Overview of NASA’s Microgravity Materials Science Program

patton.downey@nasa.gov

Presenter: James Patton Downey
NASA Marshall Space Flight Center
History

- The microgravity materials program was nearly eliminated in the middle of the aughts due to budget constraints
  - Hardware developments were eliminated.
- Some investigators with experiments that could be performed using ISS partner hardware received continued funding.
- Partnerships were established between US investigators and ESA science teams for several investigations.
  - ESA conducted peer reviews on the proposals of various science teams as part of an ESA AO process.
  - Assuming he or she was part of a science team that was selected by the ESA process, a US investigator would submit a proposal to NASA for grant funding to support their part of the science team effort.
- In a similar manner, a US materials investigator (Dr. Rohit Trivedi) is working as a part of a CNES selected science team.
- As funding began to increase another seven materials investigators were selected in 2010 through an NRA mechanism to perform research related to development of Materials Science Research Rack investigations.
  - One of these has since been converted to a Glovebox investigation
Dr. Rohit Trivedi has performed a series of solidification experiments in the DECLIC Directional Solidification Insert in the early part of 2011.

- Observed time dependent behavior showed cyclical patterns of expanding then contracting cellular tip radii

Two samples have been processed in the Materials Science Research Rack in support of Dr. David Poirier’s investigation.

- February 2, 2010 using the ESA Low Gradient Furnace / The experiment did not achieve the desired thermal gradient.
- January 1, 2011 using the ESA Solidification with Quench Furnace / Problems occurred due to a crucible to sample reaction
- Problems with the SQF insert have delayed processing additional samples
- A third sample is to be processed for Dr. Poirier’s investigation in the second half of 2012.
The microgravity materials program investigators are developing experiments to be performed on ISS in the following facilities:

- Glovebox (1 investigator)
- DECLIC (1 investigator)
- Electro-Magnetic Levitator (3 investigators)
- Materials Science Research Rack (8 investigators)

Three other investigators are performing calculations or modeling in support of flight investigations.
Thermo-Physical Properties of Undercooled Melts

• Dr. Ken Kelton, Washington University St. Louis / Quasi-Crystalline Undercooled Alloys for Space Investigation
  – ground based research completes in 2012
  – collaboration with ESA THERMOLAB investigation

• Dr. Ken Kelton, Washington University St. Louis / THERMOLAB and ICOPROSOL
  – Flight experiments in 2012-2014 utilizing the Electro-Magnetic Levitator
  – collaboration with ESA THERMOLAB and ICOPROSOL investigations

• Dr. Doug Matson, Tufts University / The Role of Convection and Growth Competition in Phase Selection in Microgravity
  – flights experiments in 2012-2014 utilizing the EML
  – collaboration with ESA THERMOLAB investigation

• Dr. Doug Matson, Tufts University / Electromagnetic Levitation Flight Support for Transient Observation of Nucleation Events
  – flight experiments in 2012-2014 utilizing EML
  – collaboration with ESA PARSEC investigation

• Dr. Robert Hyers, University of Massachusetts / Unified Support for THERMOLAB, ICOPROSOL, and PARSEC
  – flight experiments in 2012-2014 utilizing EML
  – collaboration with ESA THERMOLAB, ICOPROSOL, and PARSEC investigations
Materials Science Investigations

Metals and Alloys

• **Dr. David Poirier, University of Arizona / Comparison of Structure and Segregation in Alloys Directionally Solidified in Terrestrial and Microgravity Environments**
  – flight experiments in 2011-2012 utilizing the MSRR
  – collaboration with ESA MICAST and CETSOL investigations

• **Dr. David Poirier, University of Arizona / Effect of Varying Convection on Dendrite Morphology and Macro segregation**
  – flight experiments starting in 2014 utilizing the MSRR
  – collaboration with ESA MICAST and CETSOL investigations

• **Dr. Rohit Trivedi, Iowa State University / Dynamic Selection of Three-Dimensional Interface Patterns in Directional Solidification**
  – flight experiments in 2010-2011, reflight in 2014 utilizing the DECLIC facility’s Directional Solidification Insert (DSI)
  – collaboration with CNES DSI investigation

• **Dr. Ralph Napolitano, Iowa State University / Solidification Along an Eutectic Path in Ternary Alloys**
  – flight experiments starting in 2011 utilizing the MSRR
  – collaboration with ESA’s SETA investigation

• **Dr. Johnathan Dantzig, University of Illinois / Modeling Peritectic Microstructure Formation during Directional Solidification in Space and on Earth**
  – collaboration with ESA’s METCOMP investigation
Materials Science Investigations

