

## Cryogenic Thermophysical Properties Measurements at NASA - Goddard Space Flight Center



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# Introduction



The cryogenics and fluids branch at NASA's Goddard Space Flight center has the experience and capabilities to perform the following cryogenics measurements:

- Thermal conductivity
- Electrical resistivity
- Specific heat capacity
- Emissivity
- Absorptivity

& currently under development:

- CTE measurements



# Introduction



- Many NASA missions include cryogenic instruments and parts that need to operate at cryogenic temperatures
- It is common for NASA engineers to propose new candidate materials which have not been completely characterized at cryogenic temperatures
- Selection of these materials often rely on meeting specific criteria (e.g structural components may need to possess low thermal conductivity and high strength, or harnesses may need to have low electrical resistivity and low thermal conductivity, etc.)
- The cryogenics and fluids branch at NASA Goddard Space Flight Center has successfully conducted thermophysical properties measurements of many materials for various missions including the James Webb Space Telescope.

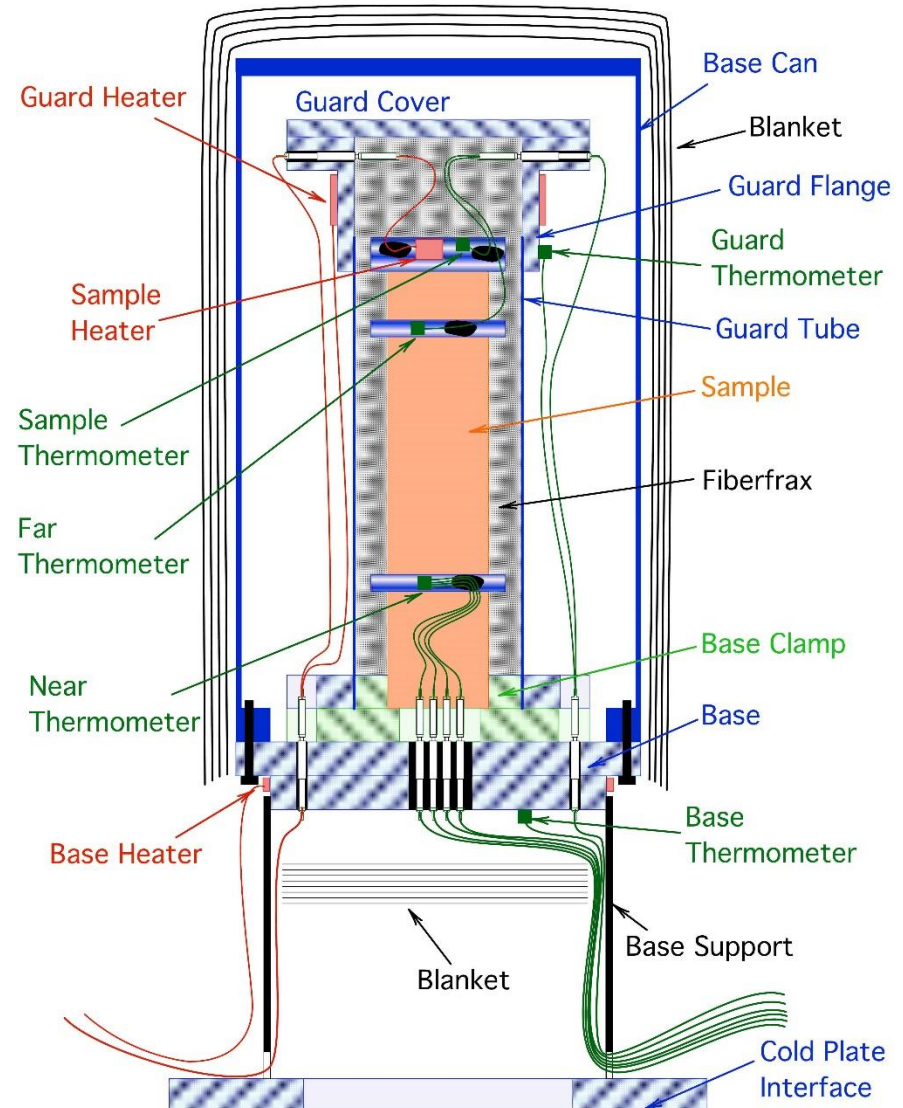


# Thermal conductivity - Intro



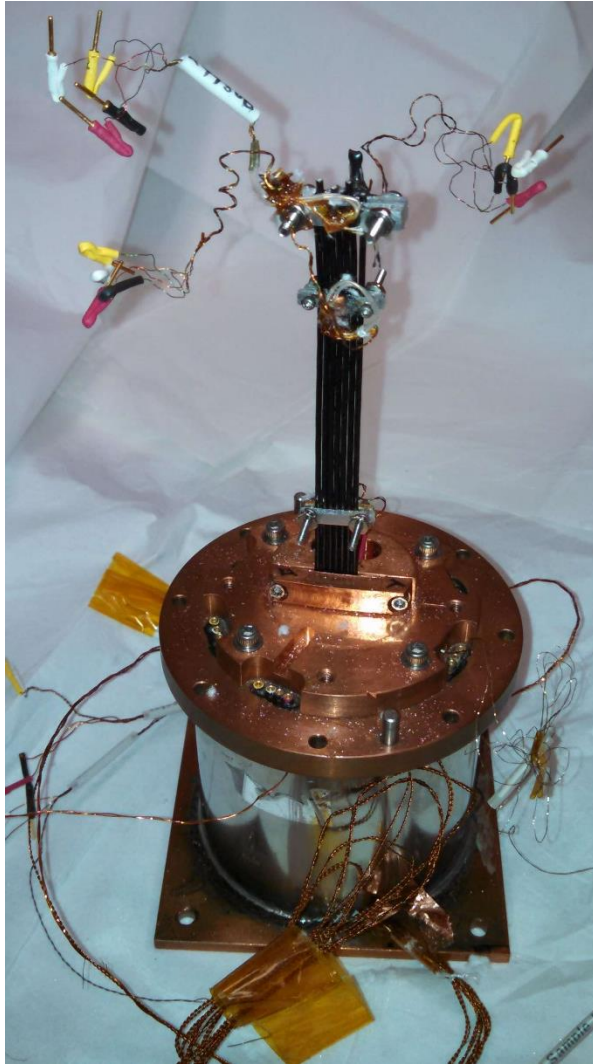
- Spacecraft and instruments include optimized materials/assemblies
  - Highly-conductive annealed pure metals
  - Engineered materials
    - Polymers
    - Alloys
    - Composites
    - Ceramics
  - Customized electrical cables/harnesses
- Candidate materials often selected based on room temp. properties
- Often longitudinal cryogenic thermal conductivity is unknown
- We developed a thermal conductivity facility for JWST in 2004
- We have characterized more than 30 samples since then

