# 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

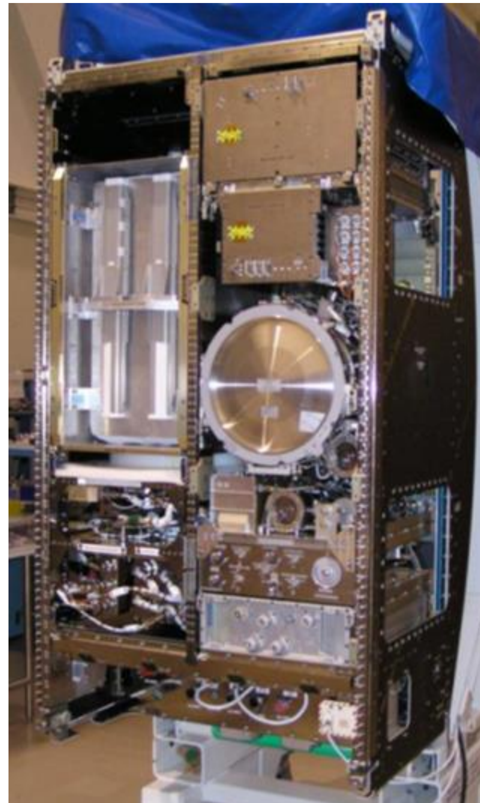
Thermal & Fluids Analysis Workshop
TFAWS 2017
August 21-25, 2017
NASA Marshall Space Flight Center
Huntsville, AL

# 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



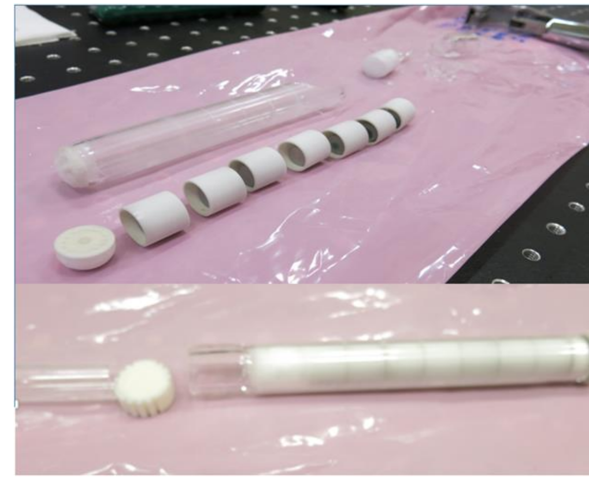
# NASA SCA Design

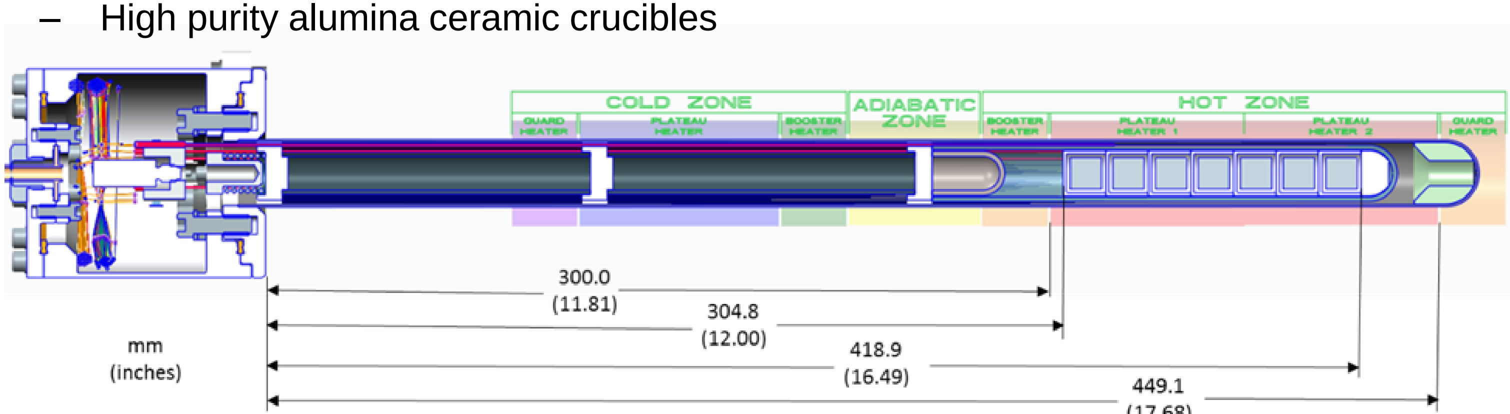
- NASA SCA cartridge
  - Single pressurized volume
  - Helium or argon gas fill
  - Reuse of sealed container
  - Interface to MSL Intermediate Support Plate (ISP)

