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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,

$$\frac{\dot{Q}}{A} = \kappa \frac{dT}{dx}$$

  - κ: 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

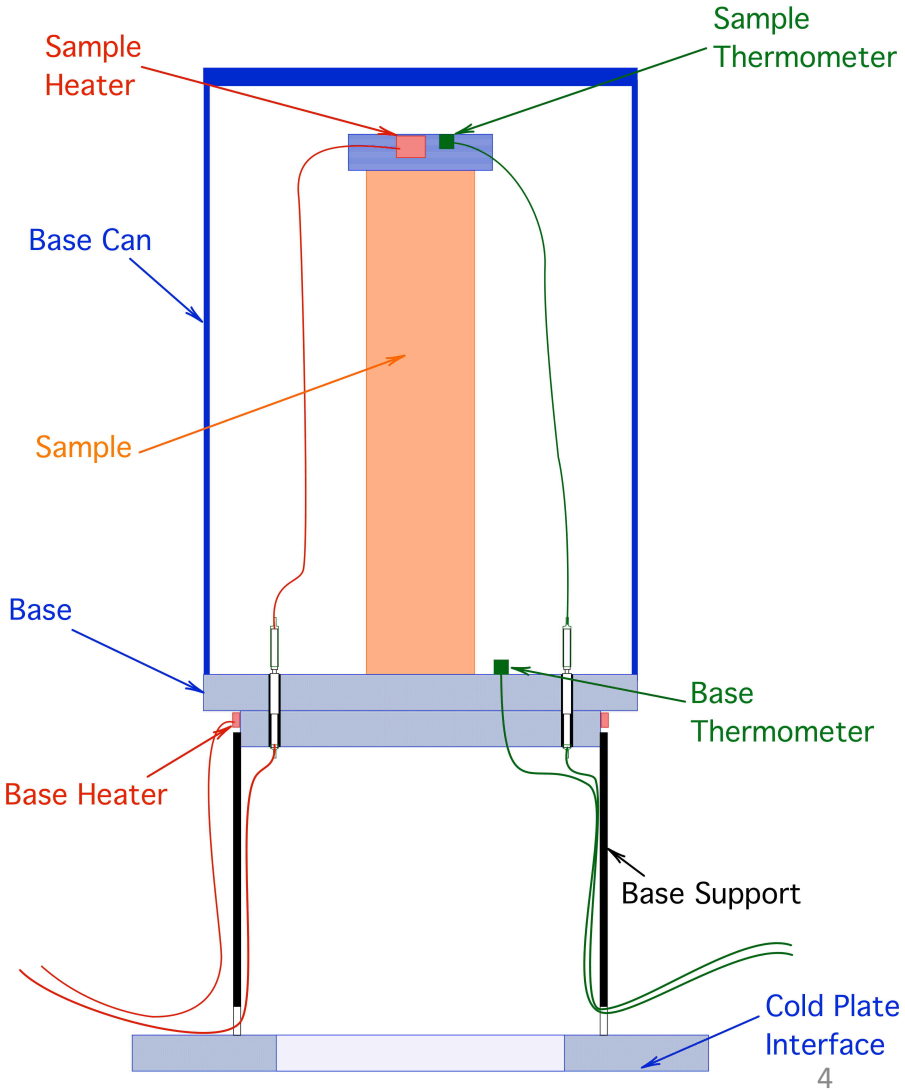
- We chose to perform "absolute" measurement
  - Relative measurements: lower precision

Simple Approach

- Control base temperature
- Apply heat to sample's free end
- Measure (small) ΔT

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

- L: sample length [m]
- A: cross section [m^2]
- $\bar{T} = (T_{\text{Sample}} + T_{\text{Base}})/2$



