Phase Change Cells

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Outline

- Phase change cell introduction
- Summary of ISS phase cell experiments
- Flight cell design improvements and ground test results
Need for Orbital Temperature Reference

• Phase transition cells for absolute temperature reference are key components of future climate monitoring missions

• Mission requirement:
  “…an SI-traceable standard for absolute spectrally resolved radiance in the infrared with high accuracy (0.1 K 3σ brightness temperature… Each of the interferometers carry, on-orbit, phase transition cells for absolute temperature,… with SI traceability [1].”

• Because temperature uncertainty will only be one of the contributors to the 0.1 K requirement, absolute temperature uncertainty will need to be lower, on the order of 0.01 K or better
Phase Transitions as Thermal References

ITS90 Phase Change Materials

<table>
<thead>
<tr>
<th>$T_{90}$/K</th>
<th>$t_{90}$/°C</th>
<th>Material</th>
<th>State</th>
<th>Uncertainty mK</th>
</tr>
</thead>
<tbody>
<tr>
<td>234.3146</td>
<td>-38.8344</td>
<td>Hg</td>
<td>Triple pt</td>
<td>0.2(0.1)</td>
</tr>
<tr>
<td>273.16</td>
<td>0.01</td>
<td>H$_2$O</td>
<td>Triple pt</td>
<td>0.05 (0.03)</td>
</tr>
<tr>
<td>302.9146</td>
<td>29.7646</td>
<td>Ga</td>
<td>Melt pt</td>
<td>0.2 (0.03)</td>
</tr>
</tbody>
</table>

- Large volume of PCM
- Long melt times
- Deep re-entrant wells
- Fragile containers
- Detailed manual heating and cooling procedures

Practical absolute uncertainty, $\sim$0.2 mK or better [2,3]
### SDL Phase Change Program History

<table>
<thead>
<tr>
<th>Year</th>
<th>Event Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>2004</td>
<td>Initial internal studies of potential for NPOESS started.</td>
</tr>
<tr>
<td>2005</td>
<td>VEGA Intl. approached to collaborate on gallium eutectic work.</td>
</tr>
<tr>
<td>2006</td>
<td>SDL &amp; IBMP agree to perform ISS testing of PCM cells.</td>
</tr>
<tr>
<td>2007</td>
<td>ESTO office begins funding support to speed development to benefit CLARREO with space qualification results.</td>
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<tr>
<td>2008</td>
<td>Vega Results show Ga eutectics as viable PCMs for calibrations.</td>
</tr>
<tr>
<td>2009</td>
<td>SDL starts IR&amp;D program to get ISS tests with PCM cells and patent technology.</td>
</tr>
<tr>
<td>2010</td>
<td>Launch opportunities delayed by Russian Calibration Institute approvals with ties to VEGA.</td>
</tr>
<tr>
<td>2011</td>
<td>Hardware launched on Soyuz, ISS experiments conducted.</td>
</tr>
<tr>
<td>2012</td>
<td>Hardware returned on Soyuz to IBMP and then to SDL.</td>
</tr>
</tbody>
</table>

**FY1** - **FY4**: SDL IR&D program.
Mini Orbital Temperature Reference (MOTR) Flight 1 Experiment Design for ISS

- PCM temperature controlled by 2 TECs and a temperature-controlled heater enclosure
- Heat exhausted to cabin through forced ventilation
- Automated experiment data stored on CF card, removable only on ground
- Expanding bellow PCM container ~1 mL volume
- Re-entrant sensor well
Flight 1 Experiment Data Analysis

- 19 on-orbit melt curves
- Temperature calibration from bath post-flight
- Multiple similar data sets obtained prior to and after flight
- Use average in plotted window as “melt curve temperature”
No continual drift trends observed over 4 years of melt data, indicating PCM contamination not a factor
All data sets are within 6 mK. Individual point within a given set vary by < ±1.5 mK
Summary of All MOTR Data Sets

ISS Data:  
Average Melt Temperature = $29.7731 \degree C$  
Standard Deviation = $0.0008 \degree C$  
Within ~0.6 mK

ALL Data:  
Average Melt Temperature = $29.7737 \degree C$  
Standard Deviation = $0.0017 \degree C$  
Within ~0.6 mK
Orbital PCM Reference Value Criteria

- Stable, reliable PCM containment
- Miniaturized assembly
- Includes multiple PCMs to improve calibration knowledge
- Capable of reliably and repeatably freezing and melting PCMs
- Capable of accurately measuring temperature during melting to within 10 mK absolute error
- Capable of tracking blackbody temperature to within a few mK
Transfer of Calibration

Calibration:
During a recalibration the TEC is powered and the PCM is controlled to a different temperature than the thermal surface to melt the PCM. Temperature data collected during the melt allows recalibration of the PCM sensor.

