

On-Orbit Absolute Temperature Calibration Using Multiple Phase Change Materials – Overview of Recent Technology Advancements

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ABSTRACT

NASA's anticipated plan for a mission dedicated to Climate (CLARREO) will hinge upon the ability to fly SI traceable standards that provide irrefutable absolute measurement accuracy. As an example, instrumentation designed to measure spectrally resolved infrared radiances will require high-emissivity calibration blackbodies that have absolute temperature uncertainties of better than 0.045K (3 sigma). A novel scheme to provide absolute calibration of temperature sensors on-orbit, that uses the transient melt signatures from multiple phase change materials, has been demonstrated in the laboratory at the University of Wisconsin and is now undergoing technology advancement under NASA Instrument Incubator Program funding. Using small quantities of phase change material (less than half of a percent of the mass of the cavity), melt temperature accuracies of better than 10 mK have been demonstrated for mercury, water, and gallium (providing calibration from 233K to 303K). Refinements currently underway focus on ensuring that the melt materials in their sealed confinement housings perform as expected in the thermal and microgravity environment of a multi-year spaceflight mission. Thermal soak and cycling tests are underway to demonstrate that there is no dissolution from the housings into the melt materials that could alter melt temperature, and that there is no liquid metal embrittlement of the housings from the metal melt materials. In addition, NASA funding has been recently secured to conduct a demonstration of this scheme in the microgravity environment of the International Space Station.

Keywords: Absolute Temperature Calibration, Blackbody, Climate Mission

1. INTRODUCTION

Future NASA climate spaceflight missions such as the anticipated Climate Absolute Radiance and Refractivity Observatory (CLARREO) will hinge upon the ability to fly absolute standards that can provide the basis to meet stringent requirements on measurement accuracy. For example, instrumentation designed to measure spectrally resolved infrared radiances to detect a climate signature of 0.1 K per decade will require high-emissivity calibration blackbodies having absolute temperature uncertainties of better than 0.045 K (3 sigma). A key requirement for these future missions is to provide traceability to SI standards on-orbit.^{1,2,3,4,5}

This emerging need provided the motivation to develop a simple, low mass, absolute temperature calibration system that could be incorporated into an existing spaceflight blackbody design – the on-board blackbody calibration system developed for NASA's Geosynchronous Imaging Fourier Transform Spectrometer (GIFTS) instrument.^{6,7} The engineering development model of the GIFTS has successfully undergone thermal vacuum functional testing and calibration at the Utah State Space Dynamics Laboratory.^{8,9} The demonstrated state-of-the-art performance of the on-board blackbody system makes it nearly suitable for a CLARREO type climate mission. What this system lacks is the on-orbit traceability to SI standards, such as ties to the melt points of standard reference materials for absolute temperature calibration, and the capability of make absolute reflectance measurements of the emitting surface. Under internal research and development funding, the University of Wisconsin Space, Science and Engineering Center has integrated a novel phase change concept into the GIFTS blackbody design and demonstrated excellent accuracies.¹⁰ A

NASA Instrument Incubator Program (IIP) grant has been awarded to advance the technical readiness of this pioneering research to a Technical Readiness Level of (6), which involves demonstration of performance in a relevant environment.

This paper begins with a description of the key features of the phase change material implementation into the GIFTS on-board blackbody system. This will be followed by an overview of the focus of the NASA IIP technology development. We will then provide a status of our IIP work, including the housing sealing technique, the accelerated life test definition along with results for gallium, water, and mercury. We will end with an overview of plans for a demonstration in the microgravity environment of the International Space Station (ISS).

2.0 A NOVEL SCHEME FOR ABSOLUTE TEMPERATURE CALIBRATION

The novel scheme for absolute temperature calibration is based on a new concept that is expected to have wide applicability for the remote temperature calibration of devices. It uses transient temperature melt signatures from three (or more) different phase change materials to provide absolute calibration for the blackbody thermistor sensors covering a wide, continuous range of atmospheric temperatures. The system uses very small masses of phase change material (<1 g), making it well suited for spaceflight application. A prototype of this absolute calibration scheme using gallium, water, and mercury has been demonstrated at the University of Wisconsin under internal funding, using a duplicate of the GIFTS blackbody system.¹¹⁻¹⁷ Figure 1 illustrates the modifications to the GIFTS blackbody design that were implemented to demonstrate the new scheme. The solid model at right shows a cut-away of the GIFTS Blackbody, which uses an aluminum light-trapping cavity that has a circumferential heater to control temperature, and two thermistors near the cone apex and six thermistors around the circumference toward the back of the cavity for measuring temperature. The top photo at the left in Figure 1 is a view looking into the back of the blackbody cavity showing the six possible circumferential locations for the custom packaged thermistors (shown at bottom left). The packaging of the phase change cells, that includes the melt material enclosed in a housing, closely resembles that of the thermistors – allowing them to be threaded into the cavity in similar fashion. This scheme will accommodate three or more phase change materials interleaved with thermistor temperature sensors.

