

Development of NASA's Sample Cartridge Assembly: Design, Thermal Analysis, and Testing

Brian O'Connor¹, Deborah Hernandez², James Duffy³
NASA Marshall Space Flight Center, Huntsville AL 35812

NASA's Sample Cartridge Assembly (SCA) project is responsible for designing and validating a payload that contains a materials research sample in a sealed environment. The SCA will be heated in the European Space Agency's (ESA) Low Gradient Furnace (LGF) that is housed inside the Material Science Research Rack (MSRR) located in the International Space Station (ISS). Sintered metals and crystal growth experiments in microgravity are examples of some of the types of materials research that may be performed with a SCA. The project's approach has been to use thermal models to guide the SCA through several design iterations. Various layouts of the SCA components were explored to meet the science and engineering requirements, and testing has been done to help prove the design. This paper will give an overview of the SCA design. It will show how thermal analysis is used to support the project. Also some testing that has been completed will also be discussed, including changes that were made to the thermal profile used during brazing.

Nomenclature

<i>°C</i>	=	Degrees Celsius
<i>CTE</i>	=	Coefficient of Thermal Expansion
<i>ESA</i>	=	European Space Agency
<i>hr</i>	=	Hours (time)
<i>ISP</i>	=	Intermediate Support Plate
<i>ISS</i>	=	International Space Station
<i>LGF</i>	=	Low Gradient Furnace
<i>m</i>	=	Meter
<i>MSFC</i>	=	Marshall Space Flight Center
<i>MSL</i>	=	Materials Science Laboratory
<i>MSRR</i>	=	Materials Science Research Rack
<i>MoRe</i>	=	Molybdenum-Rhenium
<i>NASA</i>	=	National Aeronautics and Space Administration
<i>PI</i>	=	Principle Investigator
<i>psia</i>	=	Pounds Force per Square Inch Absolute
<i>Ref</i>	=	Reference
<i>RTD</i>	=	Resistance Temperature Detector
<i>SCA</i>	=	Sample Cartridge Assembly
<i>sccs</i>	=	Standard Cubic Centimeters per Second
<i>SQF</i>	=	Solidification Quench Furnace
<i>SRM</i>	=	Science Reference Model
<i>SSITF</i>	=	Space Systems Integration & Test Facility
<i>TC</i>	=	Thermocouple
<i>VPS</i>	=	Vapor Plasma Sprayed
<i>W</i>	=	Watt

¹ Thermal Engineer, MSFC ES22 Space Systems Stress and Thermal Analysis Team, Huntsville, AL 35812.
brian.f.oconnor@nasa.gov

² Thermal Engineer, MSFC ES22 Space Systems Stress and Thermal Analysis Team, Huntsville, AL 35812

³ Design Engineer, MSFC ES21 Space Systems Design Team, Huntsville, AL 35812

I. Introduction

NASA's Sample Cartridge Assembly (SCA) project is responsible for designing and validating a payload that contains a materials research sample that will be processed in a furnace on the International Space Station (ISS). The SCA will be processed in the Materials Science Research Rack (MSRR) using the Low Gradient Furnace (LGF). MSRR, shown in Figure 1, was launched in 2009 and installed in ISS's Destiny Lab (Ref 1). The right side of MSRR consists of the European Space Agency (ESA) Material Science Lab (MSL). MSL contains a vacuum chamber that accepts different furnace inserts. ESA currently has two furnace inserts available for use by Principle Investigators (PIs), the LGF and Solidification Quench Furnace (SQF). Some of the differences between the two inserts include the SQF can establish higher gradients, has a quench capability, and a smaller bore diameter.

ESA offers a version of a SCA that can be processed using the LGF or SQF insert, refer Figure 2. ESA has processed a number of SCAs in both the LGF and SQF (Ref 2). The SCA in development at NASA Marshall Space Flight Center (MSFC) will differ from ESA's in a few ways. For example, NASA's SCA will have a larger diameter, which can hold a larger sample size. Also, NASA's SCA will have a high emissivity coating, which should allow for higher temperature gradients when processed in the LGF.

This paper will provide an overview of NASA's SCA, and the work that has been done to date. First a background of the thermal environment that the SCA will be processed in will be given. Then an overview of the design will be given. This will be followed by an overview of the thermal analysis method. Next some testing that has been accomplished will also be discussed. This includes a test that was done to measure the conductivity of the tube, and a test that was done on the first integrated SCA. Finally a change that was made to the braze profile will be shown and discussed.



Figure 1: The Material Science Research Rack (MSRR) Consists of the Material Science Lab (MSL) (right side of rack) which contains a vacuum chamber that accepts different furnace inserts such as the Low Gradient Furnace (LGF)



Figure 2: ESA's SCA (Ref 6)

II. Thermal Processing Environment

The LGF is a Bridgman furnace that consists of a hot zone and cold zone (Ref 3). The two zones are separated by what is called the adiabatic zone. For a directional solidification type of experiment (e.g. crystal growth) the temperatures of hot and cold zones are set such that the material has a solidification line in the adiabatic zone. By translating the furnace, or the sample, it is possible to directionally solidify the sample. See Figure 3 for an example of this process. It is also possible to do an isothermal type of experiment by placing all of the sample in the hot zone. It is not uncommon to have samples that need to be processed at temperatures exceeding 1100°C (2000°F).

