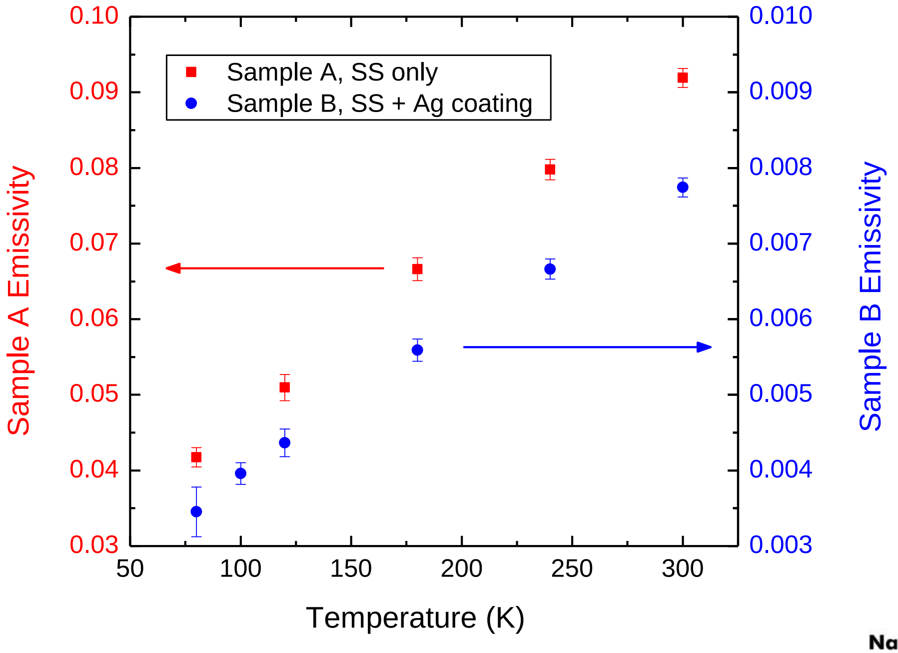
Cryogenic Emissivity Calibration of Highly Reflective Materials

Solomon I. Woods[a], Timothy M. Jung[b], Greg T. Ly[b] and Jie Yu[c]

[a]National Institute of Standards and Technology, 100 Bureau Drive, Gaithersburg, MD, USA 20899;
[b]Jung Research and Development Corp., 1706 U St. NW #204, Washington, DC, USA 20009;
[c]ITER Organization, Route de Vinon-sur-Verdon; 13115, St. Paul-lez-Durance, France



National Institute of Standards and Technology
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Talk Outline

- Background on cryogenic emissivity calibration for ITER
- Experimental setup and method
- Analysis to extract emissivity and uncertainty
- Emissivity of stainless steel and silver from 80 K to 300 K
- Emissivity of multiple sample regions determined in one cooldown
- Summary and possible extensions to the measurement technique