Figure 2 shows a typical transient temperature response of one of the blackbody cavity thermistors during a gallium melt event, where it can be seen that the melt plateau is clearly discernable to within 5 mK of the known melt temperature. First the blackbody cavity is brought to thermal stability in the constant temperature mode about 50 mK under the expected phase change temperature. Then the blackbody controller is switched into constant power mode using the power level that would bring the cavity to about 100 mK above the expected phase change temperature.

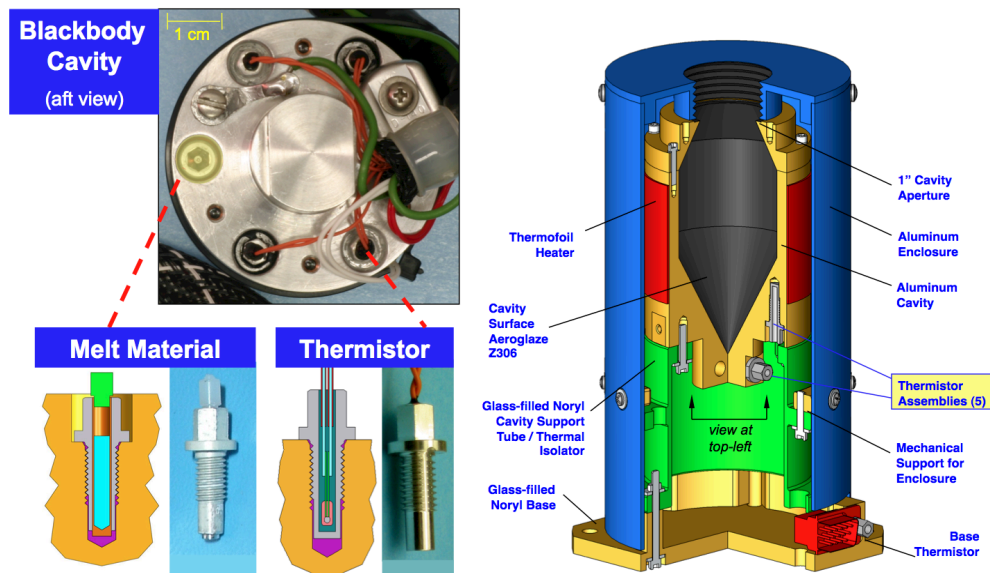


Figure 1. Configuration used to demonstrate the absolute temperature calibration concept, using a UW-SSEC mock-up of the GIFTS blackbody. Small quantities (<1g) of different phase change materials are integrated into custom housings that have the same geometry as the temperature sensors. Threaded holes in the blackbody cavity accept either thermistors or one of the phase change materials.

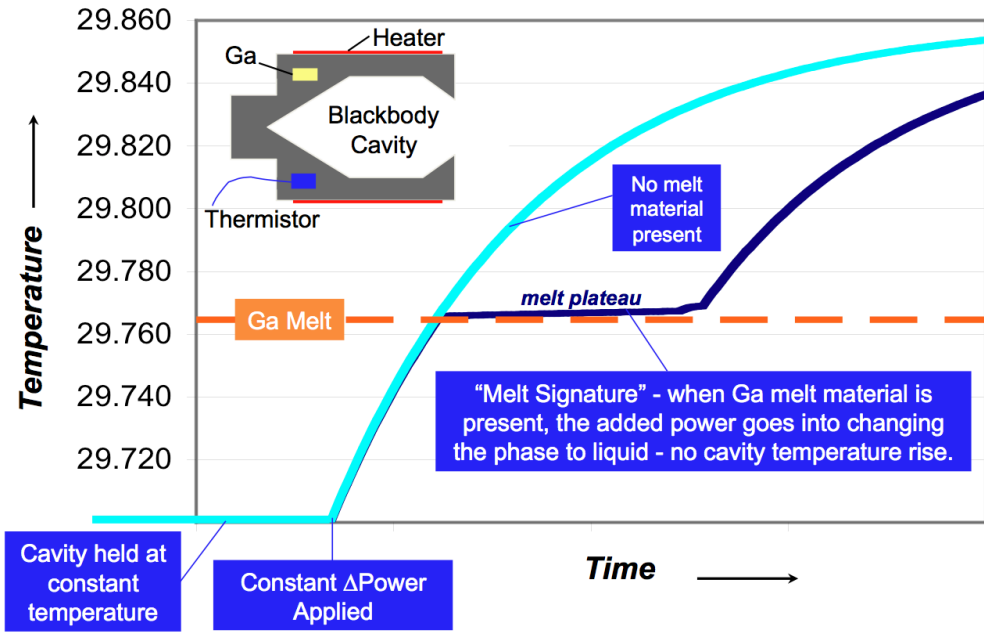


Figure 2. Sequence of events during a typical melt of gallium as configured in Figure 1. After initial stabilization in the constant temperature mode, a constant power is used to transition through the melt plateau.

Significant testing has been conducted to explore the melt plateau repeatability, and to characterize the relationship between the time taken to go through the melt and the melt plateau (mid-melt) temperature. Figure 3 illustrates that as the time to go through the melt is increased, the mid-melt temperature asymptotically approaches the known gallium melt temperature. The inset plot on the right of Figure 3 illustrates the excellent melt plateau repeatability for three different runs taken before and after exposure to accelerated life testing.