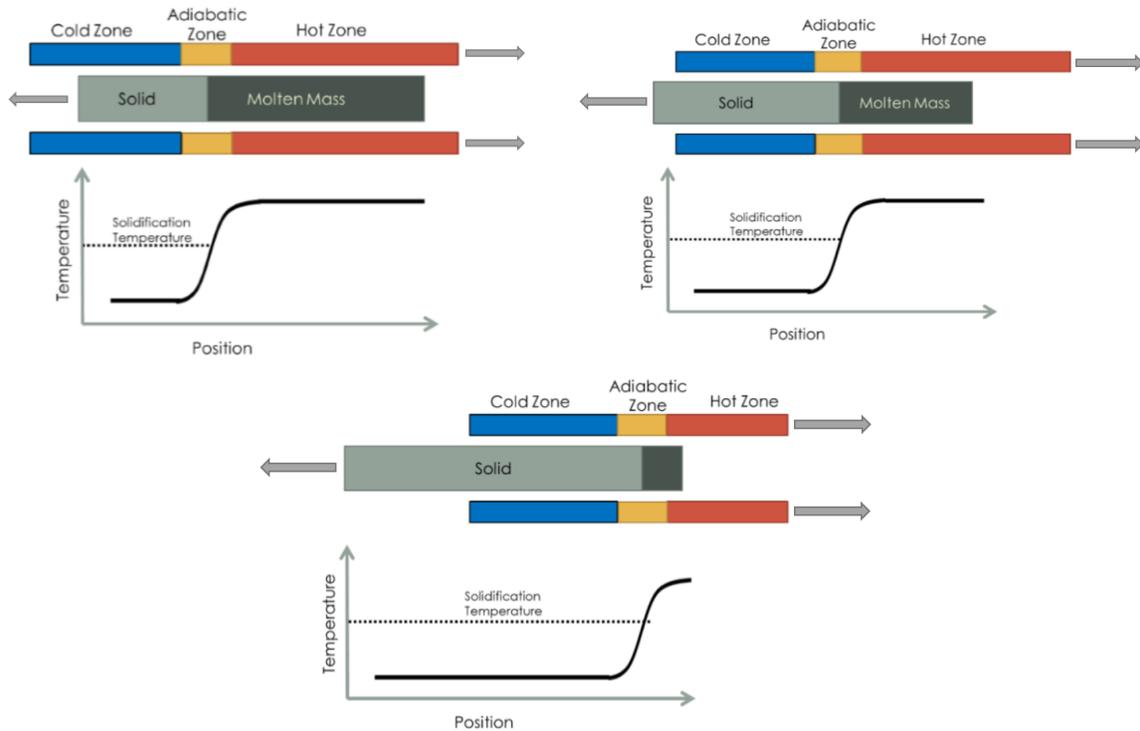


Figure 3: Directional Solidification using a Bridgman Furnace. Process Goes from Top-Left, then Top-Right, then Bottom. Either the Sample or Furnace Can Be Translated (For MSL the Furnace is Translated).

Inside the MSL the LGF is attached to a linear translating mechanism that allows the furnace to move with respect to the SCA. The primary function of a SCA is to provide a sealed container that holds the sample. Another function of the SCA is to provide instrumentation needed for science and engineering purposes, for example thermocouples in order to measure the temperature of the sample. Because of this, it is necessary to have a portion of the SCA cold enough to allow for the instrumentation to work. Therefore, during installation inside the MSL one section of the SCA is bolted onto a water chilled plate, see Figure 4.

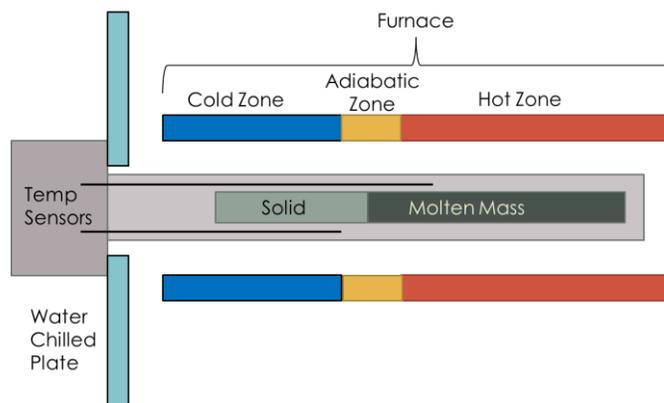


Figure 4: The Thermal Processing Environment of the SCA Consists of the Furnace and the Water Chilled Plate.

III. Design Overview

The main drivers for the SCA design include the furnace size, maximum processing environment, materials compatibility, instrumentation, and the project schedule. The furnace size is dictated by the use of the LGF furnace, which allows a maximum outer cartridge diameter of 26mm. The maximum processing environment was determined by a desire to minimize the reduction of life for the furnace. Even though the furnace can operate up to 1400°C, the life of the furnace thermocouples at this temperature is very short. Therefore the project has decided to target a maximum processing temperature just above 1200°C. The primary SCA instrumentation includes temperature sensors and a pressure sensor.

The baseline design for the for the current SCA project, which began around 2010, was inherited from a project that was canceled around 2005. The 2005 project had just completed its critical design review before it was canceled. However an entire SCA was never actually built before the project was canceled. The current project was started in 2010. It hoped to save schedule and cost by continuing where the previous project left off. Unfortunately during buildup of a SCA in 2014 numerous problems occurred that caused some portions of the SCA to be redesigned. During this redesign, thermal analysis was heavily relied on to help make design decisions. Portions of this paper will discuss some of the differences between the 2005 design and the redesign.