- 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

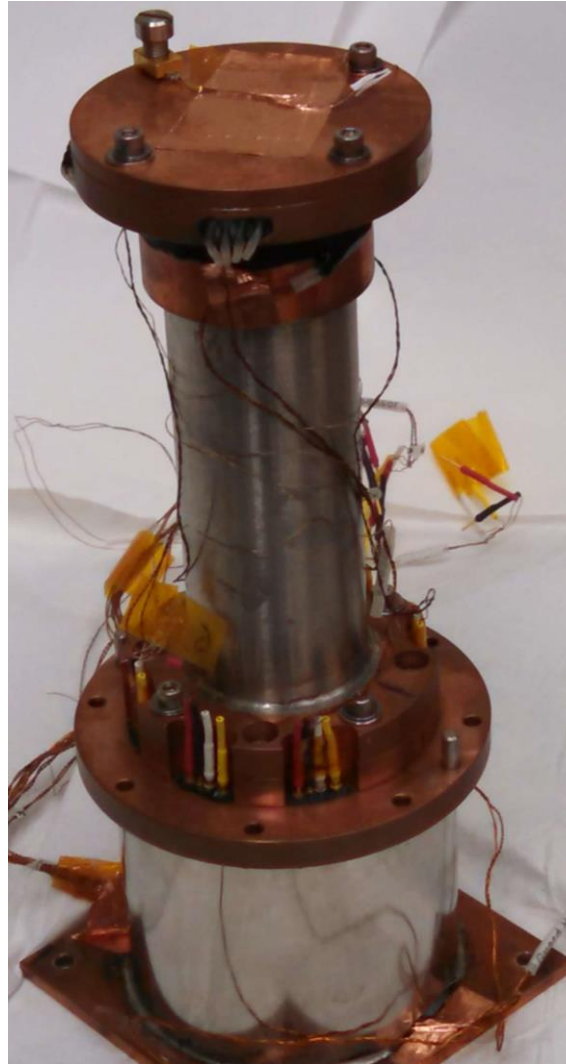




# Thermal conductivity - setup



Test sample installed on base



Guard installed and closed



Guard flange; Fiberfrax



Blanket



# Thermal conductivity - considerations



- Thermometers
  - LakeShore Cryotronics SD-package Cernox™ sensors
  - Calibrated (resistance vs.  $T$ ) from 1 to 325 K
- Heaters
  - Sample heater is 10 K $\Omega$  metal-film resistor
    - Leads: size, material chosen to give round-trip resistance less than  $\sim 10 \Omega$  inside guard
  - Base and guard heaters: 50  $\Omega$ 
    - 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



# Thermal conductivity - analysis



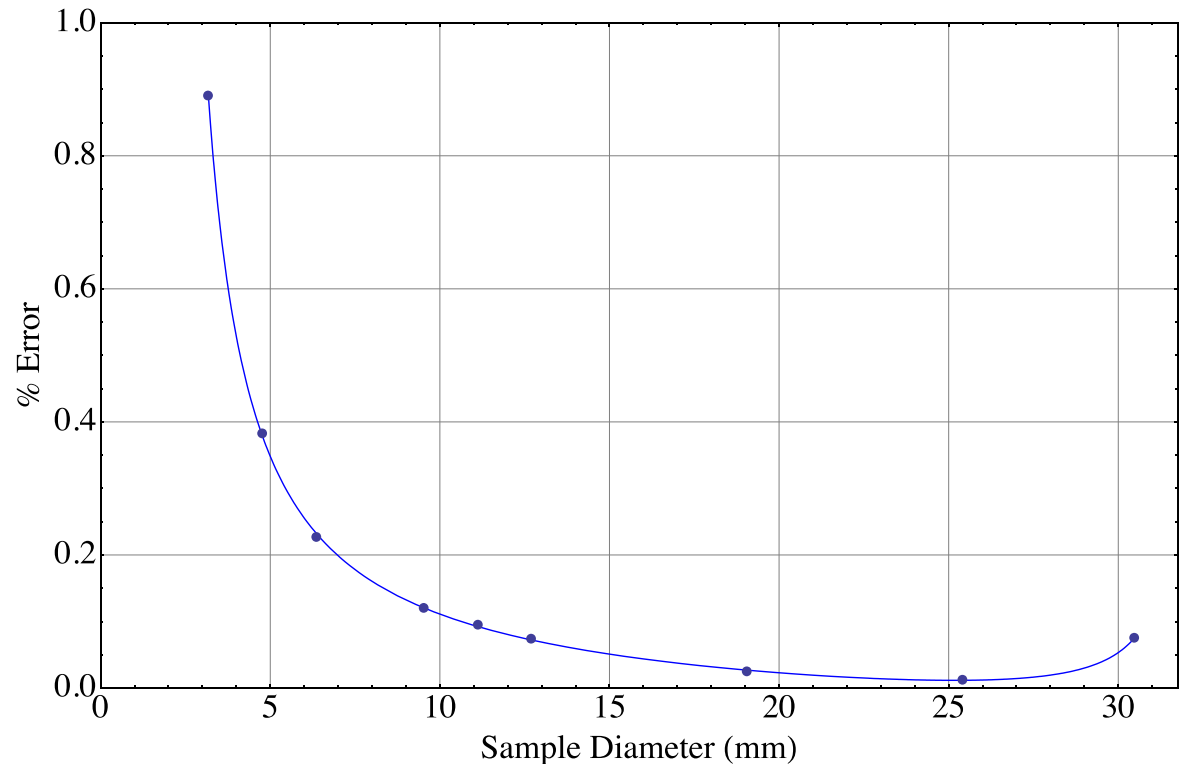
- For each value of  $\bar{T} = (T_{\text{Sample}} + T_{\text{Base}})/2$ :
  - Perform 4 different steady-state "balances"
  - For each balance, control  $T_{\text{guard}} = T_{\text{Sample}} > T_{\text{Base}}$
  - Measure  $\Delta T = T_{\text{Far}} - T_{\text{Near}}$
  - Measure  $\dot{Q}$  = sample 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  $\Delta T$  values provides statistical uncertainty in  $\frac{d\dot{Q}}{d\Delta T}$