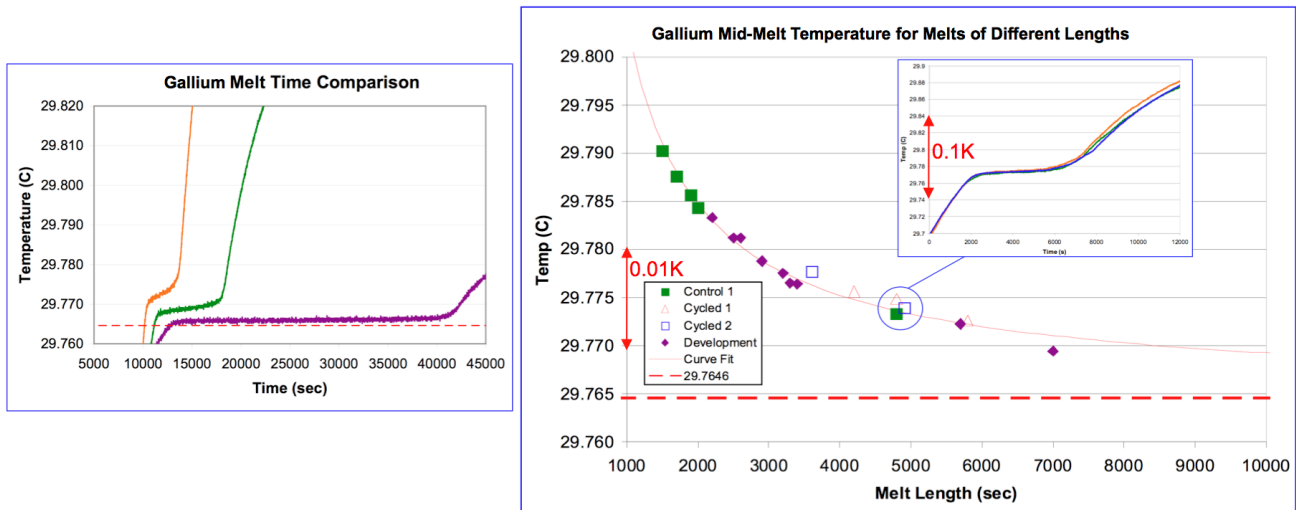


Figure 3. The plot on the left shows three different melts of different duration. The longer the melt the closer the plateau approaches the theoretical melt temperature. The large plot at right illustrates this by plotting mid-melt temperature vs. melt length – each data point corresponds to a single melt. This characteristic asymptotic behavior has shown to be invariant for a given physical configuration. The inset plot at upper right shows the three melt signatures associated with the circled data points. Data included in the plot is from three different samples taken before and after a full hot soak and abbreviated deep temperature cycling (40 cycles).

3.0 FOCUS OF NASA IIP TECHNOLOGY DEVELOPMENT

The focus of the technology development to be conducted under our NASA IIP grant is illustrated in Figure 4. After defining a suitable sealing technique an accelerated life test was defined to simulate the expected full mission temperature soak and cycling environmental exposure. Tests have been devised to show that the phase change materials have not been unacceptably contaminated via dissolution, and that the containment materials are not mechanically compromised via mechanisms such as liquid metal embrittlement. Mechanical integrity was also investigated by comparing the housing morphology both before and after the accelerated life testing.

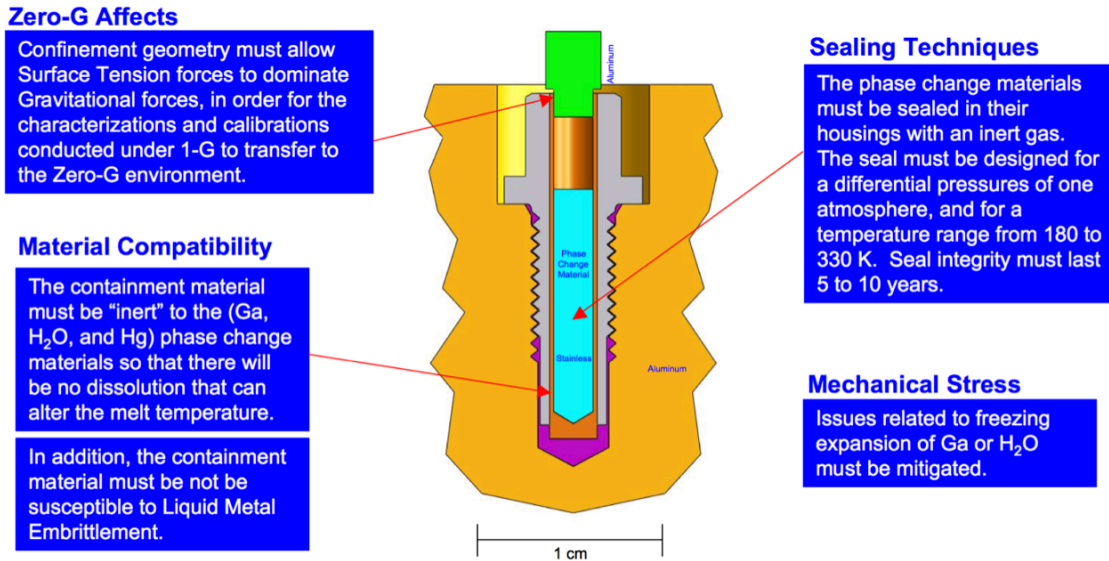


Figure 4. The major areas of research that will be addressed under the NASA IIP grant to advance this technology.

