals when grown on earth, and the information will be used by an industrial customer in planning future microgravity research.

Also used for one-g testing and development, the MMSL's bulk undercooling furnace is designed to study the effects of undercooling on the microstructure of metal alloys. Uses include a demonstration of the feasibility of obtaining significant undercooling of a large sample (up to 100 grams) and determining the best undercooling method in order to provide guidelines for space hardware design. Typical runs have used cooling rates of 0.04°C/sec.
(a) Overall view of HTDSF showing placement of furnace assembly in the magnet.  
(b) Detail drawing of furnace assembly.

Figure 3. High temperature directional solidification furnace.

Figure 4. Crystal growth furnace. Ampoule shown positioned between two heater zones.
Recent experiments using lead-tin alloys have provided insight into recalescence phenomena. Metallographic examination of undercooled samples (about 20°C) revealed a concentration of lead dendrites at the bottom. Researchers were uncertain whether the alloy first separated due to density differences and then froze, or whether the lead dendrites first grew uniformly throughout the sample and the heat released during recalescence partially melted the dendrites, allowing them to fall to the bottom. A horizontal mesh was inserted in the sample to divide it in half, and metallographic examination revealed heaps of lead dendrites both at the bottom of the ampoule and on top of the screen. This indicated that dendrites formed throughout the sample early during solidification and partially melted immediately afterwards during the pronounced recalescence heating. Evidence that the liquid was compositionally uniform during undercooling has helped the researcher define his plans for work in a low-gravity environment.

FUNCTIONAL DUPLICATES OF FLIGHT HARDWARE

One of the charter functions of the Microgravity Materials Science Laboratory is to maintain functional duplicates of flight hardware. The acoustic levitator has already been described. In addition, the MMSL possesses an isothermal dendrite growth apparatus functionally identical to the flight version and a model of the general purpose furnace used in the 1970's during the sounding rocket program and, later, on space shuttle flights. The general purpose furnace is a three-zone, resistance furnace with nine thermocouples available for monitoring the walls and heater bands. Additionally, the sample canister is capable of handling up to twelve thermocouples within a sample. The flight furnace has three sample cavities; the MMSL version has one, although its data collection capabilities are more extensive. This is important because a well-documented, ground-based experiment can be used to provide a one-g baseline as well as to predict problems which may be encountered in orbit. The MMSL's functional duplicate of the general purpose furnace can be used to help predict what power levels should result in the desired heating profiles. As the general purpose furnace is replaced with more advanced flight hardware, the MMSL furnace will be modified to provide corresponding thermal field emulation.

Solidification phenomena are being studied with the isothermal dendrite growth apparatus, a prototype of the flight hardware being developed for Dr. Martin Glicksman of Rensselaer Polytechnic Institute. This apparatus (Figure 5) will be used to test his theoretical model of dendrite formation by providing unambiguous data for dendrite growth rates and tip radii. Experiments in reduced gravity will allow a valuable extension of the range of data against which Glicksman's hypothesis may be tested. Gravity interferes with free dendrite growth on earth. A dendrite growing horizontally will become deformed as the surrounding fluid is warmed by recalescence heating. The less dense, warm fluid flows upward, retarding crystal
formation above. Cool fluid rushes in to fill the space left by the rising warm flow, causing the bottom side to grow faster. This results in the uneven growth shown in Figure 6. Crystals growing parallel to the gravitational force, while not subject to severe losses of symmetry, grow downward more quickly and upward more slowly than we expect they will in the ideal, zero-gravity case.

The isothermal dendrite growth apparatus allows the growth of a single free dendrite into an undercooled melt to be observed and photographed. Its outstanding features are excellent temperature control (±0.002°C at 60°C) and the ability to observe the growth of microscopic crystals. Succinonitrile, an organic which crystallizes in a BCC crystalline array, is used as the model material because its transparency makes it amenable to visual analysis. This proof-of-concept hardware was built in the MMSL prior to construction of the flight apparatus.

ADVANCED TECHNOLOGY DEVELOPMENT

The Microgravity Materials Science Laboratory is currently working in conjunction with Marshall Space Flight Center and JPL on two Advanced Technology Development projects, improved furnace concepts and a laser light scattering instrument. One goal of the furnace study is improvement of linearity of axial temperature gradients and furnace efficiency. The effect of using a heater in the top endcap of the sample cartridge while air-cooling its bottom endcap is being examined as a means of obtaining good axial temperature gradients. Different surface configurations of the bottom endcap are under investigation. Thermal shielding is also an important aspect of the study. Since furnace power requirements depend sensitively on the emissivity of their thermal shields, a separate study is planned detailing the radiative properties of several engineering materials as a function of temperature. Other innovative furnace designs involving interface position control in zone-melting and Bridgman furnaces are under examination by the MMSL and Case Western Reserve University.