A picture of the original SCA is shown Figure 5, and the redesign is shown in Figure 6. In general, a SCA can be effectively broken down into two parts. First is the head, which is the left side of the picture and will be bolted to the water chilled plate in the MSL. Second is the tube, which is where the material samples will be held and is inserted into the furnace. The majority of the head is made from 416 stainless steel. This material was chosen for its machinability and has a coefficient of thermal expansion (CTE) that is similar to the tube. The flange and tube having similar CTE is important when brazing the tube to the head, which occurs at 1800°F (982°C).

One of the major requirements of the SCA is to have a helium leak-rate that is less than 1×10^{-8} sccs. Because of this low leak rate it is desirable to minimize the number of potential leak paths. This is accomplished in the SCA by minimizing the number of mechanical joints. The original SCA design consisted of four metallic joints. This included two welds, one bolted Conflat, and one braze. As can be seen in Figure 5, the bolts of the Conflat joint were outside of the inner volume of the head. One of the goals of the redesign activity was to increase the inner volume, so the bolts were moved to be in line with the inner wall, see Figure 6. Another goal was to allow for reusability, so the area where the tube is attached to the head became a separate mechanical piece that is attached to the head using a Conflat joint. Therefore the redesigned SCA consists of 5 metallic joints. This includes two welds, two bolted Conflat, and one braze.

The SCA tube is a molybdenum-rhenium (MoRe) vapor-plasma-sprayed (VPS) composite tube (Ref 4). It consists of an inner liner of alumina that transitions to the MoRe. The inner liner of alumina prevents potential reactions of the MoRe with PI sample materials. A coating of zirconium diboride is applied to the outside to increase its emissivity. This will create better thermal coupling between the tube and the furnace heaters, which will result in increase thermal gradient. In order to prevent potential reactions with PI samples most of the components inside the tube are currently made of alumina or quartz.

In the original design, Figure 5, the thermocouple reference junction was at the back of the data feed-through connector. As part of modular head redesign, the reference junction was moved to a collar mounted to a standoff inside the cartridge head, see Figure 7. The collar holds 12 thermocouples transistions and the resistance temperature detector (RTD). The RTD is used to measure the thermocouple cold reference junction temperature, which is used for the thermocouple cold junction compensation.