Complications

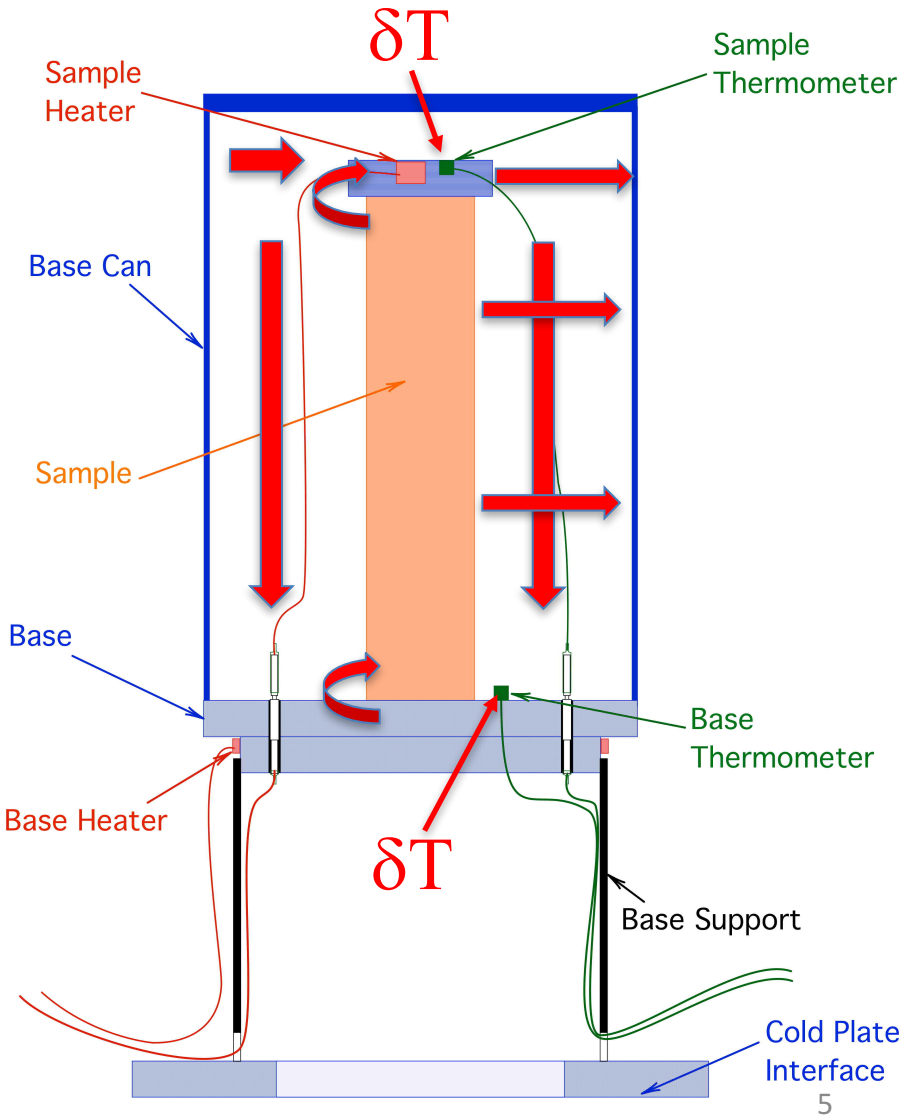
- Ohmic heating in heater leads
- Heat conducted in leads
- Heat radiated to surroundings

$$T_{Hot}^4 - T_{Cold}^4 \sim 4\bar{T}^3 \Delta T$$

- Joint resistance at base
- Joint resistance at floating end
- Absolute thermometer errors

