Cryogenic Thermal Conductivity Measurements on Candidate Materials for Space Missions

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Introduction

• Many NASA missions include cryogenic instruments
• Spacecraft and instruments include optimized materials/assemblies
  - Highly-conductive annealed pure metals
  - Engineered materials
    Polymers
    Alloys
    Composites
    Ceramics
  - Customized electrical cables/harnesses

• Candidate materials often selected based on room temp. properties
• Often longitudinal cryogenic thermal conductivity is unknown
• We developed a thermal conductivity facility for JWST in 2004
• We have characterized ~ 30 samples since then
Concept

• For one-dimensional heat flow in a material,

\[ \frac{\dot{Q}}{A} = \kappa \frac{dT}{dx} \]

- \( \kappa \): thermal conductivity [W/m/K]
- \( \dot{q} \): power [W]
- \( A \): cross sectional area [m\(^2\)]
- \( T \): temperature [K]
- \( x \): axial distance [m]

• Basic approach
  - Flow heat through sample
  - Measure temperature gradient

• We chose to perform “absolute” measurement
  - Relative measurements: lower precision
Simple Approach

• Control base temperature
• Apply heat to sample’s free end
• Measure (small) $\Delta T$

$$\kappa(\bar{T}) = \frac{L \dot{Q}}{A \Delta T}$$

• $L$: sample length [m]
• $A$: cross section [m$^2$]
• $\bar{T} = \frac{T_{\text{Sample}} + T_{\text{Base}}}{2}$
Complications

• Ohmic heating in heater leads
• Heat conducted in leads
• Heat radiated to surroundings
  \[ T_{\text{Hot}}^4 - T_{\text{Cold}}^4 \sim 4\bar{T}^3 \Delta T \]
• Joint resistance at base
• Joint resistance at floating end
• Absolute thermometer errors

\[
\frac{\dot{Q}_H - \dot{Q}_{TL} - \dot{Q}_{HL} - \dot{Q}_R}{A} = \kappa(\bar{T}) \frac{(T_F - \delta T_F) - (T_B - \delta T_B) + \Delta T_F + \Delta T_B}{L}
\]
Our Test Configuration

- Based on approach described in 1973 Moore, Williams and Graves RSI paper
- Guard surrounds sample: Controlling $T_{\text{Guard Top}} = T_{\text{Sample Top}}$ reduces sample heat radiation
- “Fiberfrax” insulation eliminates remaining sample radiation
- Intermediate thermometers eliminate joint resistance effect
- Optimizing sample heater and leads minimizes ohmic heating in leads
- Lead heat-sinking minimizes lead heat conduction
Instrumentation

- **Thermometers**
  - LakeShore Cryotronics SD-package Cernox™ sensors
  - Calibrated (resistance vs. $T$) from 1 to 325 K

- **Heaters**
  - Sample heater is 10 KΩ metal-film resistor
    - Leads: size, material chosen to give round-trip resistance less than ~10 Ω inside guard
  - Base and guard heaters: 50 Ω
    - made by winding stainless steel wire around flange
    - we don’t measure the power for these heaters

- **Temperature readout/control boxes**
  - Cryogenic Control Systems Cryocon Model 32B Controller

- **Heater voltage and current readout**
  - Keithley Model 2000 6.5-digit multi-meters
Data Acquisition and Analysis

- For each value of \( \bar{T} = \frac{(T_{\text{Sample}} + T_{\text{Base}})}{2} \):
  - Perform 4 different steady-state ”balances”
  - For each balance, control \( T_{\text{guard}} = T_{\text{Sample}} > T_{\text{Base}} \)
  - Measure \( \Delta T = T_{\text{Far}} - T_{\text{Near}} \)
  - Measure \( \dot{Q} = \) sample control power

\[
\kappa(\bar{T}) = \frac{L}{A} \frac{d\dot{Q}}{d\Delta T}
\]

- To first order, differential measurement eliminates effect of absolute temperature errors
  - \( \frac{d\dot{Q}}{d\Delta T} \) is more accurate than any single \( \frac{\dot{Q}}{\Delta T} \) value

- Least-squares fit of 4 different \( \Delta T \) values provides statistical uncertainty in \( \frac{d\dot{Q}}{d\Delta T} \)
Effect of Cal. Curve “Scatter”

- Thermometer $R$ vs. $T$ calibrations have “scatter” due to measurement uncertainty
- Assume that “true” $R(T)$ is a smooth function approximated by a smoothing fit
  - LakeShore Cryotronics provides smoothing Chebyshev Polynomial fits
  - We performed cubic spline smoothing fit on a cal. curve
- Our readout box uses cubic spline interpolation to get $T$ from $R$
  - Interpolation forces curve to go through every “scattered” point
  - Causes local $dR/dT$ errors relative to slope of “true” smooth curve
  - A local error in $dR/dT$ results in a proportional local error in $\kappa$
Evaluation of Cal. Curve Slope

- Graphed slope difference between spline-smoothed curve and spline interpolations:
  - Blue curve: interpolation of raw calibration points
  - Red curve: interpolation of Chebychev fit points
- Above 6 K, raw points give max. slope error of 0.3% (mostly below 0.2%)
- Improvement is possible by loading Chebychev fit points into readout box
Sample/Guard Mismatch Error

- To first order, keeping \( T_{\text{Sample}} = T_{\text{Guard}} \) eliminates effect of sample-guard heat leaks
  - For small \( \Delta T \) values, \( T_{\text{Sample}} - T_{\text{Guard}} \) calibration curve mismatches are assumed constant for balances with a given \( \bar{T} \)
  - Constant mismatches result in constant sample-guard heat leak
  - This does not effect \( \frac{d\dot{Q}}{d\Delta T} \)

- However, Fiberfrax effective thermal conductivity has a strong \( (T^3) \) temperature dependence

- We performed finite-element thermal model to evaluate second order effects in \( \frac{d\dot{Q}}{d\Delta T} \)
Mismatch Error for PVC Sample

- Worst-case error at 300 K

- PVC has very low $\kappa = 0.16 \text{ W/m/K}$ at 300 K

- Modeled error vs. sample diameter inside 32 mm guard

- It’s best to make sample diameter as large as practical

- This error is proportional to $1/\kappa$, so much lower for other materials
High Conductivity Samples

Cryogenics
and Fluids
Branch

- aluminum 1350
- AlBeMet: longitudinal direction
- AlBeMet: transverse direction
Medium Conductivity Samples

- aluminum nitride
- aluminum 6061-T6
Polymer Samples

Cryogenics and Fluids Branch

Epon 815 epoxy
Teflon sheet (rolled, longitudinal direction)
Torlon (extruded)  NIST fit for bulk Teflon

\[ \kappa (W/m/K) \]

Temperature (K)

\[ 0.10 \]
\[ 0.20 \]
\[ 0.30 \]
\[ 0.40 \]
\[ 0.50 \]
\[ 0.60 \]
\[ 0.70 \]
\[ 0.80 \]
\[ 0.90 \]
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Composite Samples

S-glass [(45, -45, 0)s layup, S2 glass in EX1522 epoxy matrix]

T300 [(45, 0, -45)2s layup, T300 carbon fiber with 5HS weave in RS-3C epoxy matrix]
Conclusions

• It’s not too difficult to perform high-precision thermal conductivity measurements between 4 K and room temperature

• NASA/GSFC’s cryogenics group is equipped to perform such measurements for customers at any NASA center

• Thanks to the James Webb Space Telescope program, which funded the development of the technique and facility