- 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

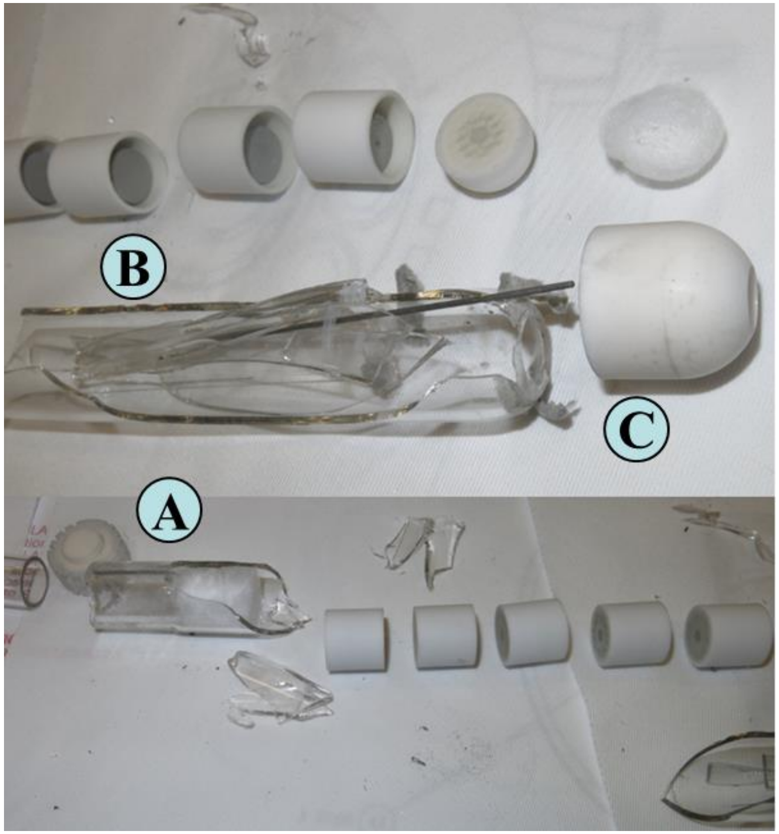
- GEDS research (Dr. Randall German Principle Investigator)
  - Determine the underlying scientific principles on how to forecast density, size, shape, and properties for liquid phase sintered
- Seven samples in each GEDS SCA
  - Compacted mixture of W, Ni, Cu, and Mn powders
  - Samples located in LGF 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 a TC locations
    C. Contact at the sphere ampoule end to alumina end plug