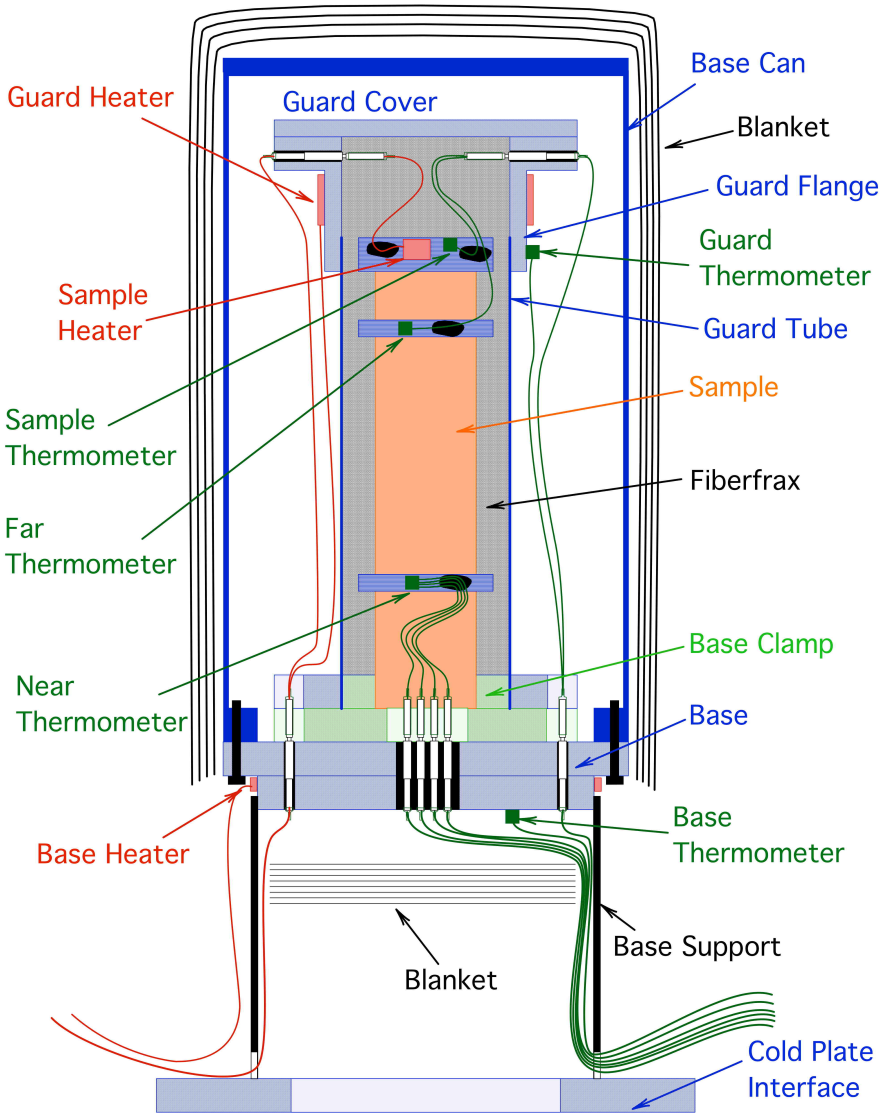
$$\frac{\dot{Q}_H - \dot{Q}_{TL} - \dot{Q}_{HL} - \dot{Q}_R}{A} =$$

$$\kappa(\bar{T})\,\frac{(T_F - \delta T_F) - (T_B - \delta T_B) + \Delta T_F + \Delta T_B}{L}$$



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

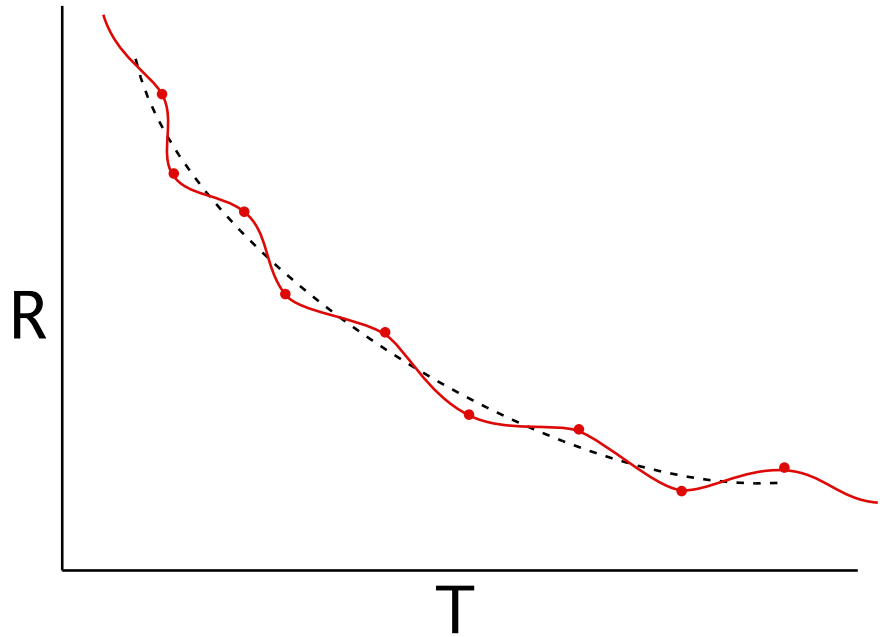
Data Acquisition and Analysis

- For each value of $\bar{T} = (T_{\text{Sample}} + T_{\text{Base}})/2$:
  - 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 ΔT values provides statistical uncertainty in $\frac{d\dot{Q}}{d\Delta T}$