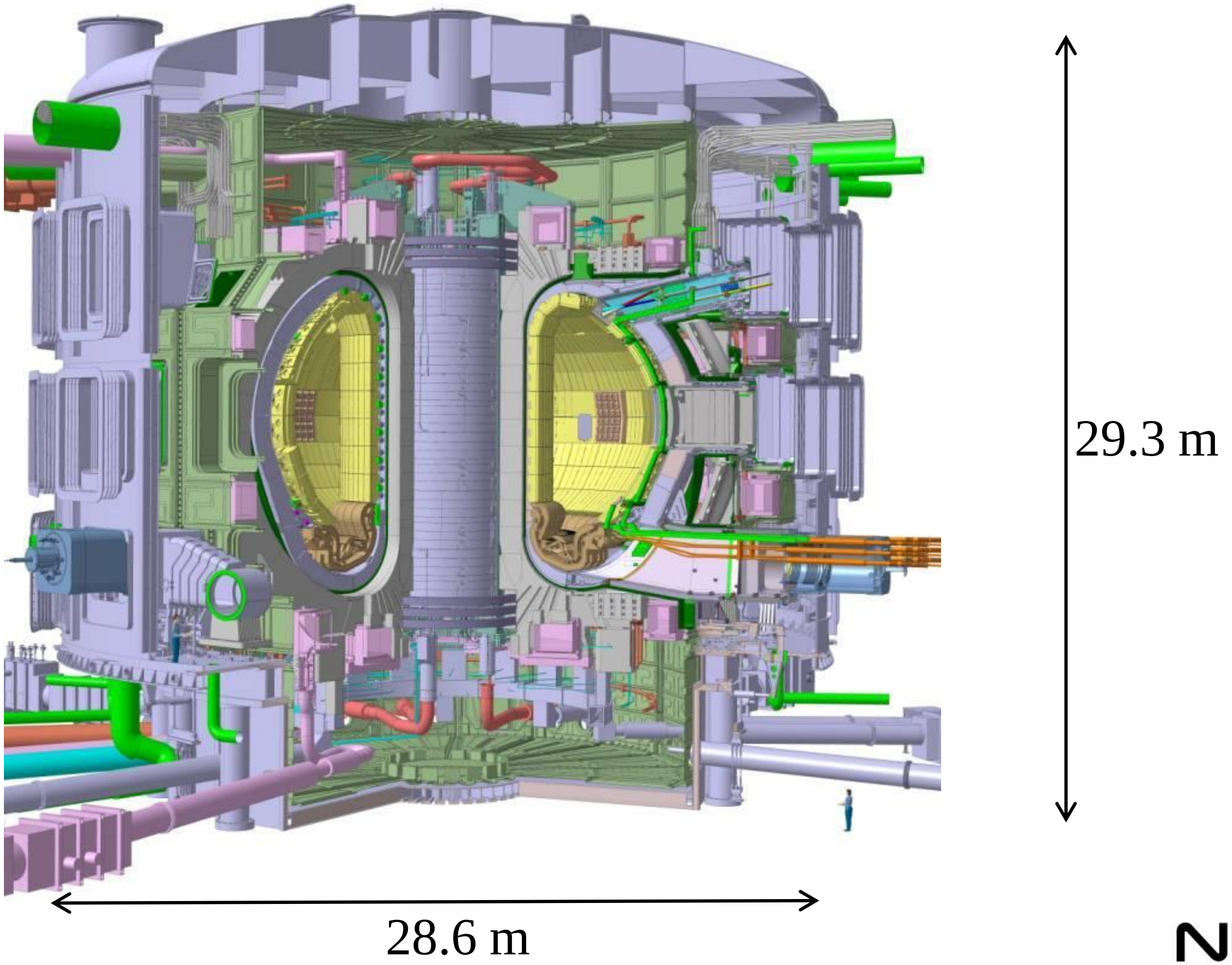
Background on Cryogenic Emissivity Calibration

- Cryogenic emissivity measurement of low emissivity materials is a challenge because signals produced are small, and the emissivity of background elements can be much higher
- Few measurements have been made of the emissivity of stainless steel and silver at cryogenic temperatures, and even fewer papers provide data at a number of different temperatures
- The 80 K thermal shields of ITER will protect the superconducting magnets (at 4.5 K) from warmer reactor regions, and they are silver-coated to minimize emissivity
- Emissivity calibrations are also critical for quantifying the low temperature emittance of coatings and components for space satellite missions, which can be essential for thermal design of these systems