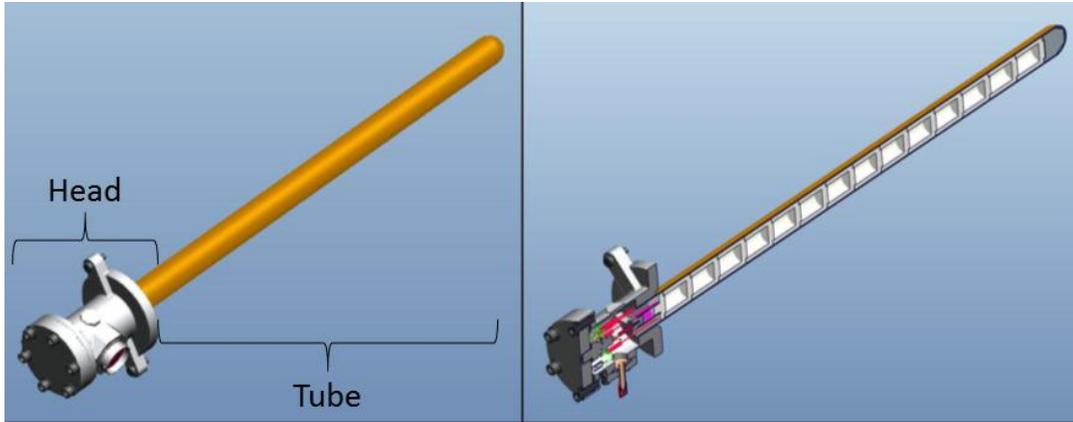


Figure 5: Creo Model of the Original SCA

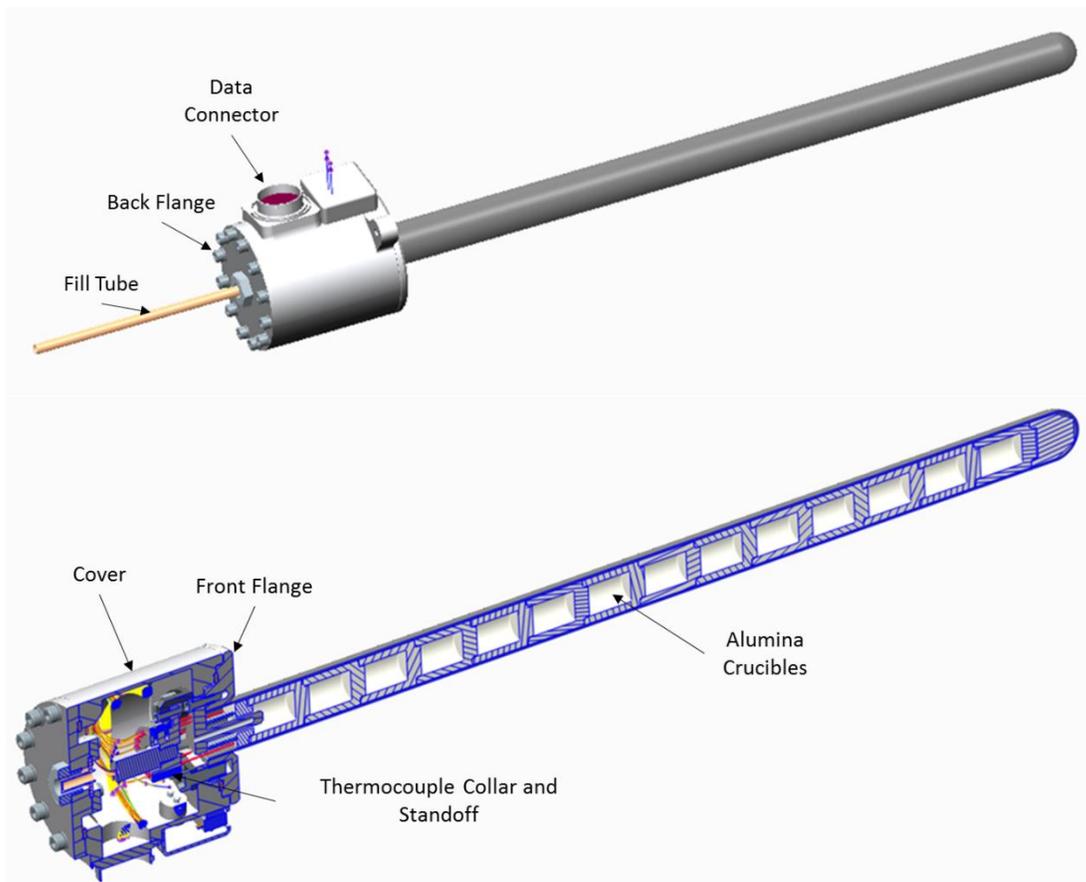


Figure 6: Creo Model of the Redesigned SCA

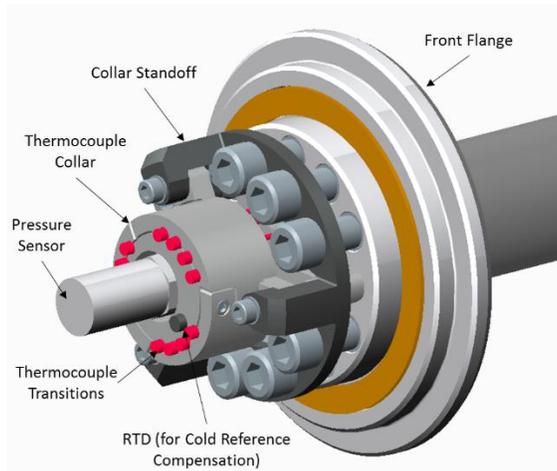


Figure 7: Close Up of Front Flange and Thermocouple Collar (Creo Model)

IV. Thermal Modeling and Analysis

Analysis Overview

Thermal analysis was performed in Thermal Desktop® (Ref 5). The thermal model of the furnace was developed from an early 2000's ESATAN model that was converted to SINDA. The furnace surfaces that are viewable by the SCA external surfaces are represented as Thermal Desktop® surface entities. All of the other components of the furnace were input using a SINDA include file. The SCA model was added to the furnace model using finite element meshes that were created in FEMAP® (Ref 6), and Thermal Desktop® native solids and surfaces. See Figure 8. The results of the model are used to help guide design and give indications of conformance to requirements. For example, that processing temperature profile requirements are attained. However the model will not be used to close verifications, this will be done through a qualification and acceptance test program. Discussed below are some of the model features, and an example of the results given by the model.

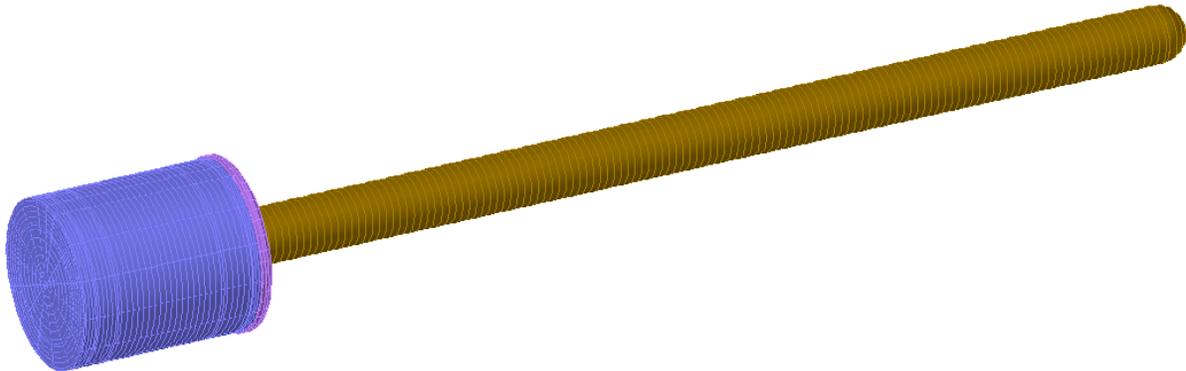


Figure 8: Thermal Model of SCA

The tube is modeled using native Thermal Desktop® entities. The largest uncertainty with this component is the thermal conductivity. This is because it is a vapor plasma sprayed tube that consists of layers of alumina, molybdenum, and zirconium diboride. The tube manufacturer measured the thermal conductivity of a Mo-Re tube using the laser flash method on a small sample piece of Mo-Re (Ref 4). It is uncertain if this method can accurately measure the axial conductivity of the tube, which is the primary heat transfer direction for the SCA. Therefore a test was done using a spare tube to calculate the axial thermal conductivity of the tube. This will be discussed further in the testing section.