**MUGS Ampoule Post Test**

# Development Testing Summary

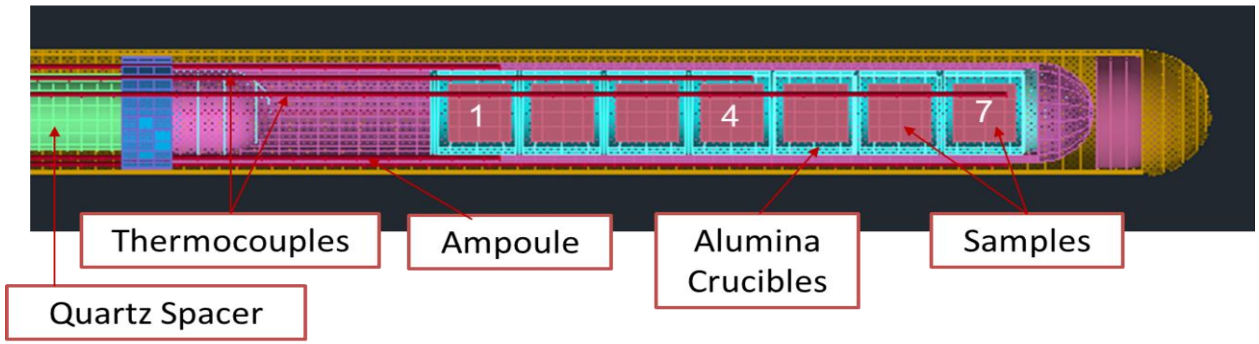
| | MUGS | MUGS V | MUGS VI | MUGS VII | MUGS VIII | G2 |
|---|---|---|---|---|---|---|
| Test Date | July 2016 | November 2016 | December 2016 | January 2017 | March 2017 | May 2017 |
| Processing Duration at heater set points | 22 minutes at 940°C/ 1210°C | 78 minutes at 940°C/ 1210°C | 78 minutes at 940°C/ 1210°C | 78 minutes at 940°C/ 1210°C | 18.5 minutes at 940°C/ 1210°C | 95 minutes at 1130°C/ 1210°C |
| Predicted sample dwell time | 9.3 minutes > 1200°C | 66 minutes > 1200°C | 66 minutes > 1200°C | 66 minutes > 1200°C | 3 minutes > 1200°C | 60+ minutes > 1200°C |
| Design Revisions (design changes additive) | None | • Re-aligned ampoule to bottom of cartridge<br>• Reduced TCs number<br>• Added quartz wool to 0.375 inch thickness<br>• Reduced spring force to 10#<br>• Increase fill pressure | • Upgrade ampoule quartz to 314C<br>• No samples or crucibles<br>• Reduced spring force to 8# | • Added full ampoule back into SCA<br>• Revised bakeout for sample and crucibles<br>• Reduced spring force to 5.2# | • 2 Type S platinum sheath TCs<br>• Reduced spring force to 4.8# | • Longer processing time<br>• 4 Type S platinum sheath TCs<br>• 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 |