4.0 SEALING TECHNIQUE

Laser welding is technique chosen to seal the phase change materials into the stainless steel housings. We have worked with Preco, Inc. of Somerset, Wisconsin to optimize the welding process to obtain repeatable high quality welds that are void of any cracks. In the case of water we also were faced with the challenge to keep the average housing temperature below the boiling temperature so as not to lose crucial melt material during the welding process. Figure 5 illustrates the tapered plug being laser welded into a housing.

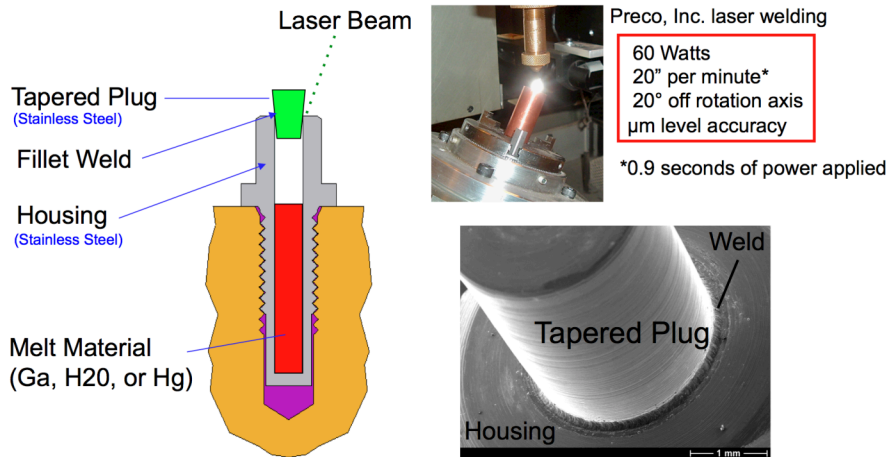


Figure 5. Laser welding of the tapered plug into the stainless steel housing that contains the phase change materials.

5.0 ACCELERATED LIFE TESTING DEFINITION AND RESULTS

The accelerated life test is designed to subject the sealed housings to the full number of deep temperature cycles expected prior to and throughout a mission lifetime. The test is also designed to subject the housings to an equivalent expected dissolution environment by exposing them to an elevated temperature for a far shorter than a mission lifetime. The accelerated life test is based upon the reasonable assumption that any reaction between the liquid metal and the housing is a thermally activated process with a rate given by an Arrhenius equation. The rate-limiting step can be controlled by either a volume dissolution or grain boundary attack or an interface reaction. To obtain a conservative estimate of the lower bound for the life, the fastest reaction can be considered which is grain boundary penetration. For FCC metals a typical activation energy value for grain boundary transport is about $E=83.6 \text{ kJ/mole}^{18}$. Figure 6 illustrates the equivalence between the accelerated life warm soak and the expected mission environment.

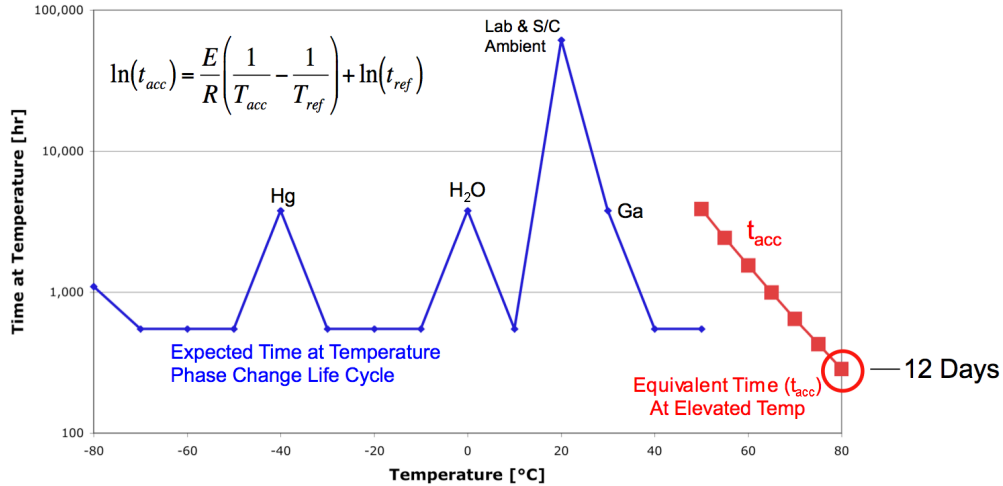


Figure 6. The accelerated life test uses the Arrhenius equation to define an equivalent time (t_{acc}) at an elevated temperature (T_{acc}) that provides an equivalent dissolution environment to the expected mission lifetime exposure of temperatures at T_{ref} for times t_{ref} . R is the universal gas constant. The series at the left in the plot is the expected full exposure. The series at the right in the plot shows the combinations of time at temperature that give an equivalent dissolution exposure. Twelve days at 80°C was picked for our testing to minimize test length while still keeping to a relatively benign temperature.