The majority of the head is modeled with native Thermal Desktop® entities. However the front flange and collar standoff were modeled using finite elements. See Figure 9. This was done to appropriately capture the heat transfer in this area, which is where the majority of the heat passes through the head. That is, the heat comes from the tube and then is conducted to the MSL cold plate.

The collar is where the thermocouple cold junction is located (inside the thermocouple transitions). Therefore the RTD that provides the temperature measurement for the cold junction compensation must be located here. The standoff integrates the collar to the head body. In order to keep the RTD below 90°C the standoff is made of aluminum to promote heat transfer from the collar to the front flange, which is cooled by the MSL cold plate. Using two different materials, stainless steel (head body) and aluminum, is not ideal for stress because of the different coefficient of thermal expansion. The 90°C temperature is required by the MSL data acquisition system for reading the RTD. In order to capture the heat transfer in this area, the standoff and collar were modeled using finite element meshes. See Figure 9.

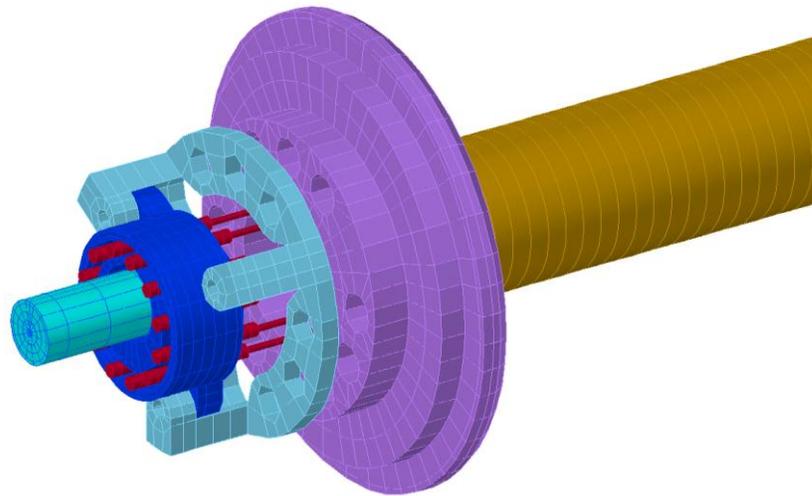


Figure 9: Detailed View of the Front Flange and Standoff with Thermocouple Collar

The alumina crucibles were modeled using finite element meshes, this was done in order to capture the cutouts that the TC's pass through, see Figure 9.

Example Results

The type of results needed from the model can be mostly seen from two separate viewpoints: from engineering concerns, and from Principal Investigator concerns. One example of an engineering concern is to make sure component temperature limits are not being exceeded. Some examples that the Principal Investigators are concerned with include the furnace-to-sample temperature difference, achievable gradients in their sample, and isothermal abilities.

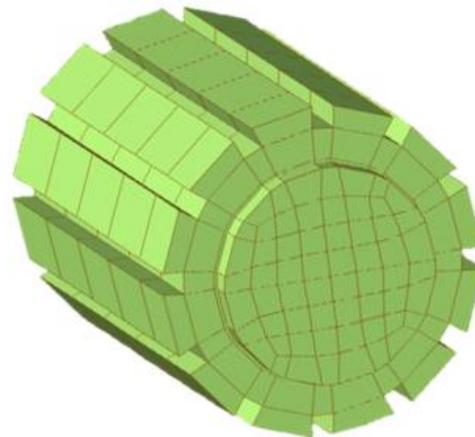


Figure 10: Detailed View of Alumina Spacer

The results shown in Figure 11 provide an example of the engineering result. It can be seen that the heat from the cartridge transfers into the front flange, and is conducted to the MSL cold plate. Also the collar, and thus the reference junction, is below its 90°C limit.

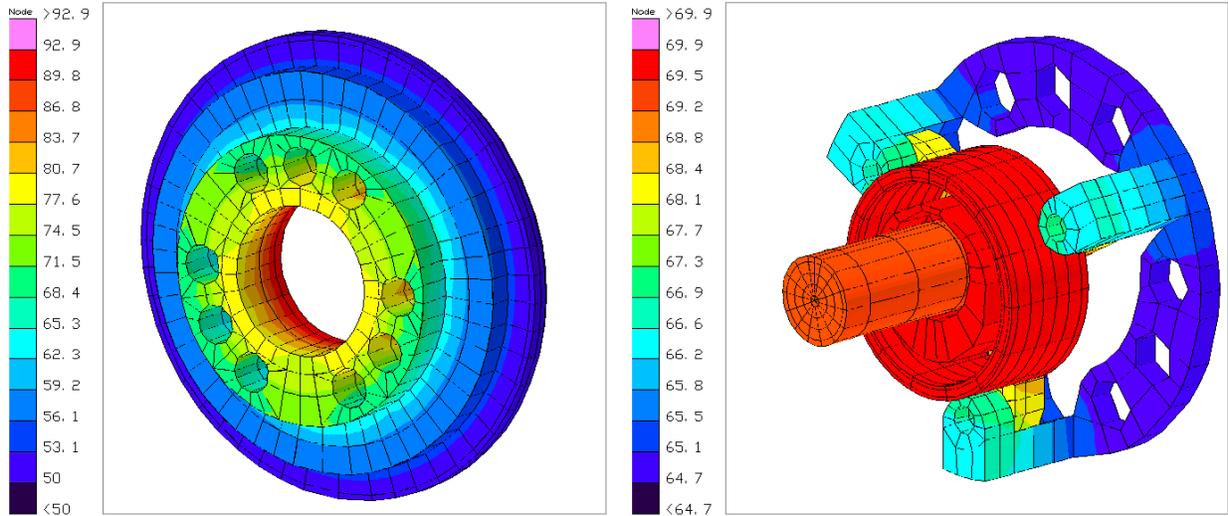


Figure 11: Example Results of Front Flange (Left Picture) and Thermocouple Collar and Standoff (Right Picture). Units are in °C.