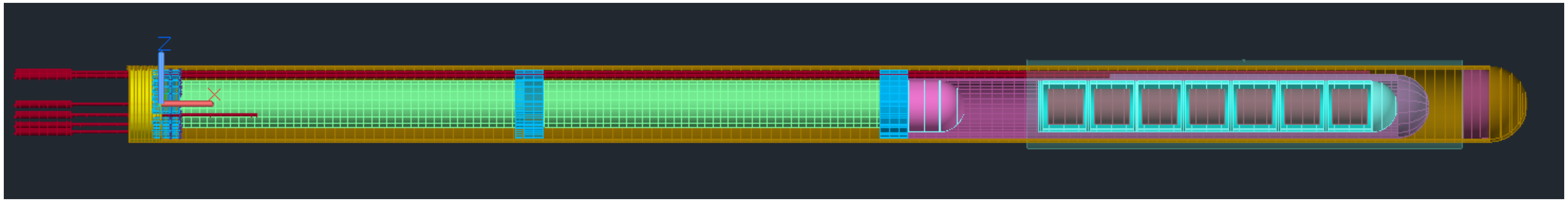
# Design Change Summary

- 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

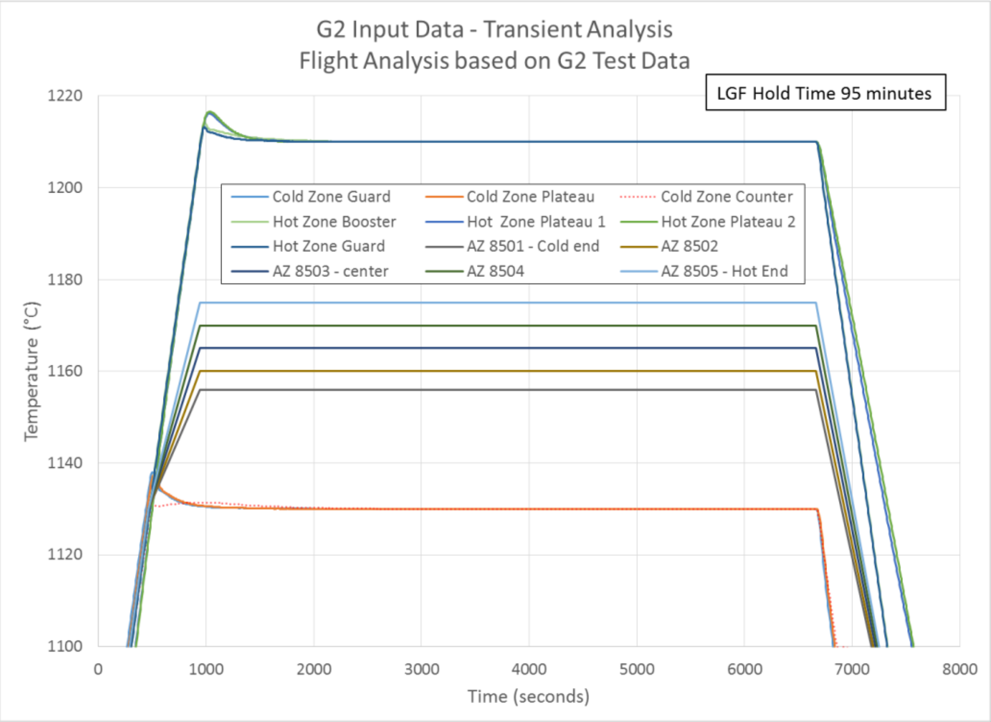
- Atypical transient analysis performed to simulate LGF response
  - Understanding the LGF transient response is critical to GEDS experiment success
  - LGF thermal model was not applicable for transient analysis
- Thermal model development
  - Represents only internal components of GEDS SCA cartridge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