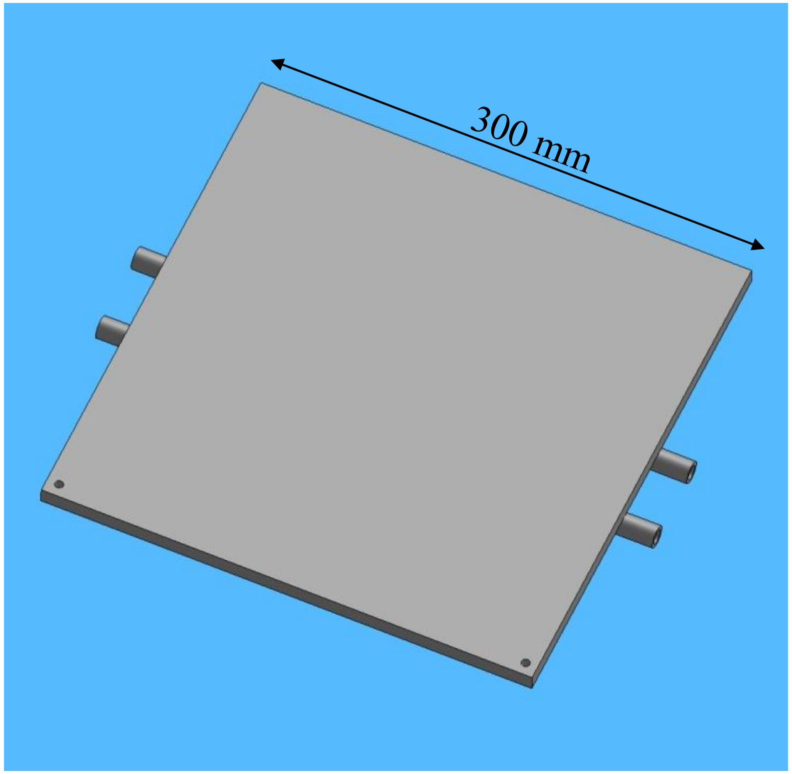
Emissivity of Thermal Shields for ITER

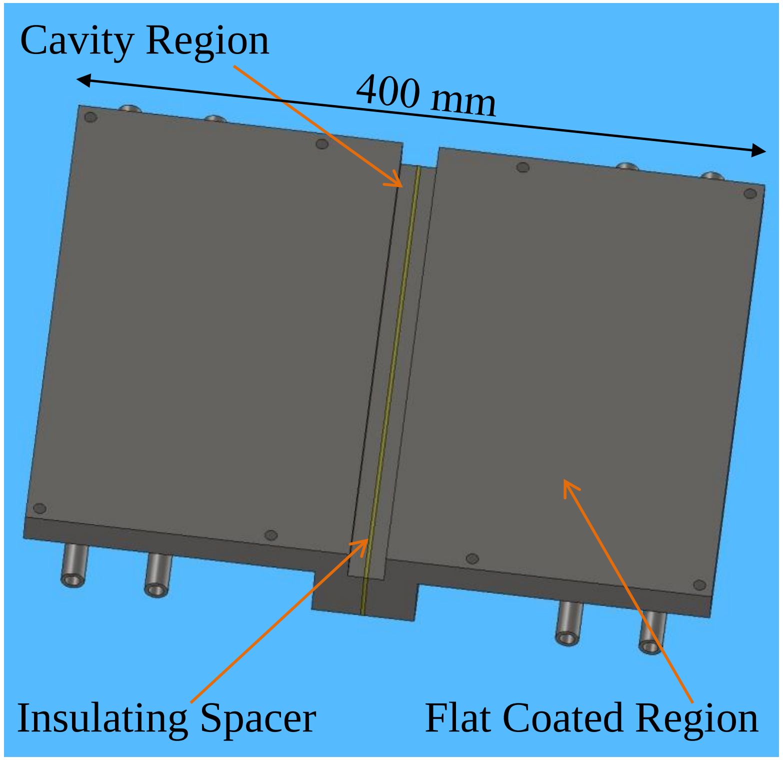
International Thermonuclear Experimental Reactor

> “The ITER cryogenic system will be the largest concentrated cryogenic system in the world…”
>
> -ITER website



