Development of NASA’s Sample Cartridge Assembly: Summary of GEDS Design, Development Testing, and Thermal Analyses

Brian O'Connor, Deborah Hernandez, Linda Hornsby, Maria Brown, and Kathryn Horton-Mullins
NASA Marshall Space Flight Center

Presented By
Deborah Hernandez

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Outline

• Background of ISS Material Science Research Rack
• NASA SCA Design
• GEDS Experiment Ampoule Design
• Development Testing Summary
• Thermal Modeling and Analysis
• Summary
• References
ISS Material Science Research Rack (MSSR)

- **ISS MSRR1** - Materials science research in low gravity
  - Destiny Laboratory, launched in 2009
- ** ESA’s Material Science Lab (MSL) -** Built by ESA
  - Process material samples at high temperatures
- **Low Gradient Furnace (LGF) insert** - Bridgman style furnace with multiple heater zones
  - Vacuum furnace
  - Two hot cavities separated by an adiabatic zone
  - Max operating temperature of 1400°C
NASA SCA Design

- **NASA SCA cartridge**
  - Single pressurized volume
  - Helium or argon gas fill
  - Reuse of sealed container
  - Interface to MSL Intermediate Support Plate (ISP)

- **Instrument Head**
  - 416 CRES
  - Ultrahigh vacuum seal with bolted joints
    - Conflat flanges comprised of a copper gasket and knife-edge flange

- **Cartridge Tube**
  - Vacuum Plasma Sprayed
  - Molybdenum-Rhenium with an Alumina liner and Zirconium Boride emissivity coating
GEDS Experiment Ampoule Design

- **GEDS research (Dr. Randall German Principle Investigator)**
  - Determine the underlying scientific principles on how to forecast density, size, shape, and properties for liquid phase sintered

- **Seven samples in each GEDS SCA**
  - Compacted mixture of W, Ni, Cu, and Mn powders
  - Samples located in LGF isothermal hot zone
  - Dwell duration above 1200°C from 3 to 60 minutes
  - Sample stack Isothermality of +/- 5°C

- **Repeatable processing profile for each of 7 SCAs**
  - Controlled by LGF Sample Processing Program (SPP)

- **Vacuum Ampoule**
  - Cartridge filled with argon to prevent ampoule permeation
  - High purity alumina ceramic crucibles
Development Testing Failure

- Development Test MUGS Results
  - The Tantalum Sheath of the Type N Thermocouples were disintegrated
  - Ampoule failure in three places by three modes
    A. Ampoule chipped at alumina spacer interface
    B. Ampoule body broken with marks at TC locations
    C. Contact at the sphere ampoule end to alumina end plug
## Development Testing Summary

<table>
<thead>
<tr>
<th></th>
<th>MUGS</th>
<th>MUGS V</th>
<th>MUGS VI</th>
<th>MUGS VII</th>
<th>MUGS VIII</th>
<th>G2</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Test Date</strong></td>
<td>July 2016</td>
<td>November 2016</td>
<td>December 2016</td>
<td>January 2017</td>
<td>March 2017</td>
<td>May 2017</td>
</tr>
<tr>
<td><strong>Processing Duration at heater set points</strong></td>
<td>22 minutes at 940°C/1210°C</td>
<td>78 minutes at 940°C/1210°C</td>
<td>78 minutes at 940°C/1210°C</td>
<td>78 minutes at 940°C/1210°C</td>
<td>18.5 minutes at 940°C/1210°C</td>
<td>95 minutes at 1130°C/1210°C</td>
</tr>
<tr>
<td><strong>Predicted sample dwell time</strong></td>
<td>9.3 minutes &gt; 1200°C</td>
<td>66 minutes &gt; 1200°C</td>
<td>66 minutes &gt; 1200°C</td>
<td>66 minutes &gt; 1200°C</td>
<td>3 minutes &gt; 1200°C</td>
<td>60+ minutes &gt; 1200°C</td>
</tr>
</tbody>
</table>
| **Design Revisions (design changes additive)** | None | • Re-aligned ampoule to bottom of cartridge  
• Reduced TCs number  
• Added quartz wool to 0.375 inch thickness  
• Reduced spring force to 10#  
• Increase fill pressure | • Upgrade ampoule quartz to 314°C  
• No samples or crucibles  
• Reduced spring force to 8# | • Added full ampoule back into SCA  
• Revised bakeout for sample and crucibles  
• Reduced spring force to 8# | • 2 Type S platinum sheath TCs  
• Reduced spring force to 4.8# | • Longer processing time  
• 4 Type S platinum sheath TCs  
• Increase cold zone setting from 940°C to 1130°C |
| **Test Result** | Ampoule failure in 3 modes | Ampoule failure in 1 mode | Ampoule intact | Ampoule intact, less sintering than expected | Ampoule intact | Ampoule intact |