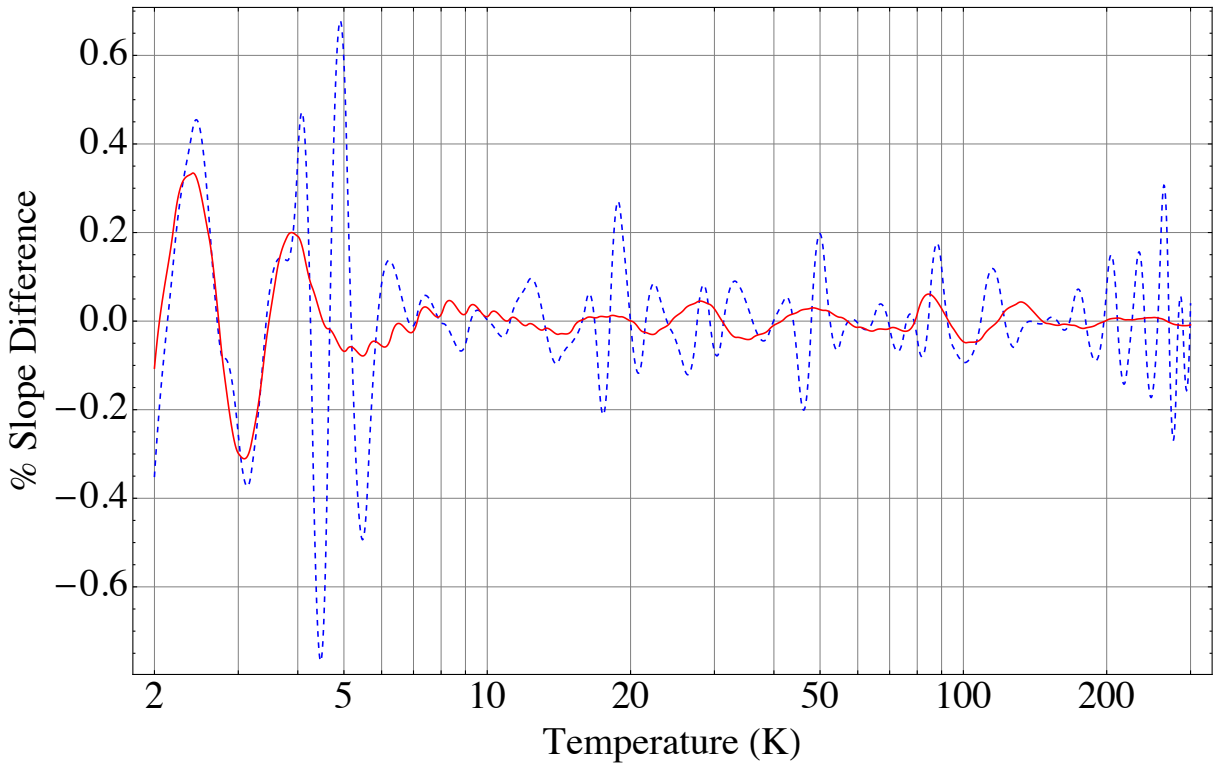
Effect of Cal. Curve "Scatter"



- 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 d*R*/d*T* results in a proportional local error in κ

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

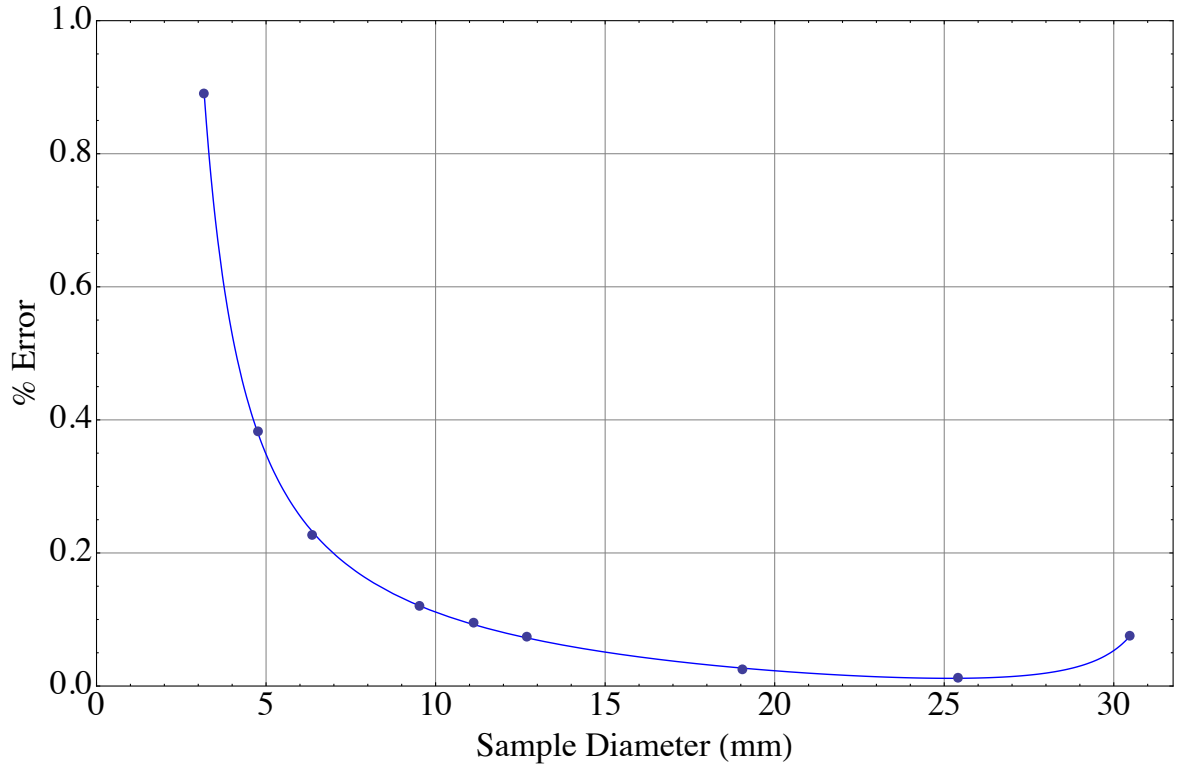
- To first order, keeping $T_{\text{Sample}} = T_{\text{Guard}}$ eliminates effect of sample-guard heat leaks
  - For small ΔT values, T_{Sample} - T_{Guard} calibration curve mismatches are assumed constant for balances with a given $\bar{T}$
  - Constant mismatches result in constant sample-guard heat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