Two Types of Thermal Shield Mock-up Samples

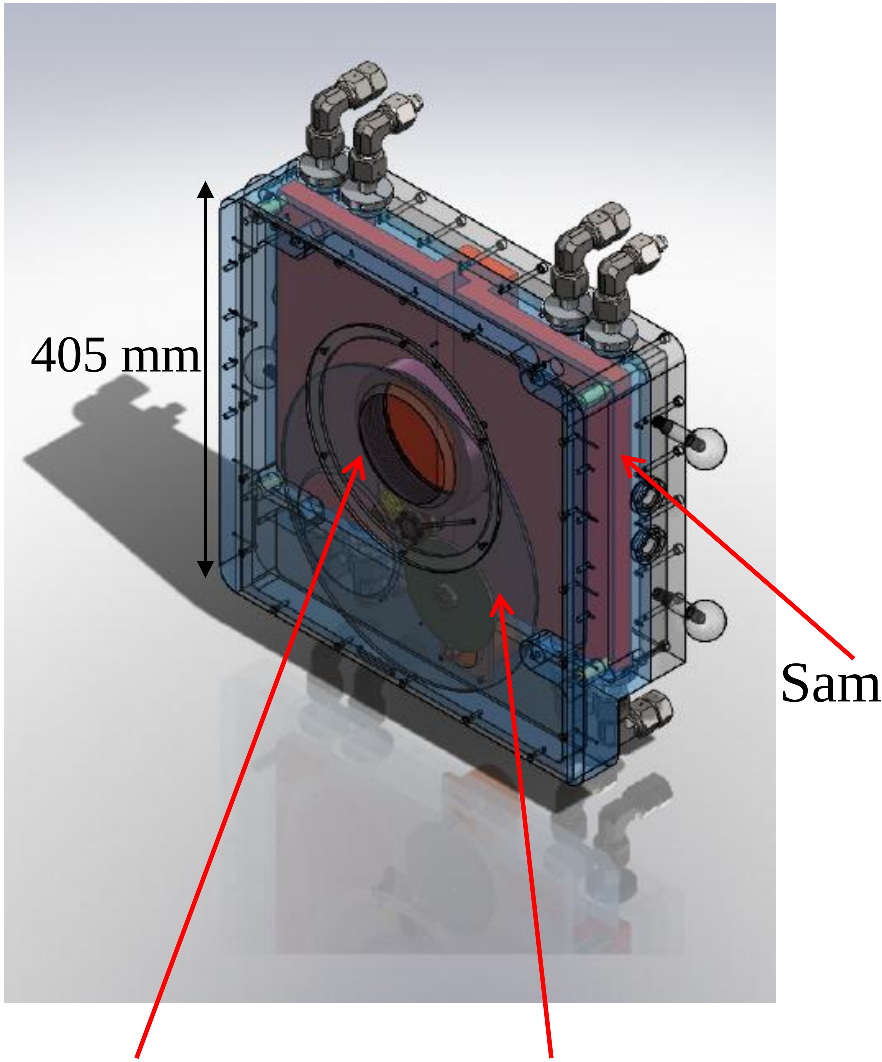




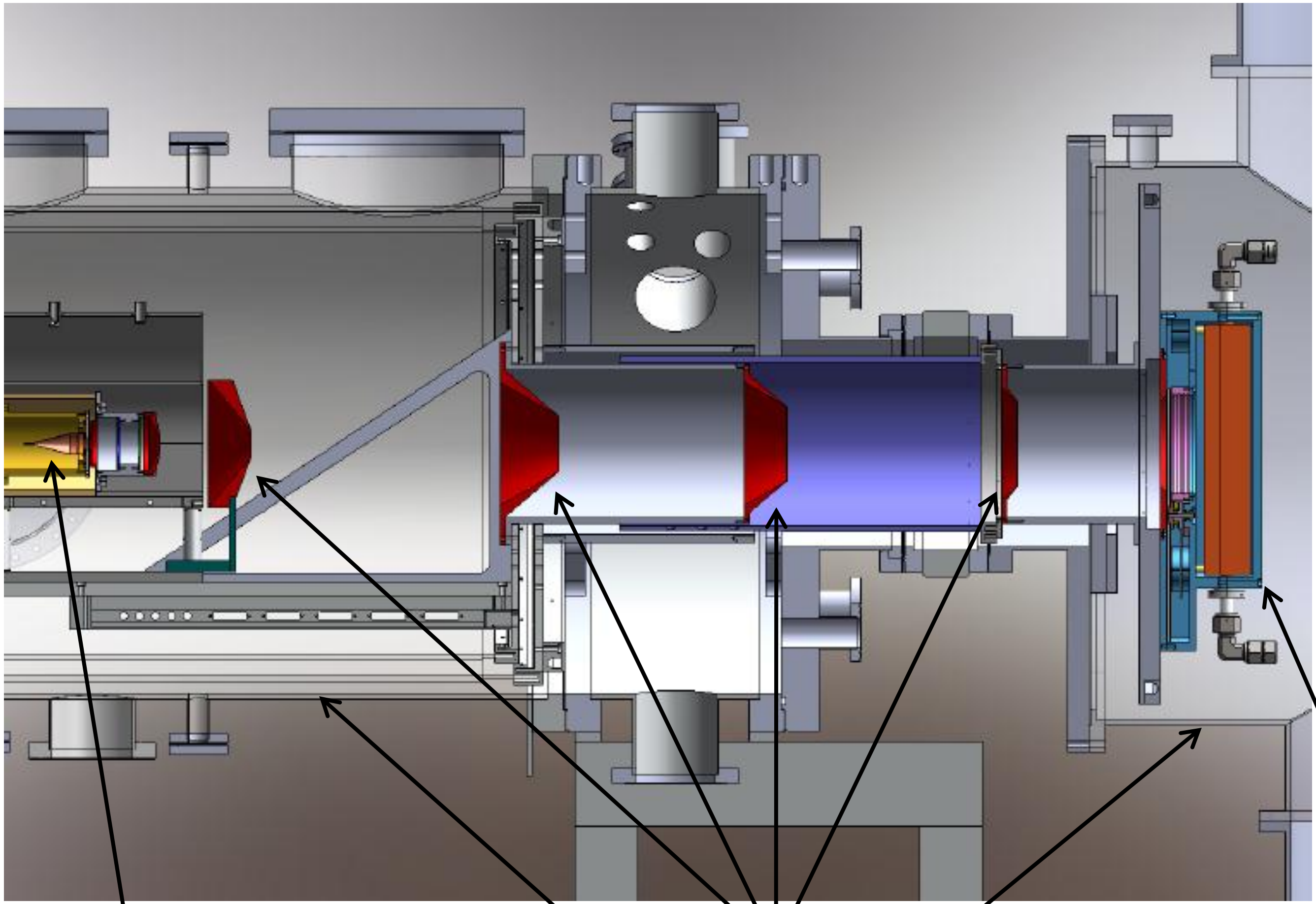
Sample A: Polished 304L Stainless Steel only
Sample B: Additional silver coating, > 5 µm thick

Sample C: Assembly with three regions: insulating spacer (G10), cavity region coated with silver, flat region coated with silver