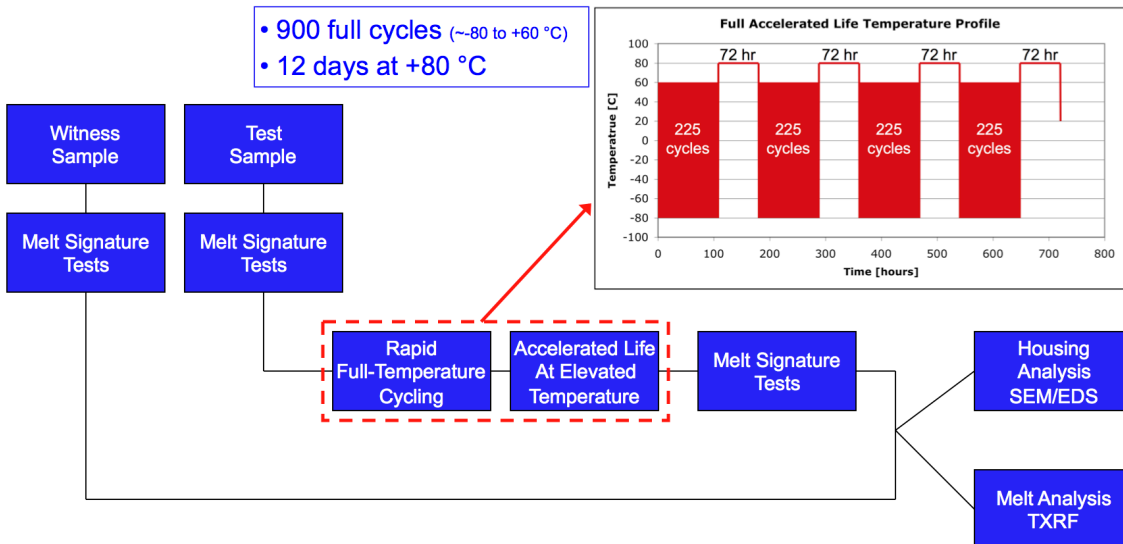


Figure 7. The accelerated life test flow used to verify that the gallium, water, and mercury melt cells.

Figure 7 illustrates the accelerated life test flow. Samples of each phase change material (gallium, water, and mercury) contained in welded housings went through the test. The test flow starts with obtaining baseline melt signatures for each sample and for a witness sample. Then the samples are subjected to 900 full temperature cycles from +60 to -80°C, interleaved between 12 days of a warm soak at +80°C. Post-test melt signatures were then obtained and compared with pre-test. Two other post-tests are included: housing and weld inspection using scanning electron microscopy, and melt material analysis for contamination using Total X-Ray Fluorescence (performed on un-welded samples only). This technique can detect melt contamination levels consistent with a <0.5mK melt temperature depression. To date we have measured both water and gallium samples using this technique and found no contamination. Because we have been able to demonstrate very high melt signature accuracies and repeatabilities, and because the X-Ray Fluorescence requires a destructive test of welded housings, we have relied on the signatures as a proxy for melt material contamination.

A status of accelerated life testing is shown in figure 8. The left of the table shows key properties of the phase change materials, including the melt temperature, whether or not the material expands or contracts upon freezing (expansion exposes the housings to mechanical work), and the possibility for liquid metal embrittlement (LME), where the liquid metal compromises the mechanical integrity of the housing by entering and significantly weakening the grain boundaries. This could happen if there was a breach in the protective oxide layer of the stainless steel housing.

	Material	Melt Point [°C]	Liq. >>Solid	LME Possible	Signatures (TEC)	Accelerated Life Test - Unsealed	Accelerated Life Test - Welded	Signatures (Blackbody)
Original IIP	Gallium	29.7	Expands	Yes	X	X	X	X
	Water	0.0	Expands	No	X	X	X	X
	Mercury	-38.9	Contracts	Yes	X	X	X	X
ISS	Gallium-Indium	16.5	Expands	Yes	X			
	Gallium-Tin	20.5	Expands	Yes	X			

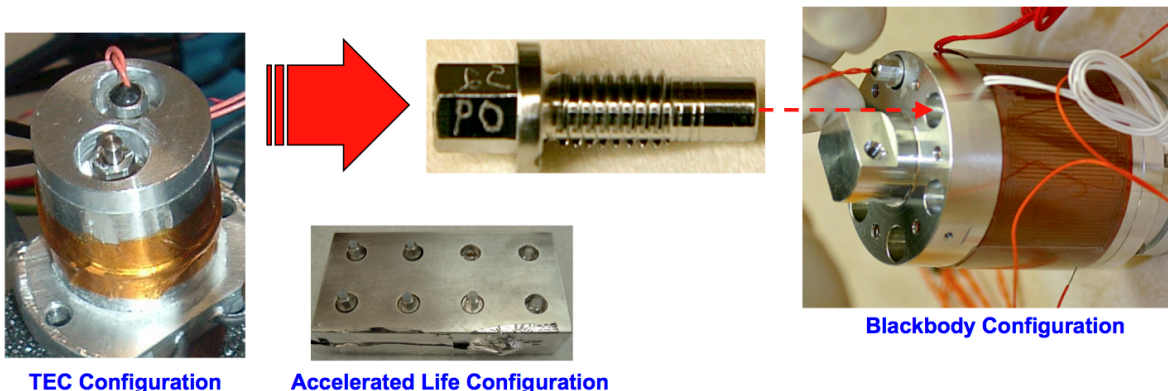


Figure 8. A table summarizing the test status is shown at top. The various test configurations are shown under the table. Signatures obtained in the blackbody configuration are close to those obtained in the TEC configuration.