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

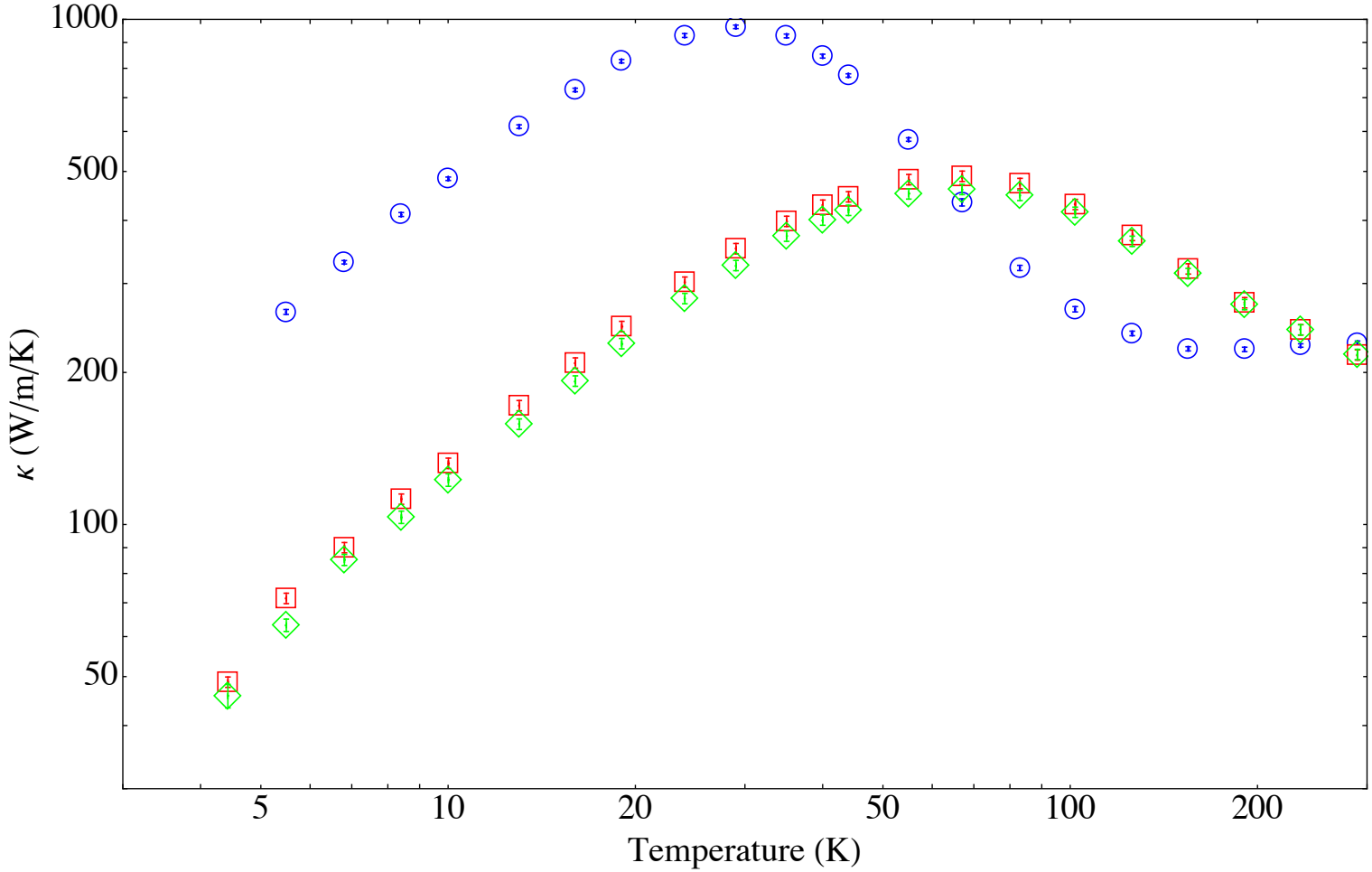
- We performed finite-element thermal model to evaluate second order effects in $\frac{d\dot{Q}}{d\Delta T}$