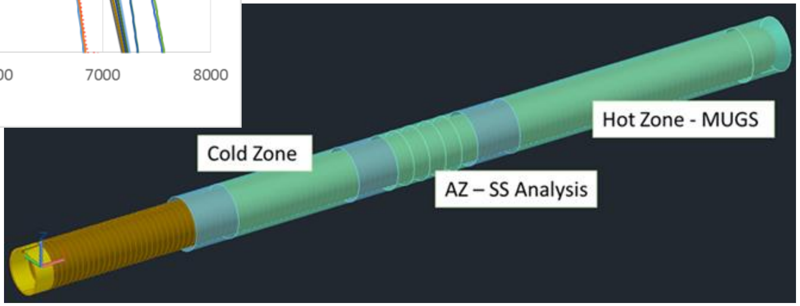


# Thermal Transient Boundary Conditions

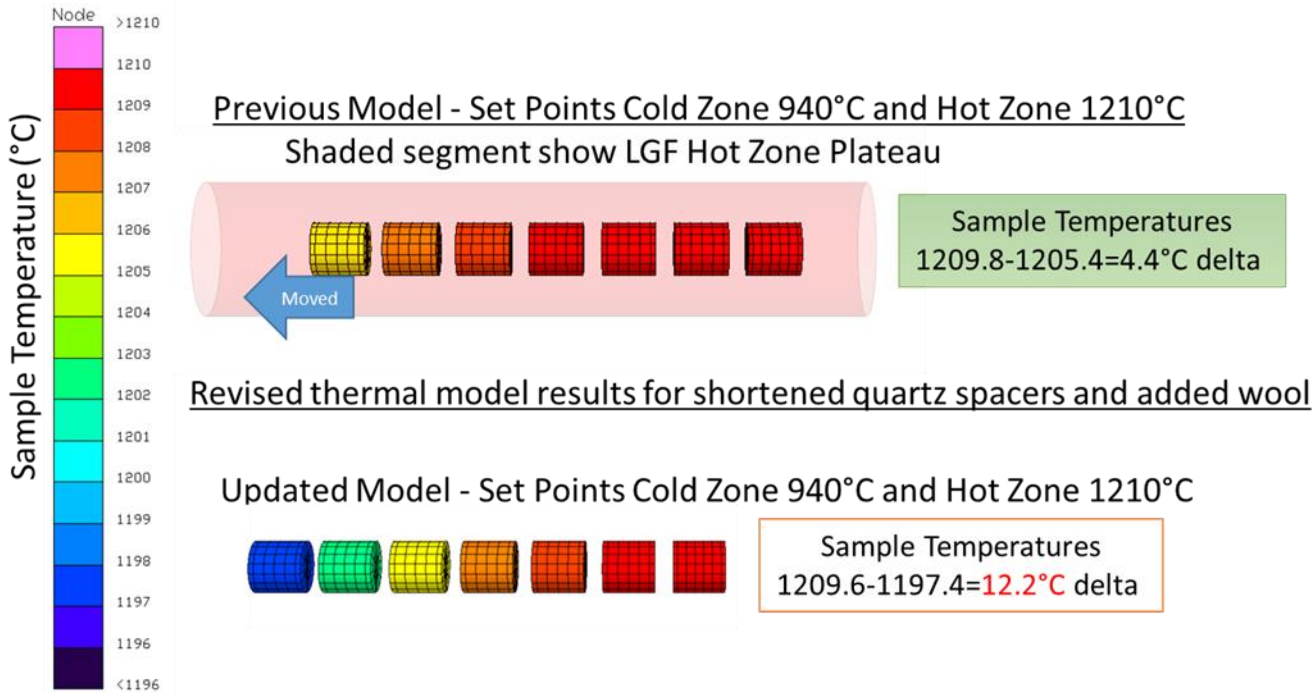
- Heated zone profile based on MUGS development test data to simulate LGF heating transient
  - Coupled with steady state GEDS model results for heating plateaus