The result shown in Figure 12 shows cartridge temperature versus position from the SCA head. Some examples of information that a PI might infer from this type of graph include the profile within each heater zone, the isothermal characteristics within the zone, and the gradient that occurs in the adiabatic zone. However a PI will want to see the temperature of their sample instead of the cartridge.

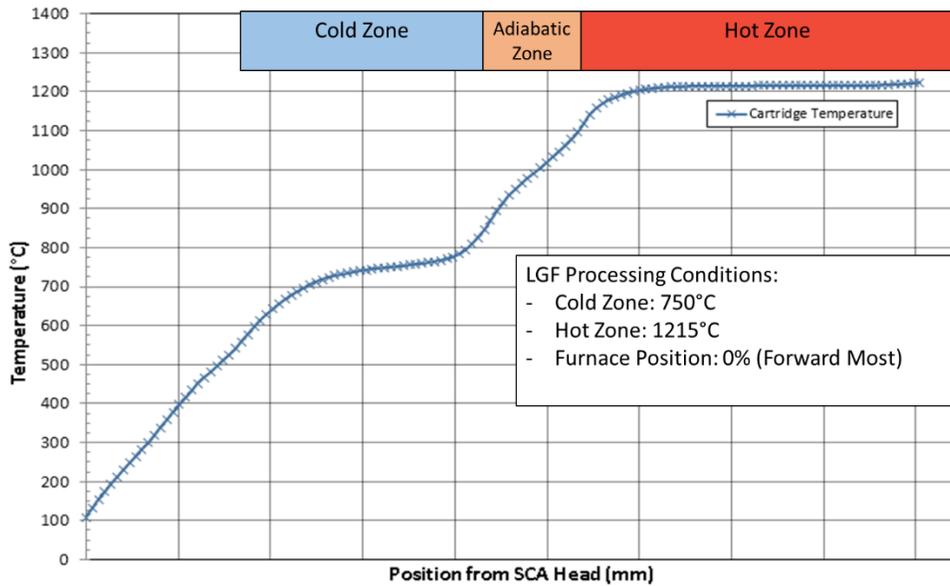


Figure 12: Example Results of Cartridge Temperature versus Position

V. Testing

The test program thus far has been broken down into three types: development testing, qualification testing, and acceptance testing. At the time of writing this paper the program is about to enter into qualification testing, so this section will discuss some of the development testing that has occurred. This includes heating a SCA in a commercial furnace, and the thermal conductivity test.

Heating in a Commercial Furnace

Before the SCA can be tested in a ground unit of the flight furnace, a SCA was heated in a commercial furnace in order to build confidence that it is acceptable. One of the primary factors for a SCA to be acceptable is to have a

helium leak rate that is below 1×10^{-4} sccs while it is being heated. Even though the commercial furnace does not have the same heating profile as the LGF, it was able to heat a portion of the tube up to the maximum expected temperature (1280°C). An off-nominal case (e.g. loss of cooling) was simulated by decoupling the head of the SCA from the water chilled plate, this allowed the head to heat up to 130°C. During the test in the commercial furnace, the SCA was heated and cooled 11 times. During the final heating cycle, the SCA was pressurized to 125 psia, which is a pressure that is much higher than the expected nominal pressure (3 psia). The SCA successfully completed all the testing and maintained a helium leak rate below 1×10^{-8} sccs, which is much lower than the required leak rate during processing (1×10^{-4} sccs).

The data obtained from the test was compared against a thermal model of the commercial furnace with the SCA inserted. Figure 13 below shows a comparison between the recorded TC's from the test and the predicted cartridge temperatures from the model. The model has yet to go through a correlation activity, but some reasons for the temperatures profile differences are suspected to be from the furnace model and assumptions on the SCA model (e.g. thermal conductivity and contact coefficients between crucibles). The outside of the furnace is exposed to natural convection, which has a high degree of uncertainty. This will affect the thermal environment that the SCA is exposed to, i.e. the boundary conditions. Therefore it is suspected that the assumption of the natural convection value is one of the prime reasons for the temperature profile differences.