Metals and Alloys

• Dr. Randall German, San Diego State University / Multi-Scale Modeling and Experimentation on Liquid Phase Sintering in Gravity and Microgravity Environments
  – flight experiment in 2015 utilizing the MSRR

• Dr. Douglas Hofmann, JPL / Study of Mushy-Zone Development in Dendritic Microstructures with Glass-Forming Eutectic Matrices
  – flight experiment in 2015 utilizing the MSRR

• Dr. Peter Voorhees, Northwestern University / Coarsening of Dendritic Solid-Liquid Mixtures: The Low Volume Fraction Limit
  – flight experiment in 2015 utilizing the MSRR

• Dr. Douglas Swenson, Michigan Technological University / Systematic Investigation of Organized Elongated Pore Formation in Invariant Liquid to Solid Metal Plus Gas Transformations
  – flight experiment in 2015 utilizing the MSRR

• Dr. Christoph Beckermann, University of Iowa / Effect of Convection on Columnar-to-Equiaxed Transition in Alloy Solidification
  – collaboration with ESA CETSOl team
  – flight experiments starting in 2011 Utilizing the MSRR

asgsr 2012
Materials Science Investigations

Metals and Alloys

• Dr. Alain Karma, Northeastern University / Integrated Computational and Experimental Studies of Complex Dendritic Microstructure Development during Directional Solidification of Metallic Alloys
  – provides calculations for ESA CETSOL investigation
  – flight experiments starting in 2011

Semiconductors/Electronic and Photonic Materials

• Dr. Jeff Derby, U. of Minnesota / Modeling of Particle Transport in the Melt and its Interaction with the Liquid Solid Interface
  – flight in 2016 utilizing MSRR
  – supports ESA’s SISSI investigation

• Dr. Ching-Hua Su, NASA MSFC / Crystal Growth of Ternary Compound Semiconductors
  – flight in 2014 utilizing MSRR
  – collaboration with ESA’s CdTe investigation

• Dr. Martin Volz, NASA MSFC / Reduction of Defects in Germanium Silicon
  – flight in 2014 utilizing MSRR
  – collaboration with ESA’s GeSi investigation
## Materials Research Projected Launch Schedule

<table>
<thead>
<tr>
<th>Experiment PI</th>
<th>2013</th>
<th>2014</th>
<th>2015</th>
<th>2016</th>
<th>2017</th>
<th>2018</th>
</tr>
</thead>
<tbody>
<tr>
<td>DECLIC</td>
<td>▲</td>
<td></td>
<td></td>
<td>▲</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Trivedi</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>EML</td>
<td>▲</td>
<td>▲</td>
<td>▲</td>
<td>▲</td>
<td>▲</td>
<td>▲</td>
</tr>
<tr>
<td>Hyers</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>EML</td>
<td>▲</td>
<td>▲</td>
<td>▲</td>
<td>▲</td>
<td>▲</td>
<td>▲</td>
</tr>
<tr>
<td>Matson</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>EML</td>
<td>▲</td>
<td>▲</td>
<td>▲</td>
<td>▲</td>
<td>▲</td>
<td>▲</td>
</tr>
<tr>
<td>Kelton</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>MICAST</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>▲</td>
</tr>
<tr>
<td>Poirier</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>RDGS</td>
<td></td>
<td>▲</td>
<td>▲</td>
<td>▲</td>
<td>▲</td>
<td>▲</td>
</tr>
<tr>
<td>Volz</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>GTCS</td>
<td></td>
<td>▲</td>
<td>▲</td>
<td>▲</td>
<td>▲</td>
<td>▲</td>
</tr>
<tr>
<td>Su</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SETA</td>
<td></td>
<td></td>
<td>▲</td>
<td>▲</td>
<td>▲</td>
<td>▲</td>
</tr>
<tr>
<td>Napolitano</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>CETSOUL</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>▲</td>
</tr>
<tr>
<td>Beckermann</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>GEDS</td>
<td></td>
<td>▲</td>
<td>▲</td>
<td>▲</td>
<td>▲</td>
<td>▲</td>
</tr>
<tr>
<td>German</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>CDM</td>
<td>▲</td>
<td>▲</td>
<td>▲</td>
<td>▲</td>
<td>▲</td>
<td>▲</td>
</tr>
<tr>
<td>Voorhees</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>FOG</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>▲</td>
</tr>
<tr>
<td>Swenson</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>FAMIS</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>▲</td>
</tr>
<tr>
<td>Hofman</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