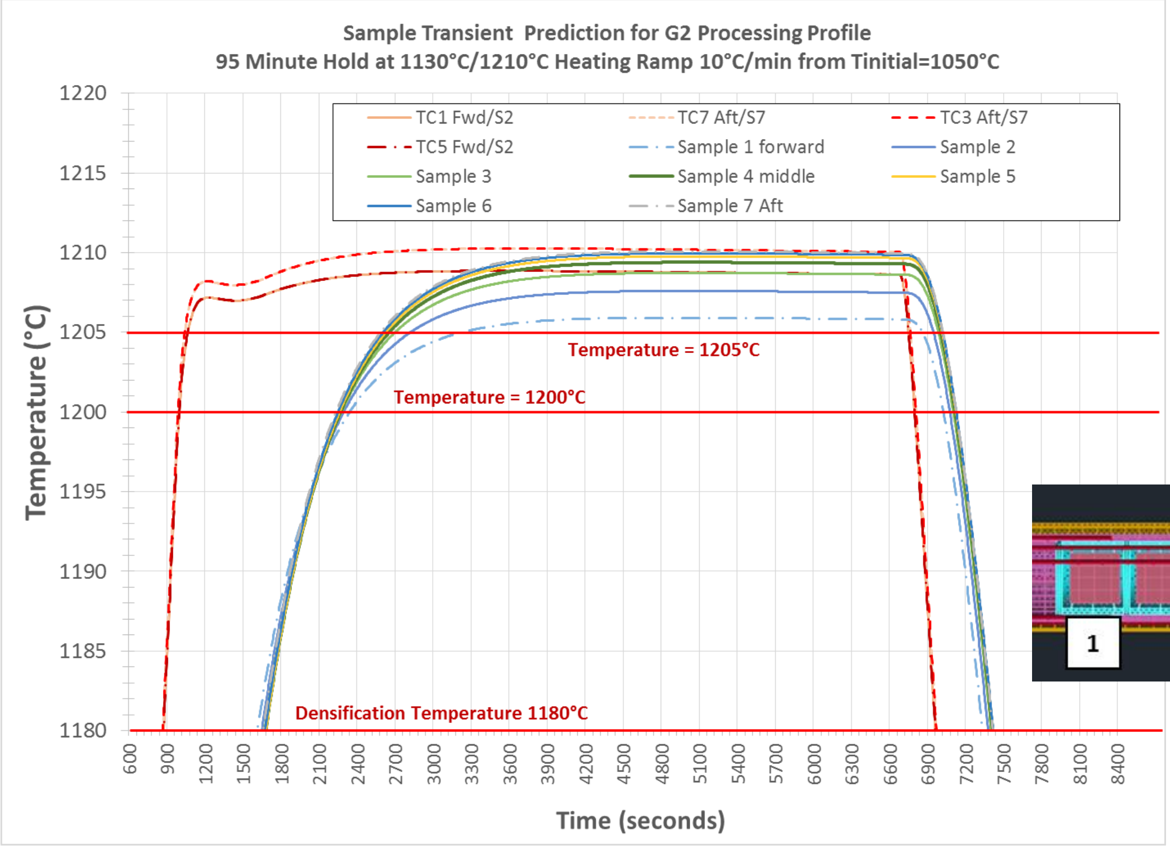
# Predicted Response For MUGS

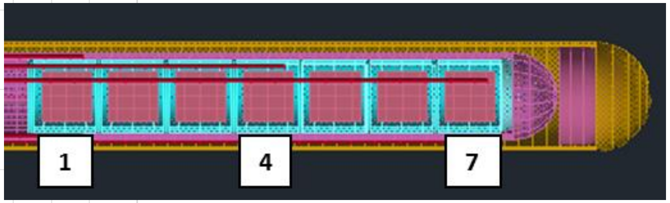
- Thermal model over predicted sample dwell time in the sintering range
  – PI MUGS sample evaluation showed long duration samples were not fully sintered
- MUGS thermal model based on initial GEDS design
  – Design changes shifted samples about 0.5 inches toward the adiabatic zone with MUGS test design changes integrated
  – Unable to increase hot zone setting above 1210° C due to design limitations