- 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





# Electrical resistivity



- Electrical wiring for cryogenic systems is typically optimized to meet conflicting thermal and electrical performance requirements.
- Samples of materials are used appropriately to enable accurate measurements of electrical resistivity.
- The material is electrically isolated, yet heat sunk well to an isothermal “platform” and cooled down via the cryocooler.
- A heater and thermometer embedded in the test plate enables precise temperature control of the sample.
- We have used precise resistance bridges such as a Picowatt AVS-47 or an LR-700 to accurately measure electrical resistivity of samples.

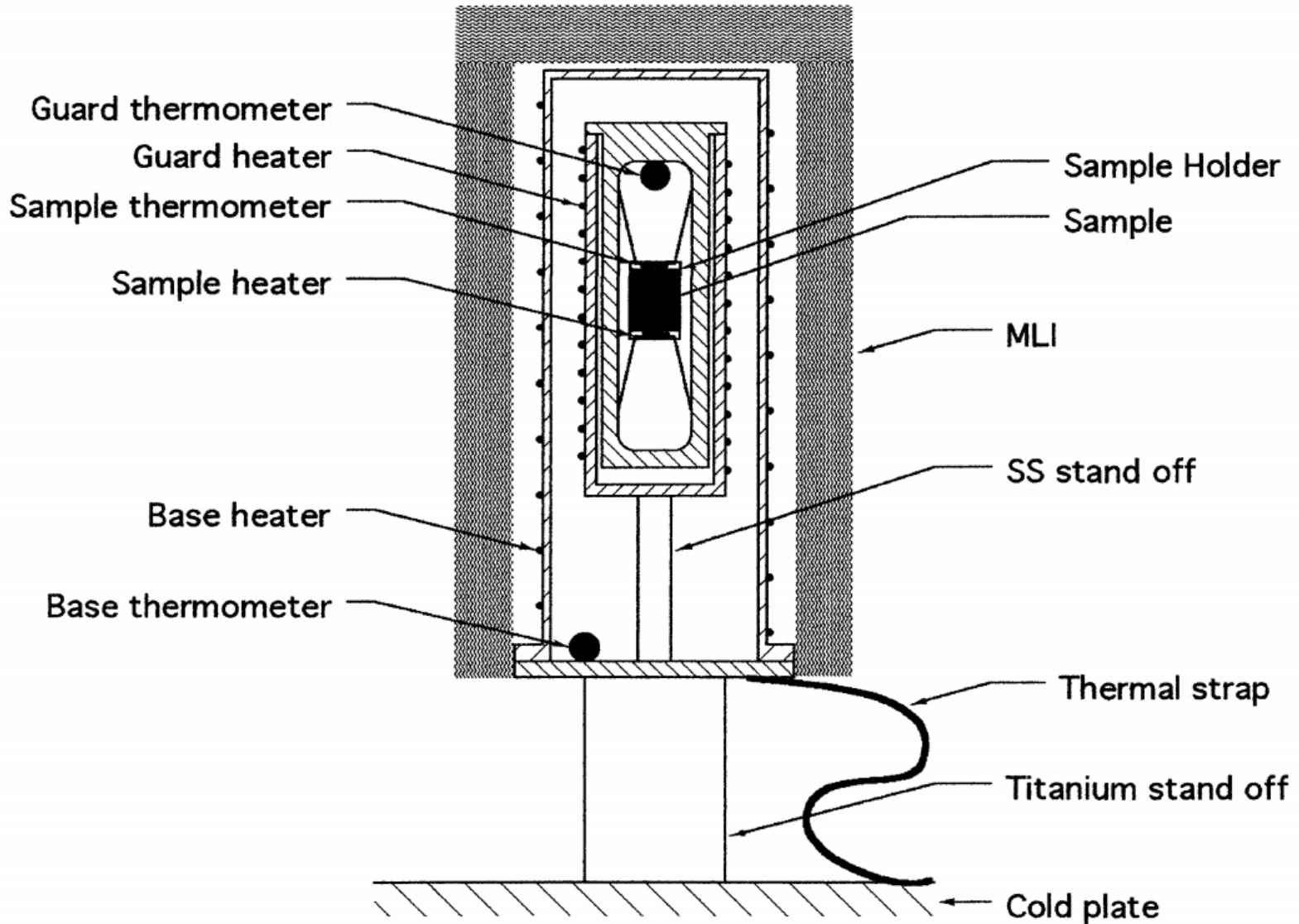


# Specific heat capacity - Intro



- Candidate structural materials for the ISIM on JWST needed to be characterized through cryogenic heat capacity measurement.
- A test configuration was designed and techniques devised to measure the specific heat capacity of several material between 4 and 290 K.
- Issues of radiative heat transfer onto the sample must have been addressed.
- Measurement was conducted with the pulse technique.
- Sample box measures 12 mm x 5 mm x 10 mm
- Cernox thermometers and metal film resistors were used as heaters for the measurement.