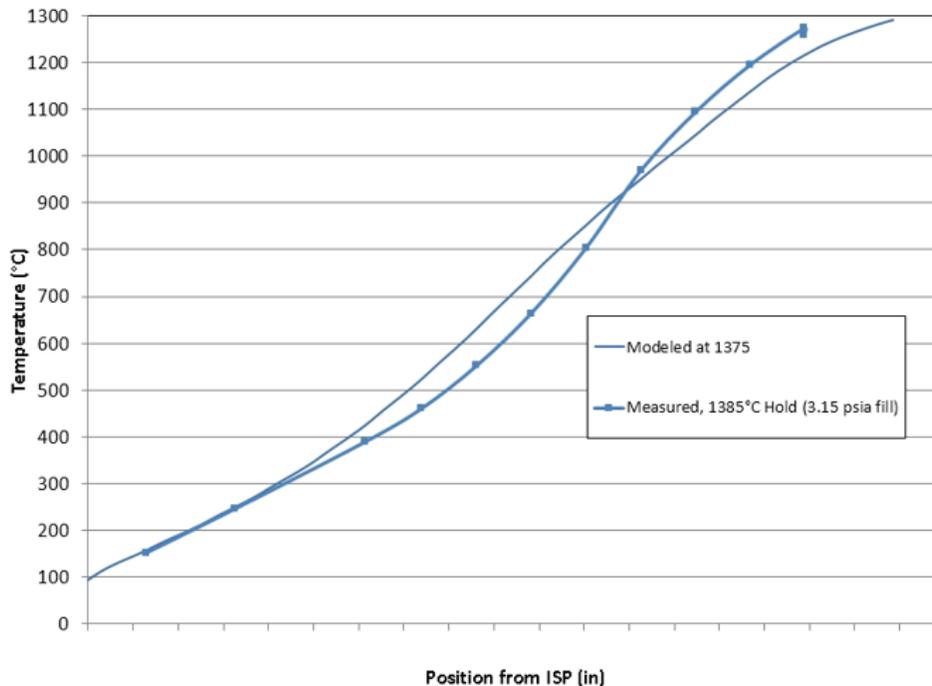


Figure 13: Comparison between Measured and Modeled Temperatures for Heating in a Commercial Furnace

Thermal Conductivity Test

As mentioned in the thermal analysis section one of the uncertainties in the SCA model is the thermal conductivity of the cartridge tube. The tube manufacture has measured the thermal conductivity of a Mo-Re tube using the laser flash method. It is suspected that this method will provide an average conductivity of the tube, but because the tube is an anisotropic composite material the axial conductivity may be different from the bulk or radial. For this reason a spare tube was tested in a fixture in order to measure the axial thermal conductivity.

A representation of the test fixture is shown in Figure 14. It consists of a boron nitride heater inserted into one end of the cartridge. The other end is bonded to a copper pedestal that is coupled to a water chilled plate. By placing RTD's in the pedestal it is possible to measure how much heat is flowing through the pedestal. In order to prevent heat losses and non-linear effects many layers of moly shielding were wrapped around the tube. Four sets of thermocouples were attached to the tube via a combination of wire tying and bonding the tips to the tube.

The test consisted of heating the top of the tube all the way to 1280°C. Unfortunately, even with the amount of insulation that was used, it is suspected that there were heat losses and gains throughout the entire height of the tube. Because of this it was not possible to get a direct measurement of conductivity from the test. Therefore it was necessary to build a thermal model that was subsequently correlated to the test, from which the thermal conductivity of the tube could be derived.

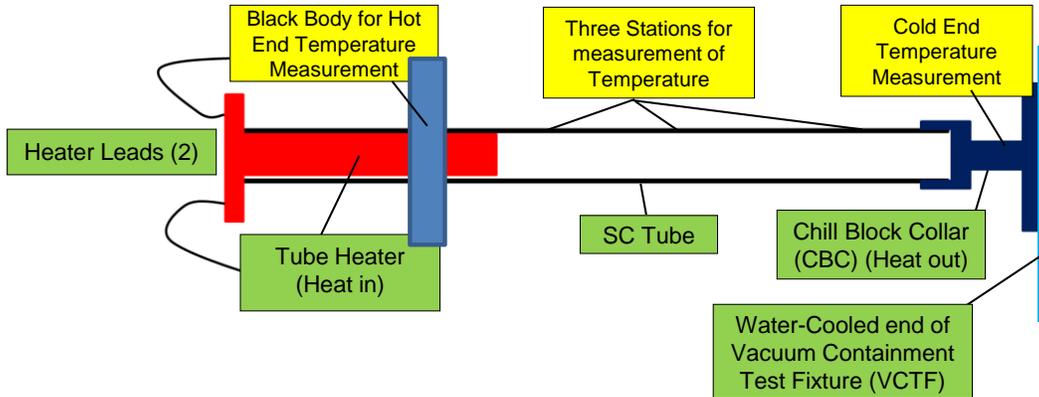


Figure 14: Comparison between Measured and Modeled Temperatures for Heating in a Commercial Furnace

VI. Brazing Profile

Originally the braze that was used to attach the tube to the head was BAg-8. Because the braze does not wet a bare tube and flange it required both to be copper plated. The braze has a melt temperature of 1435°F (779°C), where it is actually an eutectic. That is, it goes from solid to liquid without an intermediate mushy zone. The thermal profile that was used in the braze process is shown in the Figure 15. It consisted of a quick heat up to a hold temperature just 10°F (5.5°C) below the melt temperature. The parts, which consisted of the front flange and tube, were then held in order to allow them to become isothermal. Then the furnace was quickly heated above the melt temperature, and once the parts crossed 1480°F they were held for 10mins before allowing to cool. Even though this process resulted in one successful braze, there were a few that were unsuccessful. Because of this, the braze process was reassessed and updated.

BAg-13 braze was chosen for the updated design because it was found to wet both the tube and the flange without needing to be copper plating. The likely reason for this is because the braze has a higher melting temperature, due to its higher copper content, that allows for the tube and flange to reduce their oxides. The braze composition is such that it does not have an eutectic point, and has an intermediate mushy zone from 1420°F – 1640°F (771°C – 893°C). It was recommended by a consultant to update the thermal profile such that there are no temperature holds (Ref 7). Figure 16 shows the profile that was used during a BAg-13 braze. This process has been used a number of times to successfully attach the tube to the flange.