# Updated Response For GEDS

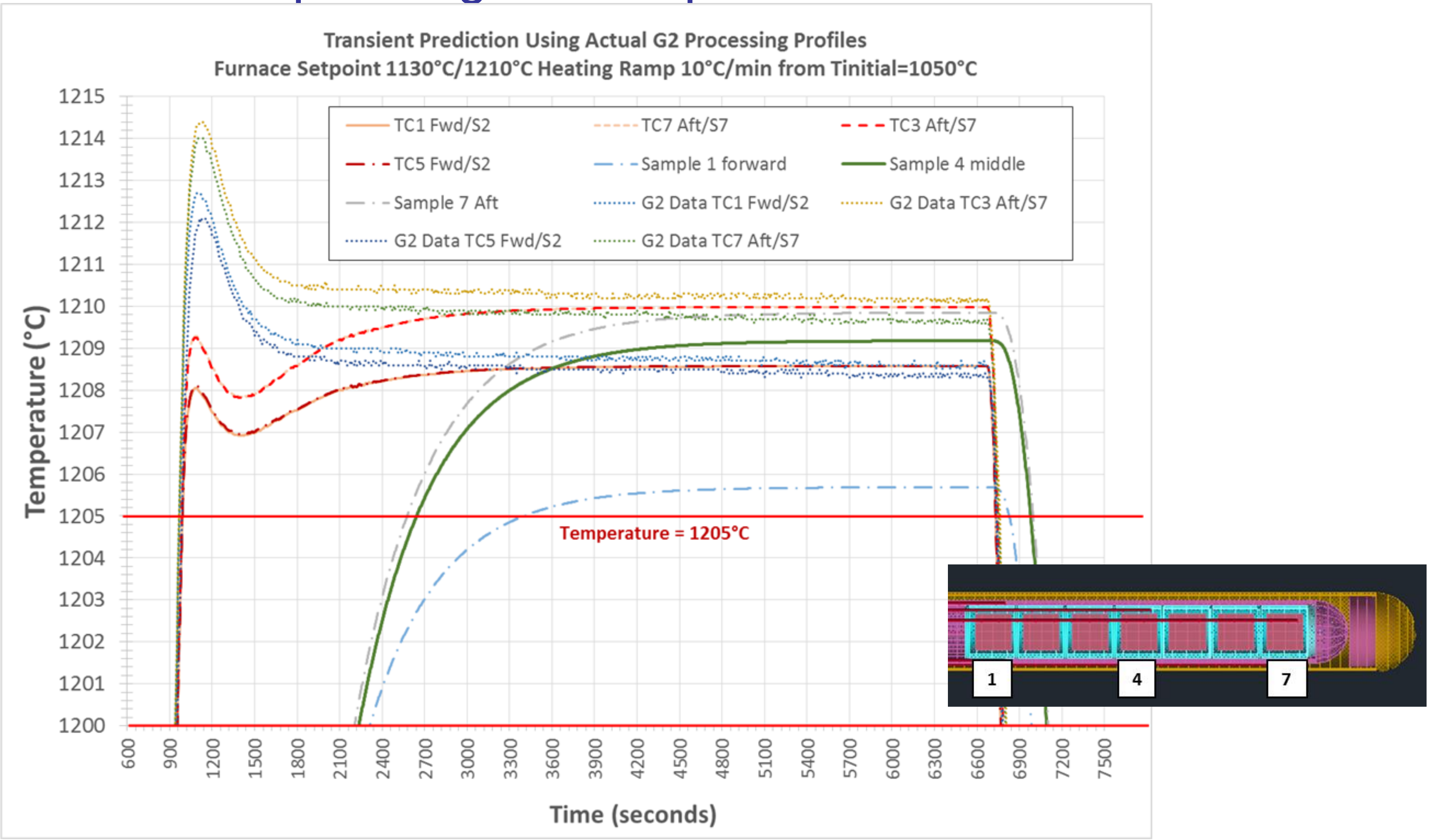
- Used updated transient model to revised processing profile prior to ground preflight test, G2
  - Model updated to Increase cold zone temperature to 1130˚ C to prevent end effects
  - Sample 1 dwell time = 60 minute calculated base on time > 1205˚ C for