# Specific heat capacity - setup





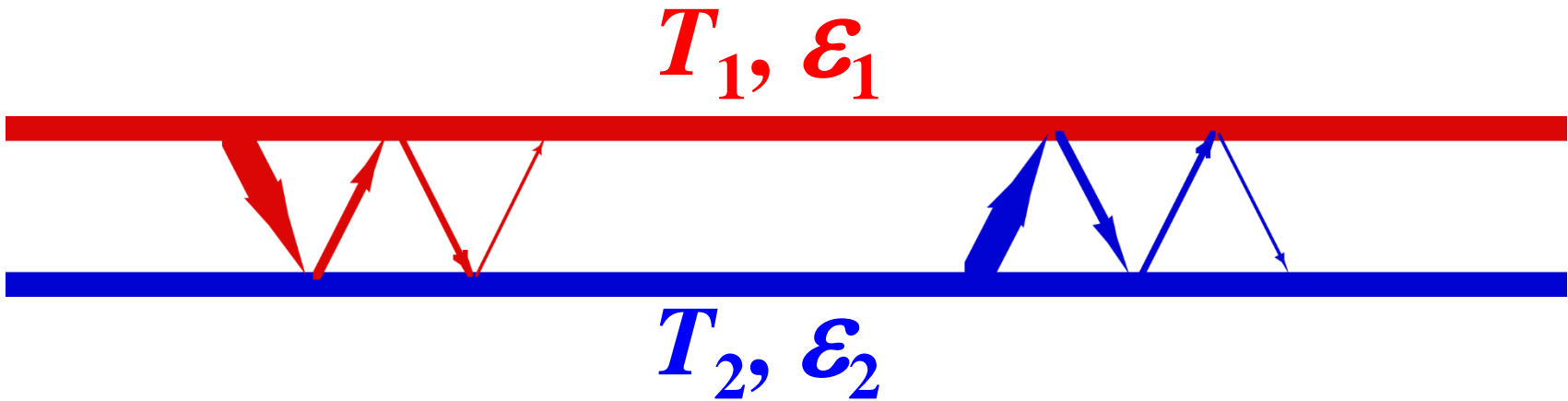
# Specific heat capacity - analysis



- The data acquisition process is automated by a LabVIEW program.
- For each data point the setpoint of the guard temperature controller is set equal to the sample temperature.
- The base temperature is set slightly below that of the guard to maintain positive control on the guard.
- Temperature gradient across guard is negligible even at high temperatures.
- Program waits until slope of base and guard temperature is zero within the error of fit, and slope of the sample temperature is constant.
- Once program detects steady state a heat pulse of known width and height is fired into the sample heater and waits for a new steady state condition:
  - 1) The quadratic term in the sample temperature is zero within the uncertainty of fit, and
  - 2) The slope is less than the pre-pulse slope.

- Grey-body thermal radiation:  
 $\varepsilon$  : Total Hemispheric Emissivity
- Important for Space-Flight Radiators to have  $\varepsilon \approx 1$ 
  - Most Space-Flight Black paints:  $\varepsilon$  drops for  $T < \sim 100$  K
  - e.g. : Ball InfraRed Black (BIRB): high  $\varepsilon$  at lower temperatures
- Previous  $\varepsilon$  measurement at low temperatures:
  - Tried to simulate space: large chamber;  $T_{\text{WALLS}} \ll T_{\text{SAMPLE}}$
  - Difficult and expensive
- James Webb Space Telescope uses latest formulation of BIRB
  - It uses radiators at  $\sim 35$  Kelvin
  - Our goal: Precise and Inexpensive  $\varepsilon$  Measurement





Radiation not absorbed makes multiple bounces.

Can show that:

$$\dot{Q} = \frac{\sigma A (T_1^4 - T_2^4)}{\frac{1}{\varepsilon_1} + \frac{1}{\varepsilon_2} - 1}$$

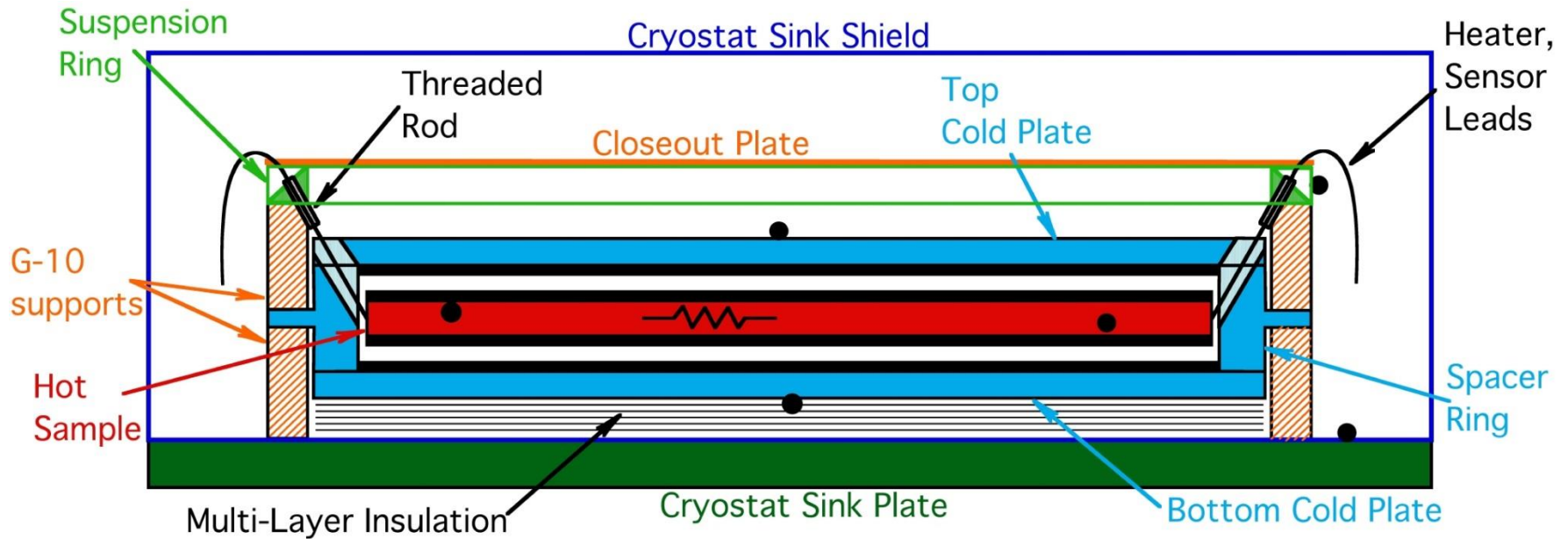
For small  $\Delta T$ :  $(T_1^4 - T_2^4) \approx 4 \cdot T_{avg}^3 \cdot \Delta T$