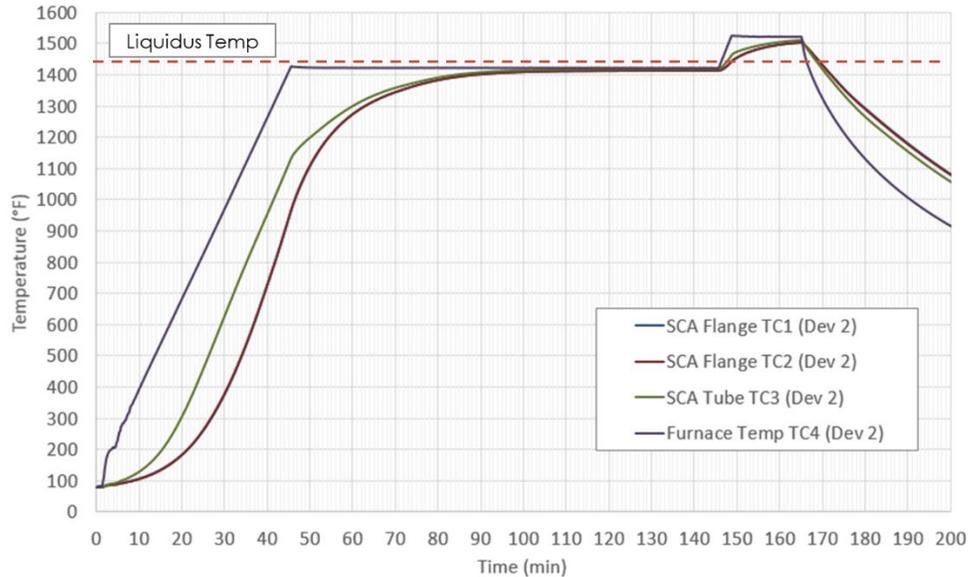


Figure 15: BAg-8 Braze Profile – With Holds

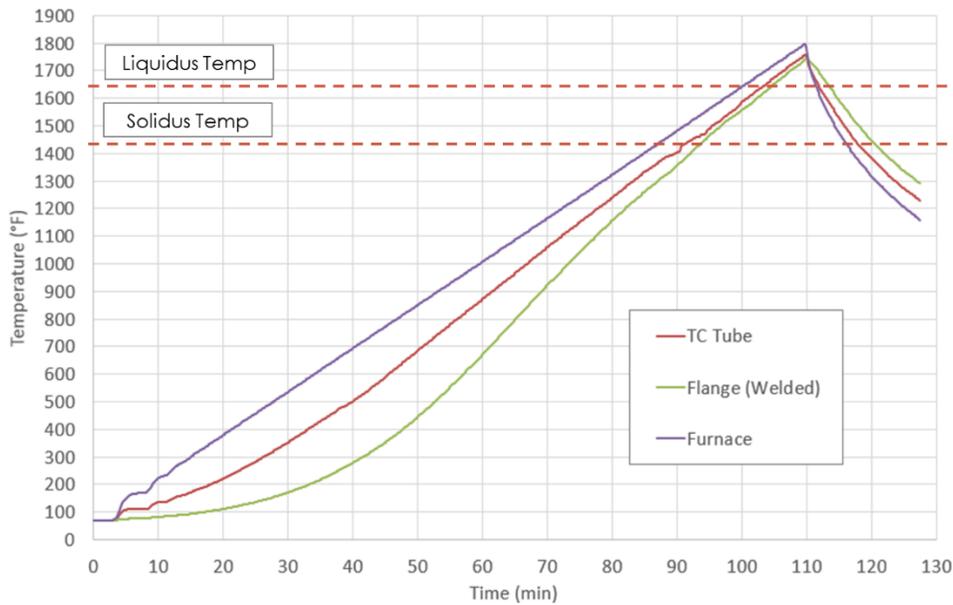


Figure 16: BAg-13 Braze Profile – No Holds

VII. Conclusion

The NASA SCA project will use SCA's for conducting materials research on the International Space Station. The project was originally started in early 2000's, but was canceled in 2005, just after it completed its critical design review. The current project, which started around 2010, picked up where the previous program left off. Unfortunately during buildup of the first integrated SCA, numerous issues were discovered that caused a redesign. During this redesign activity thermal modeling was used to help guide the design. The redesigned SCA was successfully built, see Figure 17, and tested in a commercial furnace. Thermal modeling will continue to be used in the project to provide results that are of interest to the Principle Investigators. One important input for modeling is knowing the thermal conductivity of the tube, to this end a test was done in order to measure the property. One interesting change that occurred during the redesign work was modifying the braze thermal profile from one that incorporated temperature holds to one that does not have any holds. The SCA project is about to enter qualification testing. After qualification testing the first Principal Investigator will perform their ground tests in order to finalize their furnace profile settings for flight.



Figure 17: Integrated SCA that was Tested in the Commercial Furnace

Acknowledgments

The authors would like to thank Shawn Breeding, Doug Westra, Jeff Farmer, Linda Hornsby and Joseph McConnell for their support and advice. And, of course, all the team members on the project.

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. <http://wsn.spaceflight.esa.int/docs/Factsheets/15%20MSL%20LR.pdf>. [Online]
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. 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 5.6*. April 2013.
6. FEMAP Version 11.1, Copyright 2013 Siemens Product Lifecycle Management Software Inc. [Online] www.femap.com.
7. <http://www.kaybrazing.com/brazing-articles/1000895-eliminating-holds-in-heating-rates-during-vacuum-brazing-cycles.html>. [Online]
8. <http://www.deskeng.com/de/studying-material-properties-in-space/>. [Online]