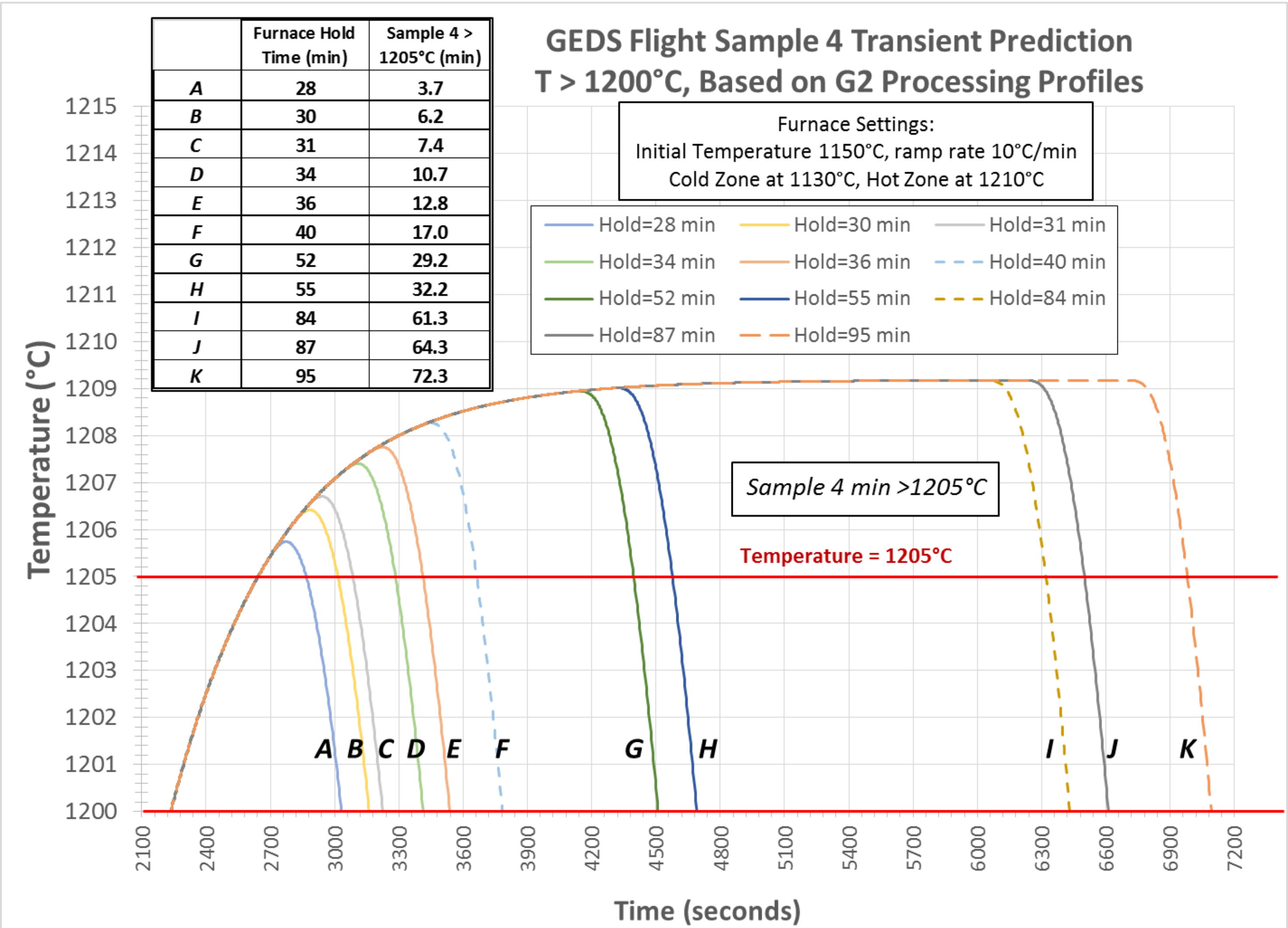
# GEDS Processing Predictions for G2

- 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




| | Furnace Hold Time (min) | Sample 4 > 1205°C (min) |
|---|---|---|
| *A* | 28 | 3.7 |
| *B* | 30 | 6.2 |
| *C* | 31 | 7.4 |
| *D* | 34 | 10.7 |
| *E* | 36 | 12.8 |
| *F* | 40 | 17.0 |
| *G* | 52 | 29.2 |
| *H* | 55 | 32.2 |
| *I* | 84 | 61.3 |
| *J* | 87 | 64.3 |
| *K* | 95 | 72.3 |

# 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


1) http://msrr.msfc.nasa.gov/. [Online]
2) *Simulation of ESA's MSL Furnace Inserts and Sample-Cartridge Assemblies: Model Development and Correlation with Experimental Data*. Johannes Dagner, Marc Hainke, and Jochen Friedrich. Rome, Italy: 35th International Conference on Environmental Systems, 2005.
3) *Development of NASA's Sample Cartridge Assembly: Design, Thermal Analysis, and Testing*. B. O'Connor, et al. Bellevue Washington: International Conference on Environmental Systems, 2015.
4) *Characterization of Vacuum Plasma Spray Formed Molybdenum-Rhenium Alloys*. J. Scott O'Dell, et al. Orlando, Florida: International Conference on Tungsten, Refractory & Hard Metals VI, 2006.
5) *Multiscale Modeling and Experimentation on Liquid Phase Sintering in Gravity and Microgravity Environments, MSRR-1 SCA Science Requirements Document (SRD)*, MSRR1-DOC-0115, Dr. Randall M. German, MSFC, Alabama, January 24, 2014.
6) T. Panczak, S. Ring, M. Welch, D. Johnson, B. Cullimore, D. Bell. *C & R Technologies (R) Thermal Desktop (R) User's Manual, A CAD Based System for Thermal Analysis and Design, Version 5.8.*