(For  $\Delta T < (0.06)T_{avg}$ , this is true to within 0.1%)

For  $\varepsilon_1 = \varepsilon_2 = \varepsilon$ :

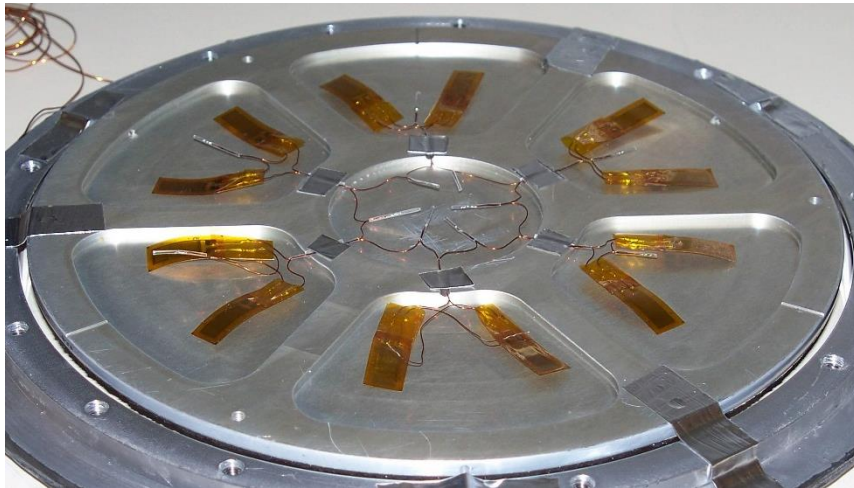
$$\varepsilon = \frac{2}{4\sigma A T_{avg}^3 \left( \frac{d\Delta T}{d\dot{Q}} \right) + 1}$$

So, measure multiple  $\Delta T$  vs.  $\dot{Q}$ , then fit  $d(\Delta T)/d\dot{Q}$   
This is just our standard thermal conductivity technique



- Hot BIRB-coated disk inside cold BIRB-lined “can”;
- Sample (disk) suspended by its thermometer, heater leads
- Control:  $T_{\text{sample}} = T_{\text{suspension}} = T_{\text{hot}}$
- $T_{\text{can}} = T_{\text{cold}}$
- Keep  $\Delta T$  small
- Measure  $\Delta T$  vs control power for constant  $T_{\text{avg}}$