Figure 9 shows an example of a weld integrity analysis for a gallium sample after exposure to an accelerated life test. A scanning electron microscope used in the energy dispersive spectroscopy mode can identify individual elements. In this case there is no evidence of a gallium breach due to the accelerated life testing. Similar analysis was used to verify that there was no evidence of gallium in grain boundaries of non-welded housings that went through a full warm soak, but abbreviated cycling tests.

The definitive test of the melt cell performance is the melt signature. Figures 10, 11, and 12 show the melt signature characterizations for gallium, water, and mercury respectively. In each plot the solid symbols were taken before the Full Accelerated Life Test (FALT), and the open symbols were taken following the test. Different numbers of samples in various orientations are included in each plot as described in the figure captions. For each of the three materials all signatures (pre and post test) fall within about 2mk of the characteristic mid-melt temperature versus melt length curve.

This excellent agreement highlights that the performance of the phase change cell configuration presented here should not be altered by the expected temperature environment, including warm soak and deep cycling.

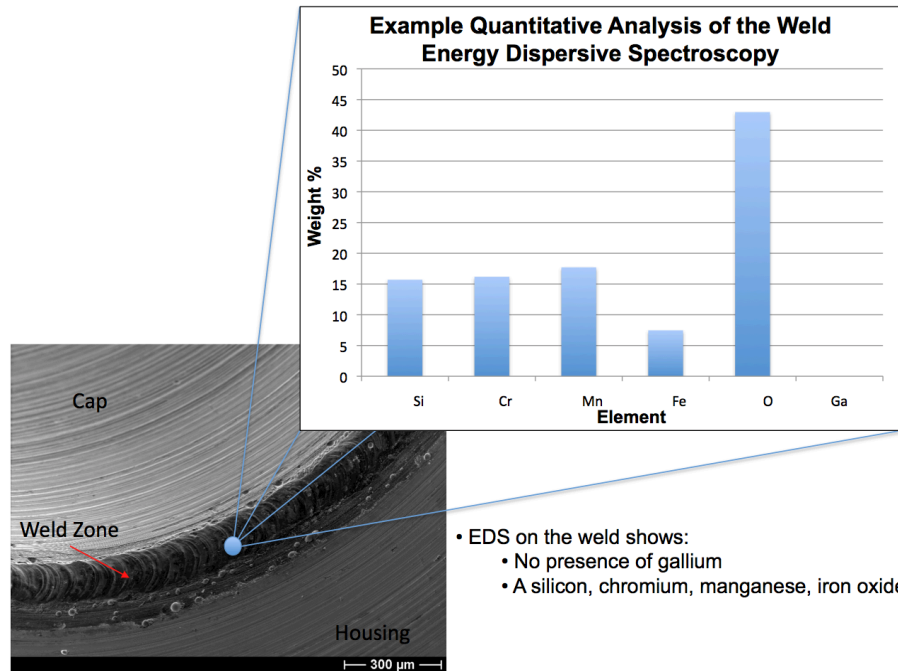


Figure 9. Weld analysis of a gallium phase change cell, using a scanning electron microscope used in the energy dispersive spectroscopy (EDS) mode can detect individual elements. In this case there is no evidence of a gallium breach through the weld.