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Design Change Summary

• SCA internal design
  – Reduced TCs number from 8 science TCs to 4 science TCs
  – Reduced spring force to 4.8#
  – Added quartz wool to 0.375 inch thickness

• GEDs Ampoule
  – Re-aligned ampoule to bottom of cartridge
  – Upgrade ampoule quartz to 314C
  – Revised bakeout for sample and crucibles

• Processing Changes
  – Increased processing time fro 78 minutes to 95 minutes
  – Increase cold zone setting from 940° C to 1130° C
Thermal Transient Modeling

- Atypical transient analysis performed to simulate LGF response
  - Understanding the LGF transient response is critical to GEDS experiment success
  - LGF thermal model was not applicable for transient analysis
- Thermal model development
  - Represents only internal components of GEDS SCA cartridge
  - No simulation of sample sintering
Thermal Transient Boundary Conditions

- Heated zone profile based on MUGS development test data to simulate LGF heating transient
  - Coupled with steady state GEDS model results for heating plateaus
• Thermal model over predicted sample dwell time in the sintering range
  – PI MUGS sample evaluation showed long duration samples were not fully sintered
• MUGS thermal model based on initial GEDS design
  – Design changes shifted samples about 0.5 inches toward the adiabatic zone with MUGS test design changes integrated
  – Unable to increase hot zone setting above 1210°C due to design limitations
• Used updated transient model to revised processing profile prior to ground preflight test, G2
  – Model updated to increase cold zone temperature to 1130°C to prevent end effects
  – Sample 1 dwell time = 60 minute calculated base on time > 1205°C for
GEDS Processing Predictions for G2

• Sample processing dwell time varies from 57 to 74 minutes depending on sample location
Predicted Sample Flight Processing

- Used G2 furnace inputs to generate flight sample predictions

<table>
<thead>
<tr>
<th>Furnace Hold Time (min)</th>
<th>Sample 4 &gt; 1205°C (min)</th>
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<tbody>
<tr>
<td>A</td>
<td>28</td>
</tr>
<tr>
<td>B</td>
<td>30</td>
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<tr>
<td>C</td>
<td>31</td>
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<td>J</td>
<td>87</td>
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<tr>
<td>K</td>
<td>95</td>
</tr>
</tbody>
</table>

GEDS Flight Sample 4 Transient Prediction

$T > 1200^\circ C$, Based on G2 Processing Profiles

Furnace Settings:
- Initial Temperature 1150°C, ramp rate 10°C/min
- Cold Zone at 1130°C, Hot Zone at 1210°C

Sample 4 min $>1205^\circ C$

Temperature $= 1205^\circ C$
Summary

• GEDS design development challenging
  – GEDS Ampoule design developed through MUGS testing
  – Short duration transient sample processing
  – Unable to measure sample temperatures

• MUGS Development testing used to gather data
  – Actual LGF like furnace response
  – Provided sample for sintering evaluation

• Transient thermal model integral to successful GEDS experiment
  – Development testing provided furnace response
  – PI evaluation of sintering anchored model evaluation of processing durations
  – Thermal transient model used to determine flight SCA sample processing profiles
References