Mismatch Error for PVC Sample

- Worst-case error at 300 K
- PVC has very low $\kappa = 0.16$ 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

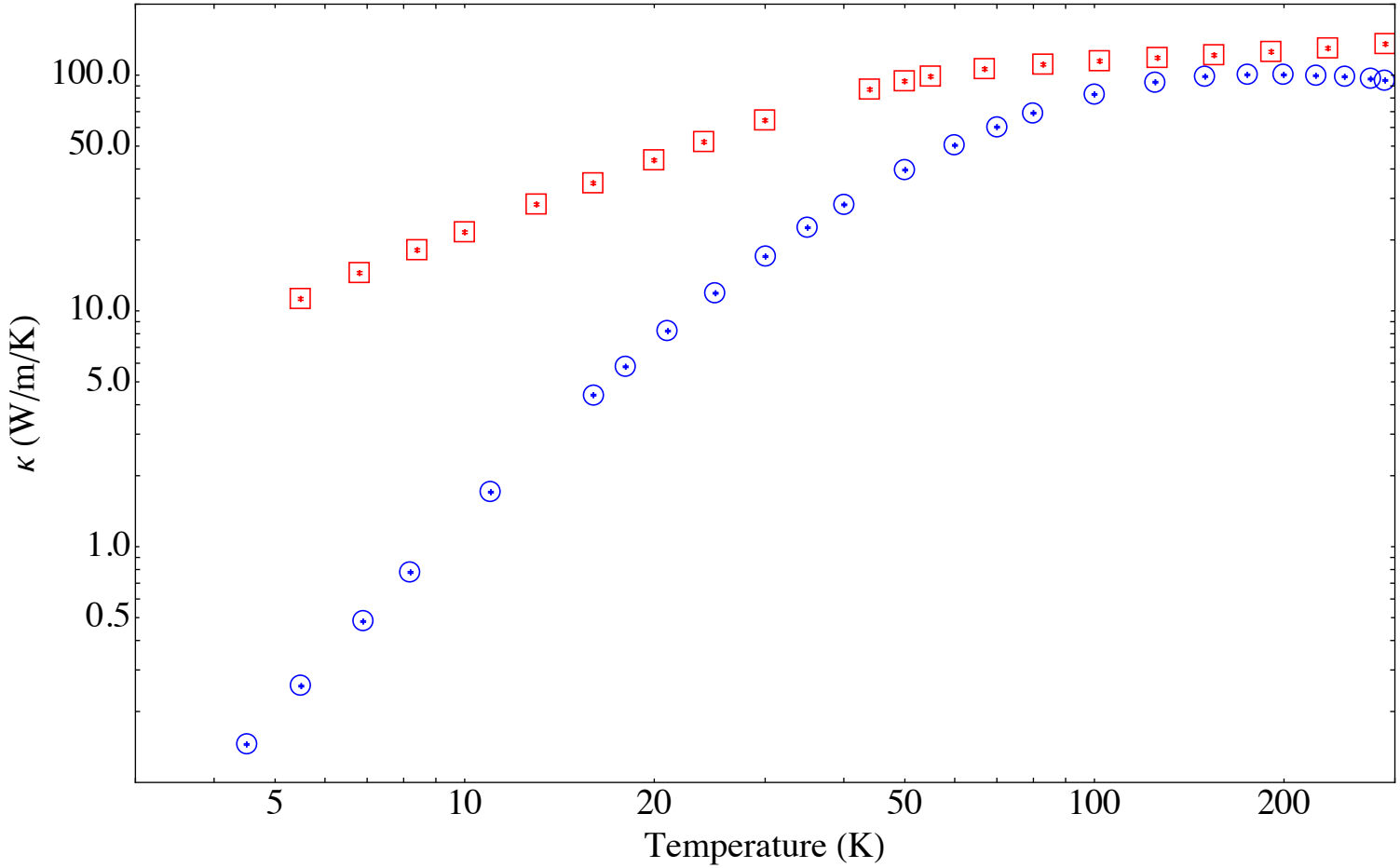