The goal of the second Advanced Technology Development project is to determine the desirability, feasibility and minimum design requirements for a space station based laser light scattering instrument. Such instruments find wide use terrestrially for characterization of submicron particles. Using the current ground-based equipment as a springboard, the MMSL will specify a small, reliable laser light scattering instrument which may lead to a flight version. This basic laboratory tool could be used to measure particle size distribution and the shape of polymeric and biological molecules too delicate to survive the return trip from orbit to earth. Plans include the use of an array of solid state photodiodes rather than the conventional, swinging-arm goniometer, fiber optics to make component arrangement more flexible, and small, solid state lasers.
Figure 5. Growth chamber of the isothermal dendrite growth apparatus.

Figure 6. Horizontally grown dendrite showing deviation from symmetry caused by the free convection inevitably present in one-g.
CHARACTERIZATION LABORATORIES

In addition to the equipment already described, the MMSL has a range of standard analytical tools. A small metallography laboratory contains the basic equipment needed to cut, mount, grind, polish, and etch specimens. A light microscope, macroscope and cameras, all capable of up to 1000x magnification, can aid researchers in preliminary examinations of surface morphology.

Open for research since February 1987, the ceramics laboratory provides opportunities to study samples before and after processing. Electrical resistance, critical cooling rate, softening point, and viscosity are among the capabilities of the equipment. A range of furnaces are also available.

The MMSL's polymers lab, opening in 1988, will perform an analogous service for polymer scientists. A laser light scattering instrument can be used to monitor polymerization reactions and determine the size and shape of particles ranging between 0.001 and 3 microns. Rheology equipment, a Fourier transform infrared spectrometer, vapor pressure and membrane osmometers, and a thermal analysis system will also be available.

In addition to its own labs, the MMSL can offer access to the extensive facilities of Lewis Research Center's Materials Division, of which it is a part. Microstructural characterization techniques include optical microscopy, electron optics, and x-ray diffraction. A chemical characterization laboratory can make qualitative and quantitative analyses of elemental constituents for trace levels as low as parts per billion. The polymers and ceramics branches offer extended instrumentation and expertise to interested researchers.

COMPUTATIONAL FACILITIES

NASA Lewis Research Center owns a number of large, mainframe computers, which are accessible to visiting researchers. In addition, the MMSL includes access to an engineering workstation dedicated to microgravity process modelling. Because use of this computer is limited to a small group of users, large programs can be run in lengths of time comparable to those required by Lewis Research Center's supercomputer. The lab has numerous small computers used for experiment control and as personal computers. As a result, word processing, CAD, and plotting capabilities are available to visiting scientists. Mathematical modelling is also among the services offered by the MMSL.

FUTURE EQUIPMENT

Future acquisitions of the MMSL will depend in part upon user input. If a microgravity researcher has an experiment in mind, but the needed equipment is unavailable, the MMSL may construct or purchase the required apparatus.
ACCESS TO FLIGHT OPPORTUNITIES

Because of the range of its ground-based equipment and its staff of knowledgeable engineers, the Microgravity Materials Science Laboratory is a logical place to develop flight experiments. In addition, the MMSL can aid access to true low-g facilities. Lewis Research Center houses drop towers with free fall times of 2.2 and 5 seconds. The 20 foot diameter of the 5 second tower makes it amenable to large experimental packages. Flying in a parabolic trajectory, the Lewis Research Center Learjet can offer 15-20 seconds of microgravity. Promising experiments may be further developed for performance on orbit.

USE OF THE MICROGRAVITY MATERIALS SCIENCE LABORATORY

The Microgravity Materials Science Laboratory is open to users from U.S. industry, academia or government. It is free of charge to researchers who will publish their results in open literature within two years of the completion of their work in the MMSL. In the case of proprietary research, a reasonable charge will be negotiated for use of the laboratory.

The process for gaining access to the MMSL is outlined in Figure 7. First, potential use should be discussed with the laboratory staff. After agreement that the proposed project is within the range of activities undertaken in the lab, a short proposal should be developed. The submitted proposal outlines the experiment and its scientific basis and clearly explains the effects of microgravity on the investigation. Following a favorable review of the proposal, an entry agreement is negotiated detailing the level of effort to be provided by NASA.

REFERENCE

ENTRY TO MICROGRAVITY MATERIALS SCIENCE LABORATORY

IDEA FROM INDUSTRY, GOVERNMENT, OR UNIVERSITY → INFORMAL DISCUSSION WITH MMSL STAFF → BRIEF, FORMAL PROPOSAL SUBMITTED TO AND EVALUATED BY NASA LEWIS → ENTER MMSL

NEGOTIATE AGREEMENT

NON-PROPRIETARY RESEARCH (TO BE PUBLISHED WITHIN TWO YEARS)

PROPRIETARY RESEARCH

NEGOTIATE CONTRACT, FEE FOR USE OF LABORATORY

Figure 7. Gaining access to the Microgravity Materials Science Laboratory. Lab Manager: Thomas K. Glasgow; tel. (216) 433-5013; Mail Stop 105-1, NASA Lewis Research Center.