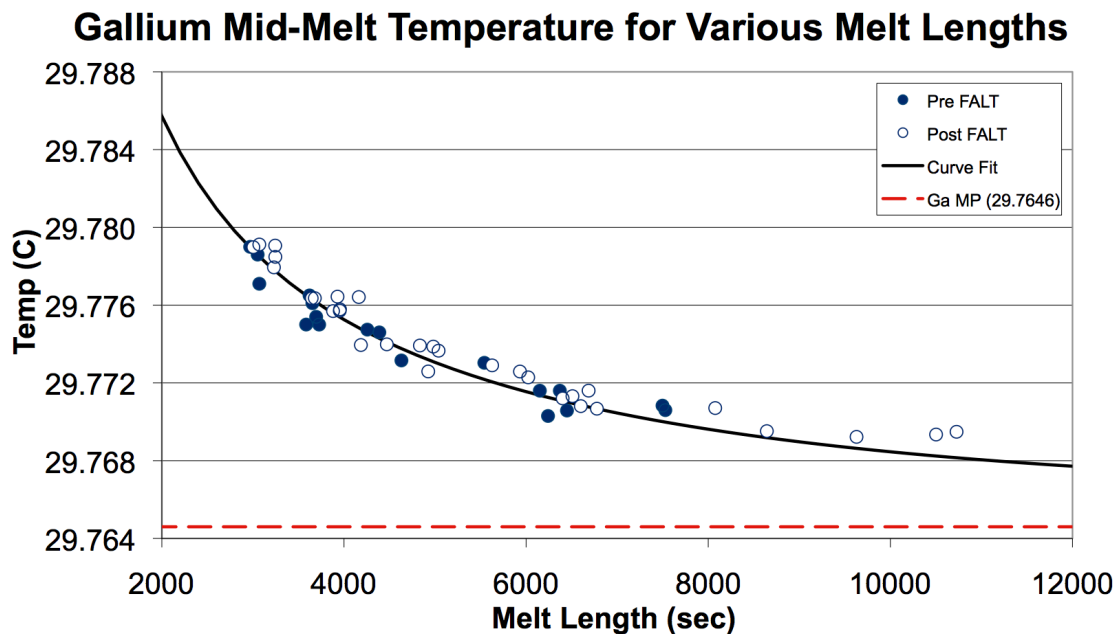


Figure 10. Gallium signature comparisons show excellent agreement between pre and post accelerated life testing. The plot includes data from six different samples, two were run upside-down.

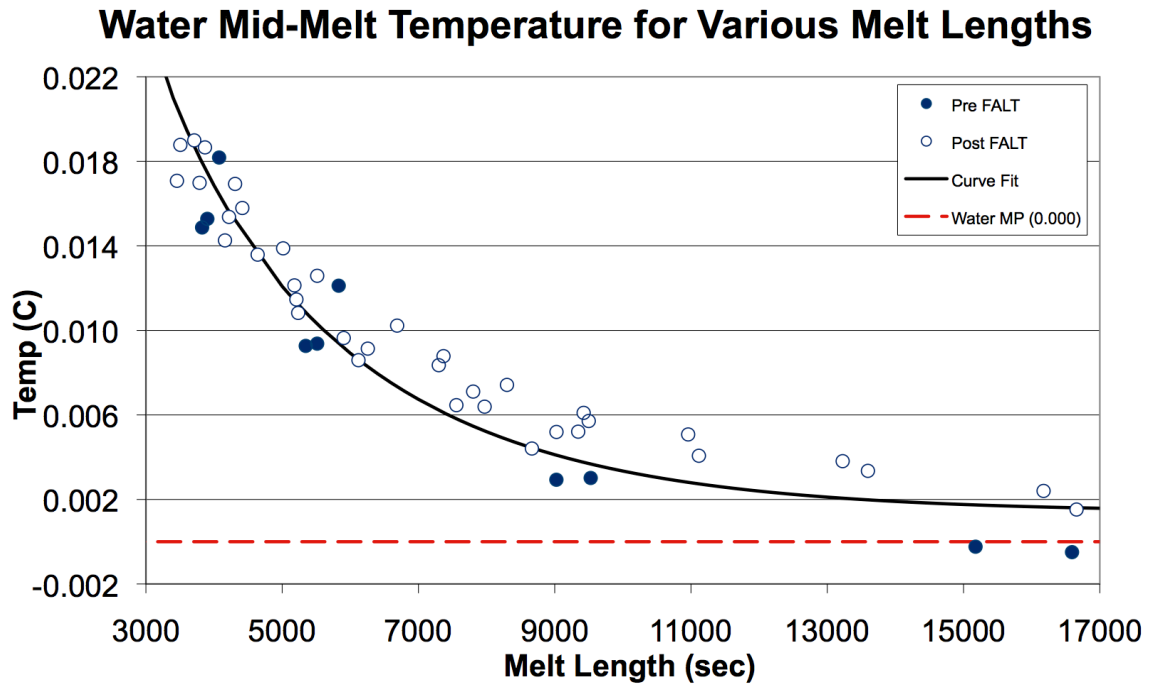


Figure 11. Water signature comparisons show excellent agreement between pre and post accelerated life testing. The plot includes data from three different housings in both normal and upside-down configurations.