- ▲ EML Sample
- ● SQF LMR Cartridge
- ○ SQF US made Cartridge
- ▲ LGF US made Cartridge
- △ DSI
- ●●● CSLM samples

asgs92012
Microgravity Science Glovebox (MSG)

**Removable Side Ports**
16” diameter on both Left and Right sides for setting up hardware in Work Volume

**Glove Ports**
Four identical glove ports are located on the left and right side loading ports and the front window

**DC Power Switching And Circuit Breakers**

**Front Window Glove Ports**
Four 6” diameter glove ports can be fitted with any of three different sized gloves or blanks

**Core Facility**
Retractable Core Facility includes the Work Volume, Airlock, Power Distribution & Switching Box, and the Command and Monitoring Panel

**Airlock**
Provides a “Pass Through” for hardware to enter the Work Volume without breaking Containment. The lid of the Air Lock opens up into the floor of the Work Volume

**Airlock Glove Port with Blank**
A Single 4” diameter glove port can also be fitted with any of three different sized gloves or a blank

**Stowage Drawers**

Engineering Unit Located at MSFC
Coarsening of Solid/Liquid Mixtures

Hardware capabilities
- 185C Processing
- 4RTDs
- Quench via an air pressurized water spray through a burst disc
- 4 samples

Above: Sample Processing Unit and Electronics Control Unit (power and data) in Glovebox

Right: Sample Processing Unit without cover
Microgravity Science Glovebox (MSG)

- Work Volume (WV) - Volume
  - 0.255 m³ = 255 liters
- Work Volume - Dimensions
  - 906mm wide x 637mm high
  - 500mm deep (at the floor)
  - 385mm deep (at the top)
- Maximum size of single piece of equipment in WV (via side access ports)
  - 406mm diameter
- Maximum size of single piece of equipment in WV (via the airlock)
  - 254 x 343 x 299 mm
- Payload Attachment
  - M6 threaded fasteners in floor, ceiling, & sides
- Power available to investigation
  - +28V DC at useable 7 amps
  - +12V DC at useable 2 amps
  - -12V DC at useable 2 amps
  - +5V DC at useable 4 amps
  - +120V DC at useable 8.3 amps
- Maximum heat dissipation
  - 1000W Total
    - 800W from coldplate
    - 200W from air flow
- General illumination
  - 1000 lux @ 200mm above WV floor
- Video
  - 4 color Hitachi HV-C20 cameras
  - 2 Sony DSRV10 Digital Recorders
  - 2 Sony GV-A500 Analog 8mm Recorders
- Data handling connections
  - T61P Laptop Computer
  - Two RS422-to-MSG for investigations
  - One MIL-BUS-1553B-to-MSG for communication via MLC
  - Ethernet LAN 2
- Filtration
  - 12 HEPA/charcoal/catalyst WV filters
- 1 HEPA/charcoal/catalyst Airlock filter
- Up to Two Levels of Containment
  - Physical barrier of MSG structures, gloves, etc.
  - Negative pressure generated by MSG fans.
- Other resources available /Gaseous Nitrogen, Vacuum
DECLIC - Dispositif pour l'Etude de la Croissance et des Liquide Critiques

DECLIC is a multi-user facility to investigate low and high temperature critical fluids behavior, chemical reactivity in supercritical water, directional solidification of transparent alloys, and more generally transparent media under micro-gravity environment on board the International Space Station (ISS).

Three inserts exist
• Directional Solidification Insert
• High Temperature Insert
• Analysis of (Critical) Liquids Insert

Graphics and description on this and the following page are taken from CNES web sites.
DECLIC in an EXPRESS Rack
The DECLIC Directional Solidification Insert has the following properties/capabilities

- **Samples** – Succinonitrile/water, 1 cm diameter
- **Hot Zone** – Maximum temperature of 160°C with ±2mK/hr stability
- **Cold Zone** – Minimum temperature of -30°C with ±2mK/hr stability
- **Gradient** – Up to 70°C/cm
- **Translation Rate** – 0.1-30µm/sec with 1% stability over 100mm of travel
- **Axial Wide Field of View** – 7mm with 7µm resolution
- **Axial Narrow Field of View** – 3mm with 5-6µm resolution
- **Perpendicular Wide Field of View** – 7.8mm with 36µm resolution
- **Perpendicular Narrow Field of View** – 7mm with 16µm resolution
- **Interferometry Field of View** – 7mm with 7-13µm resolution
Electro-Magnetic Levitator

Located in a European Drawer Rack inside the Columbus Module

- Gas Module
- Levitation Power Supply/Water Pump Module
- Experiment Module (vacuum chamber, RF coil, sample chamber containing 18 samples, diagnostics)
- Experiment Controller Module