High Conductivity Samples



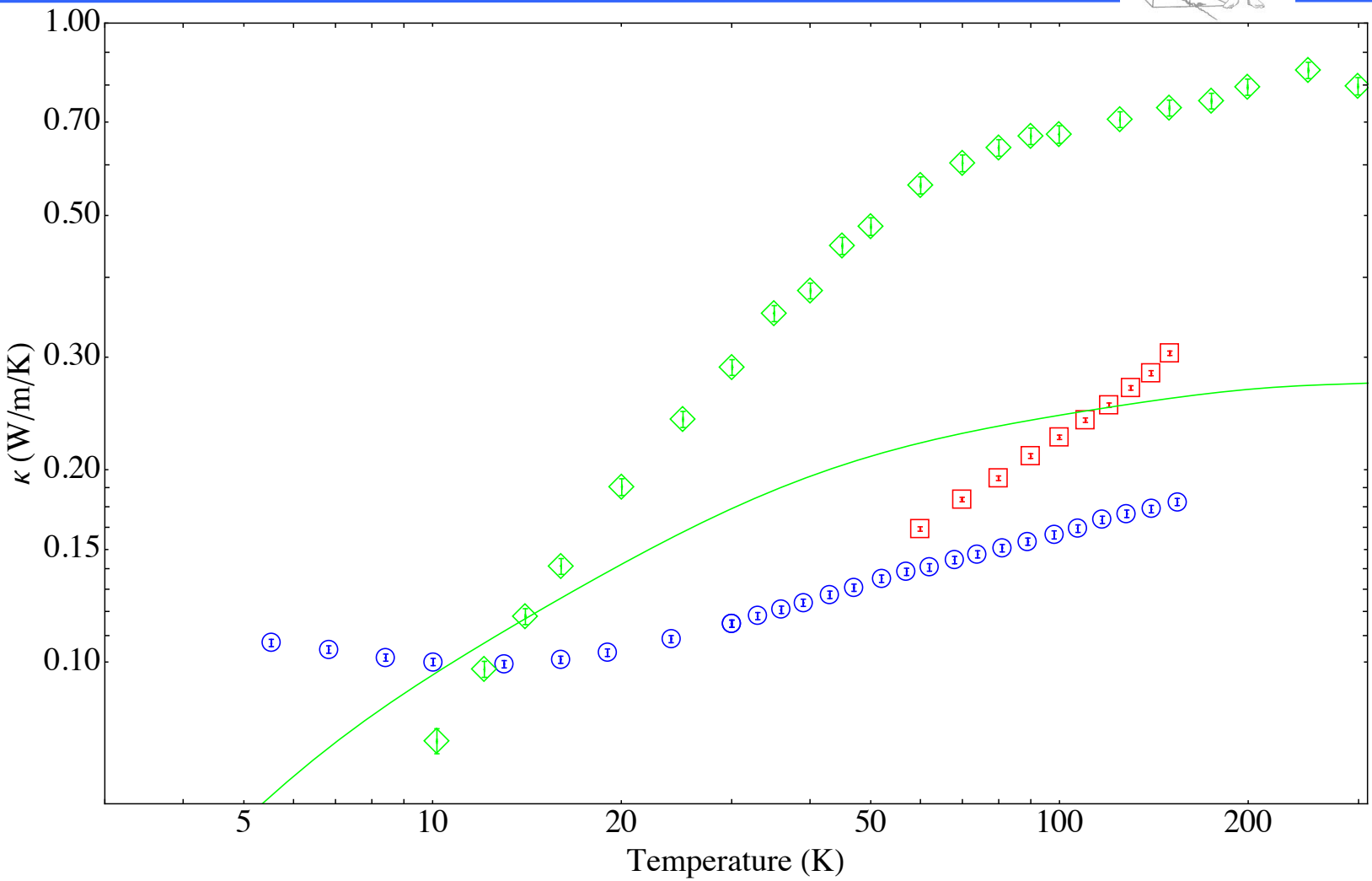
○ aluminum 1350
□ AlBeMet: longitudinal direction
◇ AlBeMet: transverse direction