Transfer:
When the TEC is not powered it acts as a thermal link to the thermal surface. If adequately insulated it will come to equilibrium with the thermal surface. The PCM sensor can be compared to thermal surface sensors’ readings.
Orbital 3 PCM Cell Design

Phase-change cell with three cavities to be used for on-orbit temperature calibration

Secondary cap (SS 304)
Redundant welds contain PCM
Threaded primary cap (SS 304)
Phase-change material ~0.3 mL

Ga 29.7646°C
GaSn 20.46°C
GaIn 15.63°C

Thermistor
Thermistor locator (Teflon)
Cell body (SS 304)
Bath Bi-phase Equilibria Temperatures

- Gallium bi-phase equilibria were within <1 mK of the ITS90 Ga fixed point at 29.7646
- Most eutectic melt temperatures are reported to >10 mK absolute accuracies
• Unpowered PCM Cells equilibrated to within 2.5 mK of Inner Block Thermistors at 24 °C and at 1 °C.
• Agrees with thermal model delta of ~2 mK equilibrium
Thermal Model of a Ga Melt Curve

Condition: Blackbody at 273.15 K
Temp sensor between orange and red nodes

Piece of the melting gallium
Thermistor will read somewhere between these cell nodes
Gallium Gradients vs. Melt Time

Gradients in cell and duration of melt vary with heating power applied.

Mid-melt offsets in measurements agree with thermal model (~45-75 mK)
Ga Melt Point Repeatability

- TEC heating with constant current
- Copper block held at constant 8.5 ±0.01 °C
- Melt durations very consistent
- Plateau mid-point repeatability ~2 mK
GaIn Melt Point Repeatability

- Melt durations varied by ~13%
- Inconsistency possibly due to small constant current variations in the TEC
- Mid-points group to ~5 mK
- Clear correlation between mid-point temperatures and melt durations
GaSn Melt Point Repeatability

- Melt durations varied by ~20%
- Inconsistency possibly due to small constant TEC variations
- Mid-points group to ~5 mK
- Clear correlation between mid-point temperatures and melt durations
Conclusions

• Orbital testing of Ga phase transitions showed no change in melt point under 0G conditions and verified long-term repeatability of melt curves

• SDL improvements of design include smaller cells and addition of two new melt temperatures for GaSn and GaIn eutectics

• SDL ground testing of cells with Ga and eutectics demonstrates 2-3 mK repeatability of three melt temperatures

• Ground testing and thermal modeling improve understanding of:
  – thermal gradients within cells and their effect on interpretation of melt curves
  – true bi-phase equilibrium temperatures of melt materials

Questions?
References


5. GE Sensing, “Thermometric Ultrastable Probe Thermistors,” SP series Datasheet, [copyright 2006].

Additional Slides
Sealed Cells vs. Pressure Dependence of Fixed-Point

- For contamination issues PCM containers must be sealed
- 1 atm pressure changes melt temperature of water by 10 mK [3]
- Container must allow PCM expansion without changing fixed-point temperature

Flexible container:
- No internal voids
- PCM can expand container
- PCM vacuum filled
- Complex filling
- Complex container
- Moving parts

Rigid container:
- PCM filled at 1 atm
- Internal gas voids compress as PCM expands
- Location of voids in space?
SDL Temperature Sensor Testing

- Heraeus PRT and GE thermistor excellent size and long term stability [5,6]
- GE Thermistors tracked standards PRT ±3mK, with calibration improvement to ~1mK
- Heraeus PRTs tracked ±10-15mK (worse than larger wire PRTs)
- Heraeus shock resistance 40g at 10-2kHz
Ga Melts of Various Lengths

1-2 hour melt curves are ~50-60 mK higher than melt point at center of melt due to cell thermal gradients. Agrees with thermal models which predict ~50-80mK. Longer melts within 5 mK of true melt temperature.
GaSn Melts of Various Lengths

1-2 hour melts are only ~30-40 mK above eutectic melt point
GaIn Melts of Various Lengths

1 hour melts are 50 mK above eutectic melt point
>5 hour melt was within 5 mK of eutectic melt point when it was stopped
Supercooling Behaviors

Ga and eutectics cool below their melt points after reaching temperatures well above as liquids.
GaIn Behavior Improvements

- Tested in bath at slower ramp rates
- Ga rich mixture shows slightly less supercooling
Acknowledgements

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Questions?