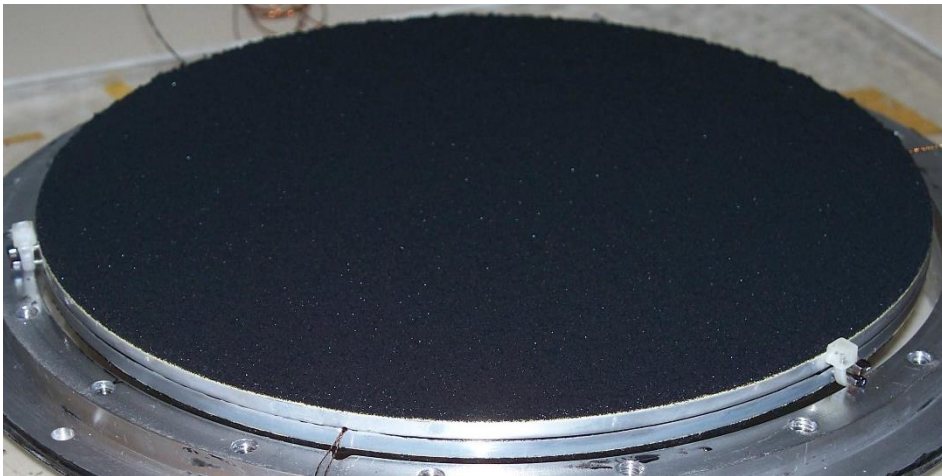
# Emissivity - setup



Heater elements positioned on sample disk



Sample heater elements epoxied/taped



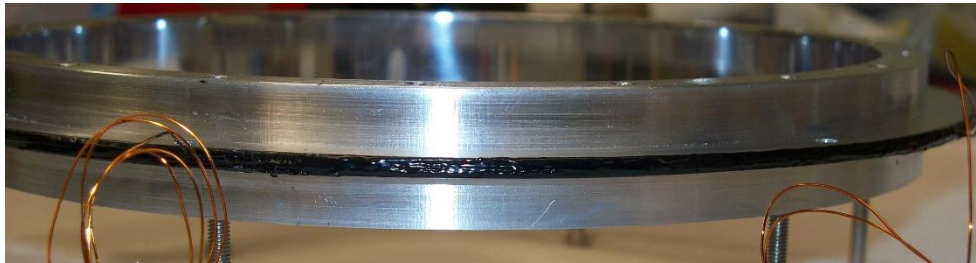
Two sample halves epoxied together



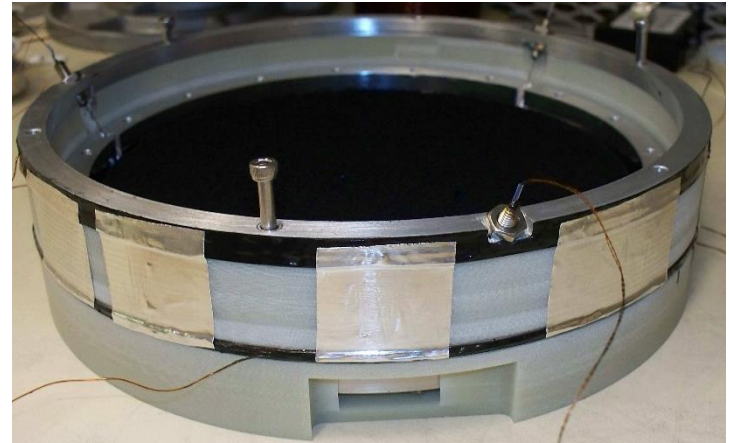
Suspension leads



# Emissivity- setup



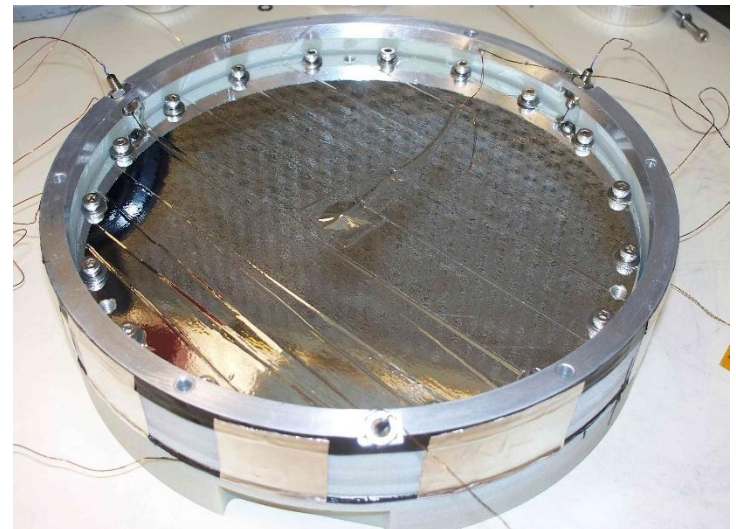
**Wire heater on spacer ring**



**Sample hanging from suspension ring**



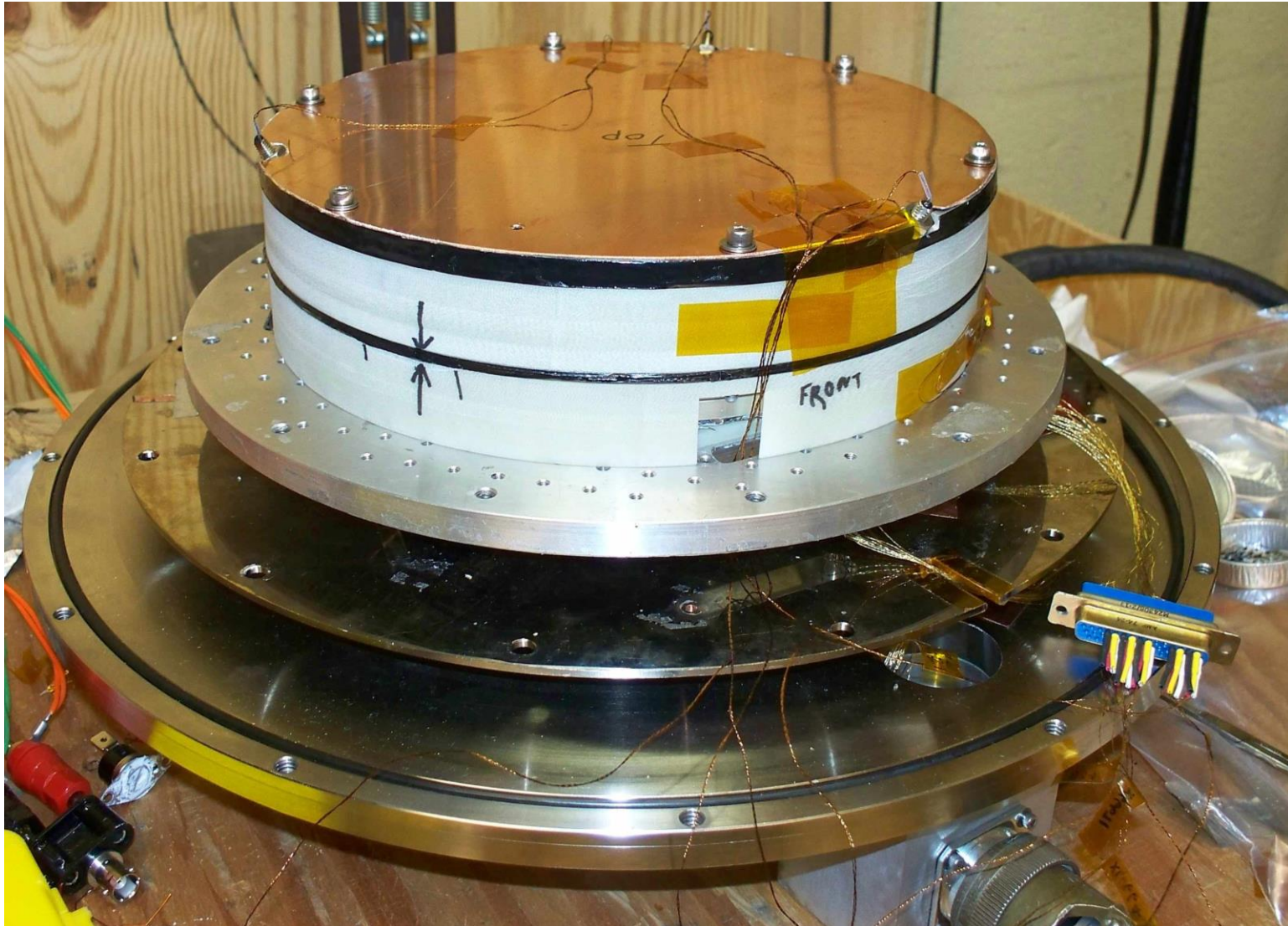
**Bottom cold plate and spacer ring**



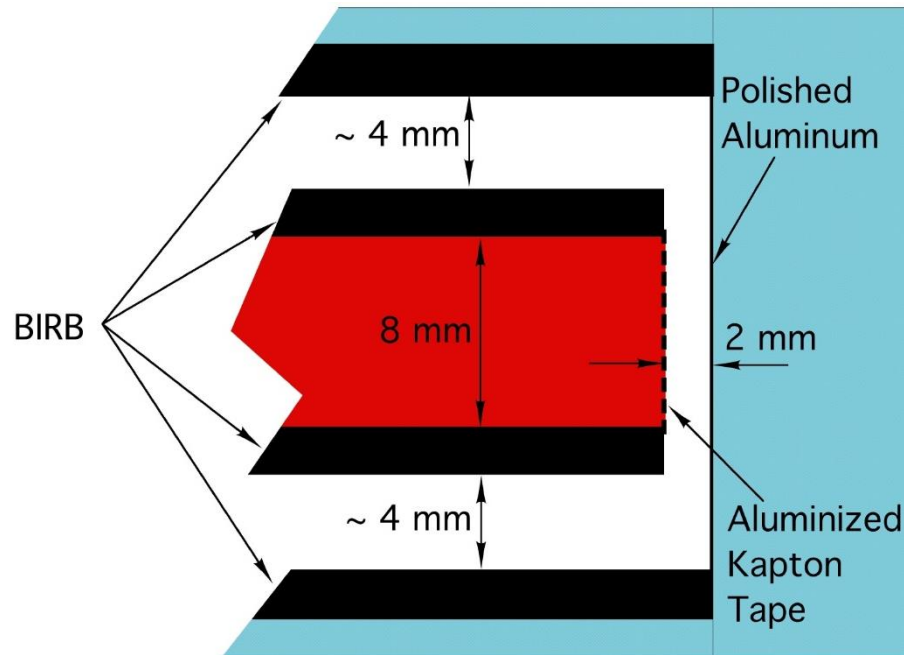
**Top cold plate has been installed**



# Emissivity - setup







- Edge effect makes our setup different from “infinite planes”
- Thermal Desktop model shows our raw  $\epsilon$  overpredicted by 0.85%
  - A correction was applied to our  $\epsilon$  data

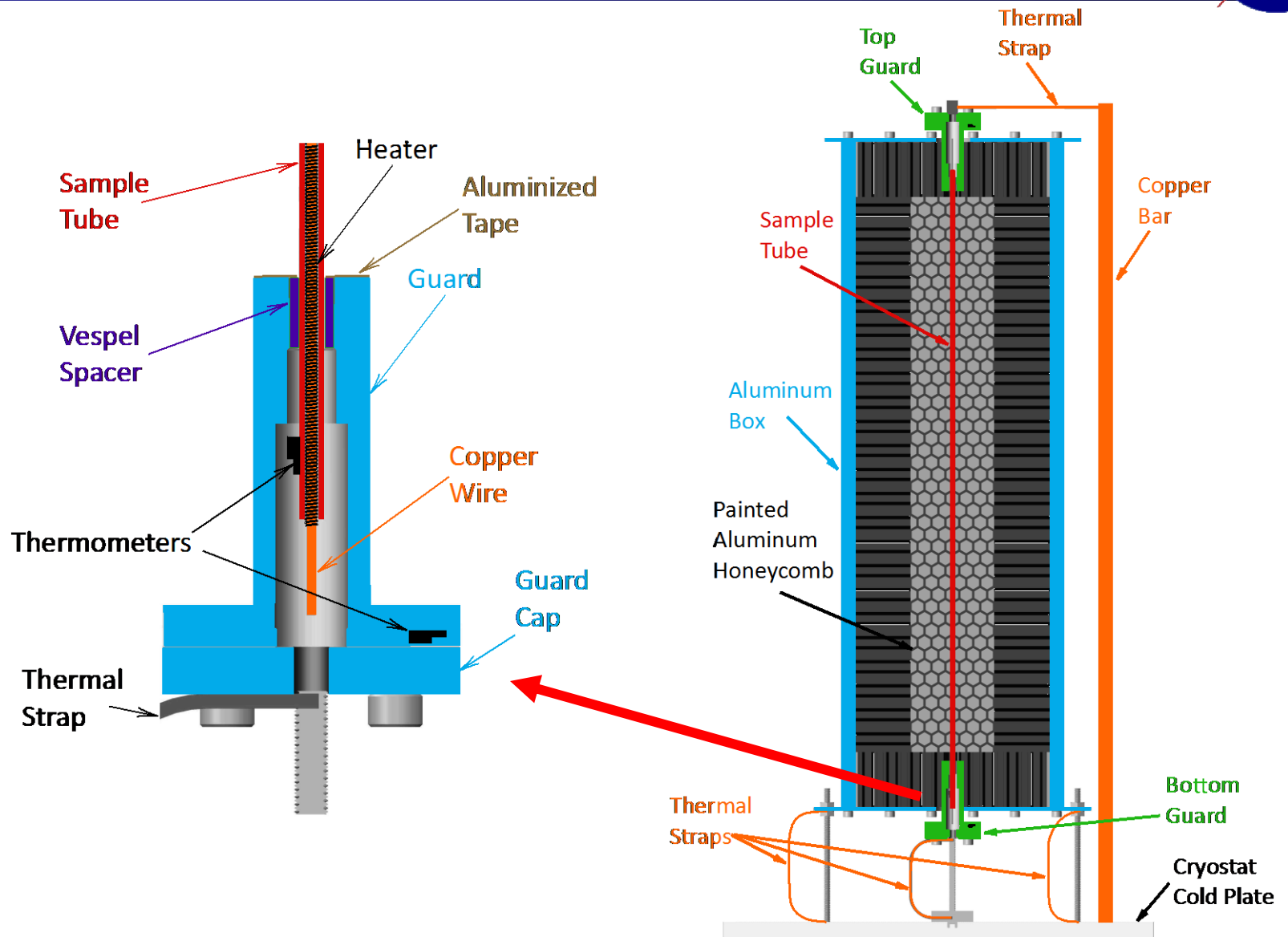


# Emissivity - Absorptivity



- Mid Infrared Instrument (MIRI) on James Webb Space Telescope (JWST)
    - Gas will flow through several meters of 2 mm O. D. stainless steel tubing
      - JWST finite element thermal model predicts spacecraft temperatures
      - Environment surrounding tubing will be as warm as 120 + Kelvin
      - Tubing is gold-plated to keep its thermal absorptance low
  - Predicted JT performance depends strongly on tubing absorptance, and emittance
    - JWST had strong desire to measure  $\alpha$ , and  $\epsilon$  directly
  - Our approach to the measurement:
    - Suspend tubing sample inside a blackbody cavity
- For absorptance:**
- Control tubing temperature at 18 Kelvin
  - Vary cavity temperature and measure power absorbed by tubing
- For emittance:**
- Control cavity temperature at low temperature and measure power in tubing
  - Vary tubing temperature