Medium Conductivity Samples



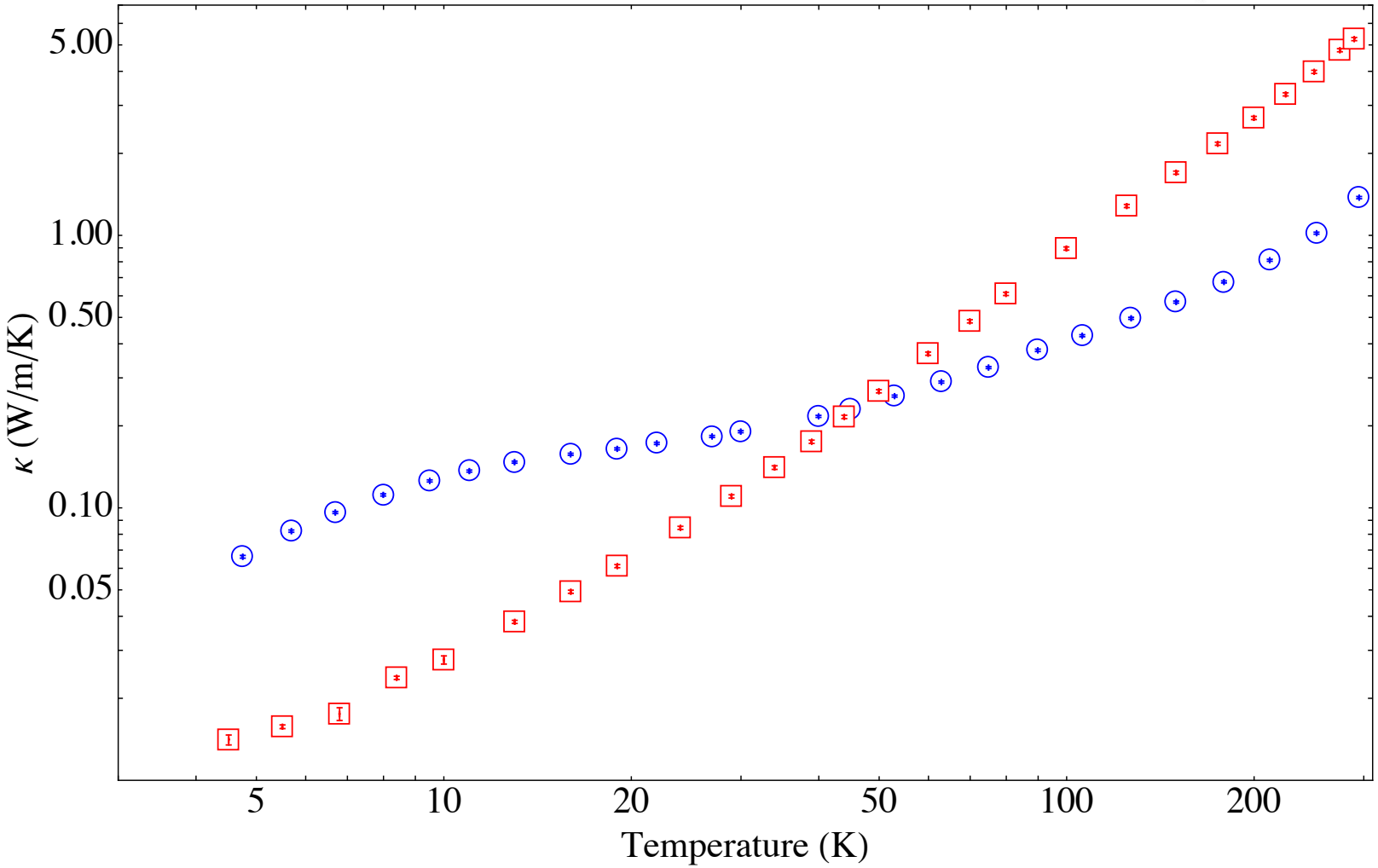
○ aluminum nitride
□ aluminum 6061-T6

Polymer Samples



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

Composite Samples



○ S-glass [(45,-45,0)4s 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