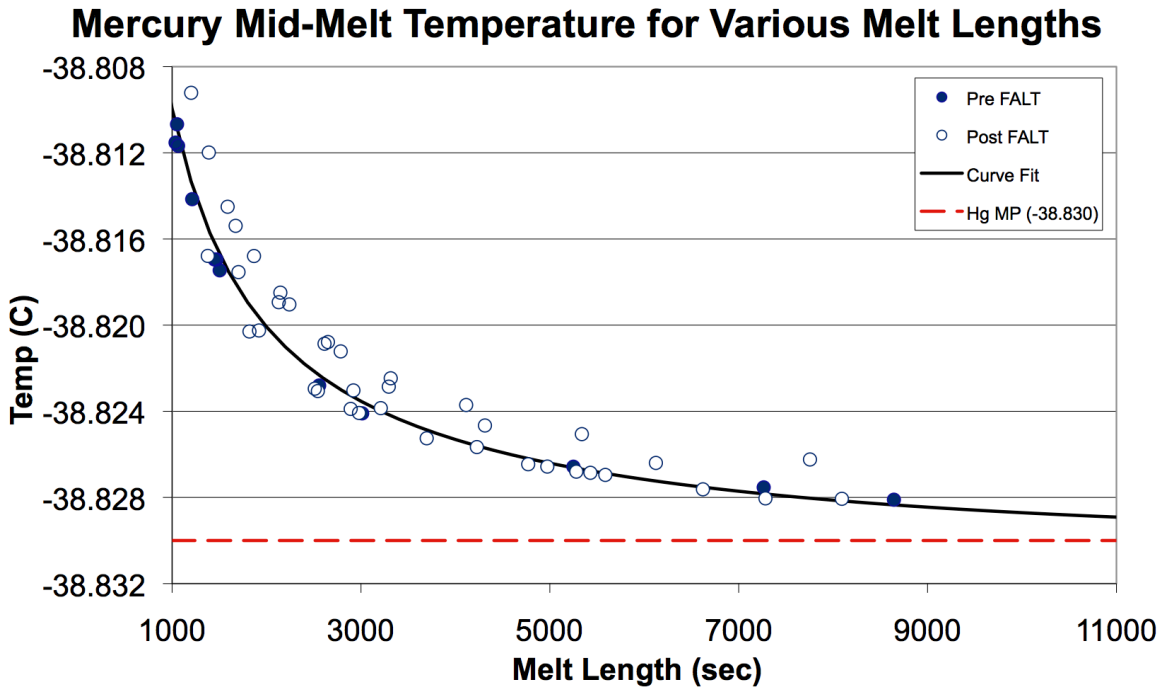


Figure 12. Mercury signature comparisons show excellent agreement between pre and post accelerated life testing. The plot includes data from four different housings in normal, upside-down, and sideways configurations.

6.0 MICROGRAVITY DEMONSTRATION ON THE ISS

NASA has provided a separate grant from the IIP to demonstrate the melt cell performance on the International Space Station, sometime within the next year. The configuration planned for this demonstration is shown in Figure 13. The phase change cells are contained in the aluminum melt block, along with the temperature sensor to be calibrated. The aluminum shield cap is thermally coupled to the top Thermo Electric Cooler (TEC) plate. Two TECs are sandwiched between the melt block and a heat sink, which the TECs use to draw/reject heat in order to heat and cool the melt block. The bottom TEC provides a level of isolation from ambient temperature fluctuations, which allows the top TEC to maintain greater stability. The outer case is held at constant temperature using a heater to provide a stable environment. Because the Experiment Support Package does not have the capability to get to the freezing point mercury, we plan to use a binary eutectic of gallium as our third material along with gallium and water.

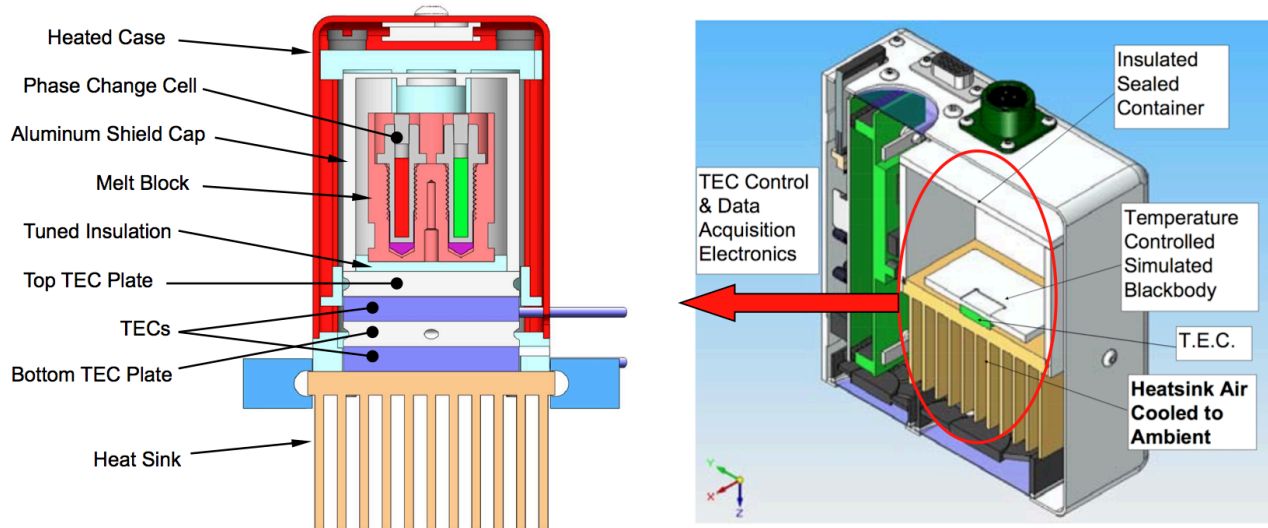


Figure 13. The configuration for microgravity demonstration of the phase change includes three different melt materials and makes use of the Experiment Support Package developed by Utah State Space Dynamics Laboratory.

To obtain a melt signature, the top and bottom TECs are first held at a temperature just below the melt point and the system allowed to reach thermal equilibrium. Then the temperature of the top TEC is increased slightly, and the melt block temperature slowly rises to a new equilibrium above the melt point. While the material is melting, the temperature of the melt block increases much more slowly, yielding a melt plateau that is used to calibrate the sensor.

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