# Emissivity - Absorptivity





# Conclusions



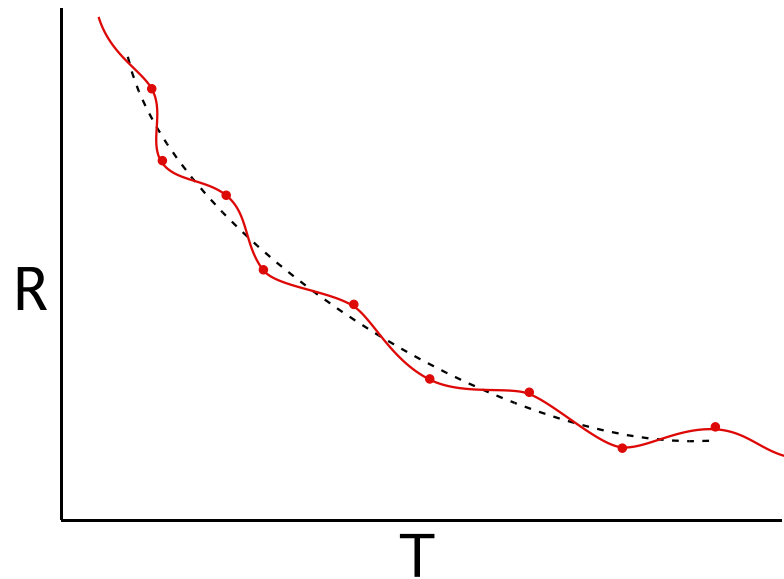
- In most cases it's not too difficult to perform high-precision thermophysical properties measurements between 4 K and room temperature
- We are in a unique position given our expertise and experience with such measurements and have developed appropriate facilities and apparatus to conduct these high precision measurements.
- We are able to measure thermal conductivity, specific heat capacity, radiation properties (i.e. emittance , absorptance), and CTE measurements is currently under development for future use.
- NASA/GSFC's cryogenics group is equipped to perform such measurements for customers at any NASA center

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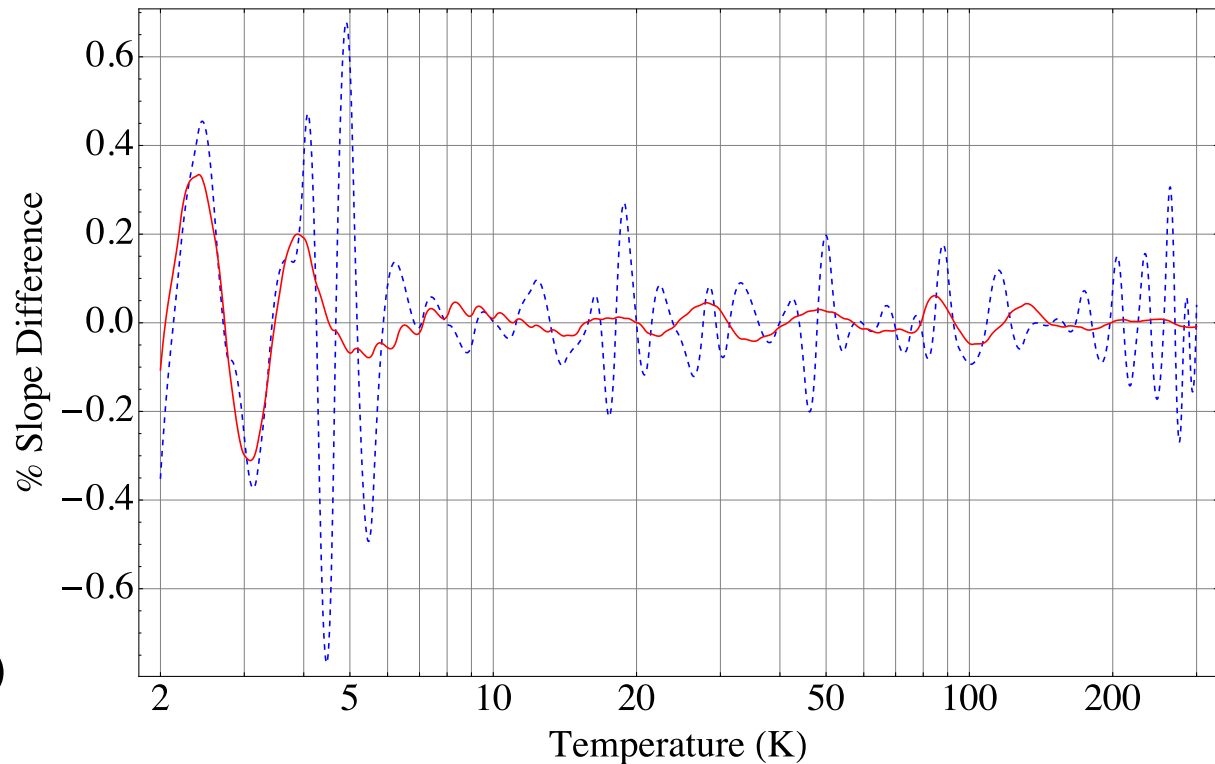
# Reference slides





- 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  $\kappa$

- 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





# Thermal conductivity



- 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  $\frac{d\dot{Q}}{d\Delta T}$