At Left: Levitation Coil and Sample Holder

Photo and Figures on this and following chart from ESA documents

asgr 2012
Electro-Magnetic Levitator

Experiment Classes:
- class A: undercooling and nucleation triggering
  - fast solidification possible (up to 1 MHz temperature measurement required)
- class B: modulation calorimetry and radiometric measurements
  - heater modulation
- class C: surfaces tension and viscosity
  - heater pulses
  - frequency sweep
- class D: thermal expansion
- class E: electrical conductivity

Note: class B, D, E also on solid sample for calibration purposes

Sample temperature $T$
- $T_{\text{superheating}} = 800°C - 2400°C$
- $T_{\text{melting}} = 600°C - 2200°C$
- $T_{\text{undercooling}}$

Holding time

Homogenization/purification

Melt Cycle 1 min - 30 min

Sample positioning and sample processing

Repetition of melt cycle on command

Start of random solidification
Or controlled nucleation by trigger needle

Sample positioning

Delta T = $\Delta T = 0 K - 500K$
Electro-Static Levitator

- The MSFC ESL facility provides an ideal method for study of high-temperature materials.
- Levitated samples do not contact a container and will not be contaminated by the container or react with it. Only the sample is heated, not the instrument and instrumentation.
- The ESL can provide measurements of thermophysical properties, which include creep strength, density and thermal expansion, emissivity, specific heat, phase diagrams, viscosity and surface tension.
- Data can be obtained at ultra-high temperatures for materials being developed for propulsion applications.
- Samples: 2-3 mm diameter spheres (30-70 mg)
- Heated by lasers: 200W Nd:YAG or 300W CO₂
Materials Science Research Rack (MSRR)

Project Manager: Shawn Reagan/MSFC

Status:
• Operational aboard the ISS

Purpose:
• To provide a facility onboard the ISS to conduct materials science research/technology experiments

Relevance/Impact:
• The MSRR can be utilized for multi-Program tasks
• The MSRR accommodates the operation of the European Space Agency Materials Science Laboratory (MSL)
Materials Science Laboratory

Built by EADS Astrium for ESA

Status:
• Operational aboard the ISS with the LGF and SQF

Purpose:
• Provide operational support for furnaces including
  – Low Gradient Furnace
  – Solidification and Quenching Furnace

Relevance/Impact:
• The MSL can be utilized for multi-Program tasks

http://www.spaceflight.esa.int/users/materials/facilities/facilities/msl.html
The Solidification and Quench Furnace and the Low Gradient Furnace have the following features

- Heater elements that operate from 500-1400°C
- Rotating magnetic fields
- 150mm translation
- Approximately 100mm of sample processing
- Solidification translation rates from 0.01μm/sec to 0.2mm/sec
- 26 mm ID for LGF sample crucibles/ampoules, 16 ID for SQF
- Ability to interface with up to 12 thermocouples in the sample cartridge assemblies

The figures and photos on the following three pages are from ESA documents
Low Gradient Furnace

- Cold cavity
- Adiabatic zone
- Hot cavity

200 mm
Liquid Metal Ring
Sample Cartridge Assemblies

• The samples processed in the MSRR furnace inserts use cartridges to provide a required level of chemical containment of the experimental samples.

• US program is undertaking the design and manufacturing of Sample Cartridges Assemblies for some of the US investigators who are developing MSRR experiments.
  • Currently, this is the only flight hardware development in the NASA microgravity materials program.

• Some cartridges will still be bartered from ESA.
  • Experiments that desire a quick quench are best accommodated by the unique, proprietary cartridge design developed by ESA for the SQF.

• The US built cartridge tubes are to be constructed via vacuum plasma spray process and will have the following features
  • A high emissivity Zirconium Boride outer coating to provide good thermal exchange with the furnace and enable relatively high gradients
  • A Mo-Re core to provide high temperature capability
  • An Alumina inner liner to provide good chemical compatibility with most metals
At Top: Possible Cartridge Layout for Dr. German’s investigation

At Right: Tube cross-section

Sample Cartridge Assemblies

Instrummented head:
- Pressure sensor
- Pt100 T/C for ref temp
- SACA ID resistor (ID code)
- SACA temp limit resistors
- Touch temp T/C
- Microswitch

Mounting surface
Preload spring
Vacuum pinch tube

OD = 25.6 mm

Zirconium Boride (.127 mm thick)
Base Metal Mo41Re (.762 mm thick)

Data cable
End cap
465 mm
Thermocouples (6)
Crucibles with samples (7)
Mo41Re cartridge
Ceramic spacers

Asgsr 2012