Smaller Mock-up Sample in Enclosure



ITER Thermal Shield Experimental Layout



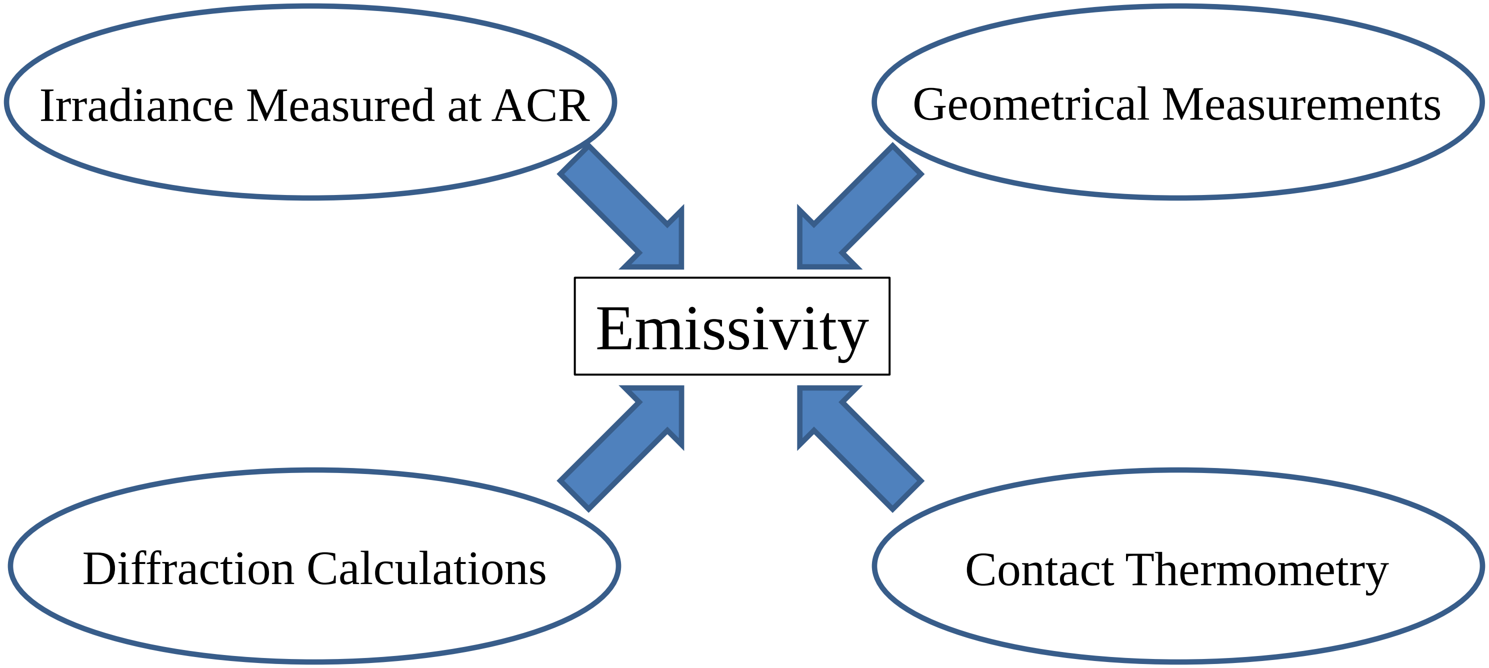
ACR detector

Baffles

Mock-up sample enclosure

Cryogenic vacuum chambers

Emissivity Measurement Technique I



Irradiance Measured at ACR

Geometrical Measurements

Emissivity

Diffraction Calculations

Contact Thermometry

Emissivity Measurement Technique II

Measurement provides low uncertainty by using a double background-subtraction technique and by quantifying signals from shutter and background

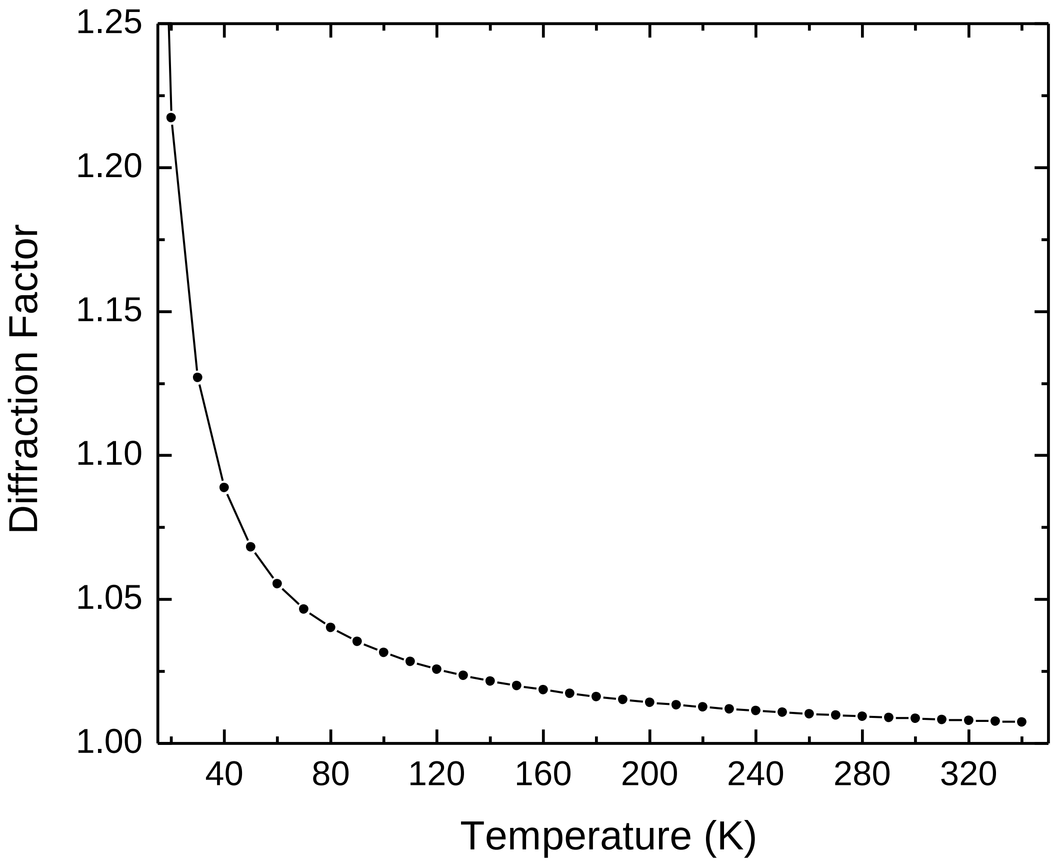
Background subtraction:

- Every ACR measurement was made with shutter open and shutter closed
- Background measurement with sample at 20 K was made to quantify "offset signal" of the system

Signals from shutter and background:

- Quantified the emitted signal from background and the emitted and reflected signal from shutter
- Used historical data to quantify the emissivities of background (Z306) and shutter
- Used contact thermometry to quantify temperatures of background and shutter

Emissivity Measurement Technique III



* Diffraction factor is about 4 % at 80 K and 0.86 % at 300 K, for our measurement geometry

Analysis to Extract Emissivity

emissivity at setpoint

ACR power at setpoint

ACR power at 20 K

$$\varepsilon_{s}\big|_{T_s=T_{set}} = \left(\frac{(S_s - S_{sh})^{(meas)}}{\mathscr{D}_s}\Bigg|_{T_s=T_{set}} - \frac{(S_s - S_{sh})^{(meas)}}{\mathscr{D}_{sh}}\Bigg|_{T_s=T_{base}} \right) \Big/ (\Gamma T_s^4)\big|_{T_s=T_{set}} +$$

$$\left(\left[\varepsilon_{sh} T_{sh}^4 \frac{\mathscr{D}_{sh}}{\mathscr{D}_s} - (\varepsilon_{sh} - \varepsilon_s^{(0)})\varepsilon_b T_b^4 \frac{\mathscr{D}_b}{\mathscr{D}_s} \right]_{T_s=T_{set}} + \left[\varepsilon_s^{(0)} T_s^4 \frac{\mathscr{D}_s}{\mathscr{D}_{sh}} - \varepsilon_{sh} T_{sh}^4 + (\varepsilon_{sh} - \varepsilon_s^{(0)})\varepsilon_b T_b^4 \frac{\mathscr{D}_b}{\mathscr{D}_{sh}} \right]_{T_s=T_{base}} \right) \Big/ (T_s^4)\big|_{T_s=T_{set}}$$

shutter & background contribution at setpoint

shutter & background contribution at 20 K

ε_α = emissivity of element α; s = sample, sh = shutter, b = background
T_α = temperature of element α; s = sample, sh = shutter, b = background
$\mathscr{D}_\alpha$ = diffraction factor associated with element α; s = sample, sh = shutter, b = background
Γ = configuration factor

** Woods SI, Jung TM, Ly GT and Yu J. Broadband Emissivity Calibration of Highly Reflective Samples at Cryogenic Temperatures. *Metrologia* 2012; **49**:737-44

Analysis to Calculate Uncertainties

Propagate uncertainties of all parameters through equation for emissivity, according to:

$$u_c^2 = \sum_{i=1}^{n} \left(\frac{\partial \varepsilon}{\partial x_i} \right)^2 u^2(x_i) + 2 \sum_{i=1}^{N-1} \sum_{j=i+1}^{N} \frac{\partial \varepsilon}{\partial x_i} \frac{\partial \varepsilon}{\partial x_j} u(x_i, x_j)$$

total variance

variance of parameter x_i

covariance of parameters x_i and x_j

…where ε is a function of numerous measurable parameters, as defined on the previous slide

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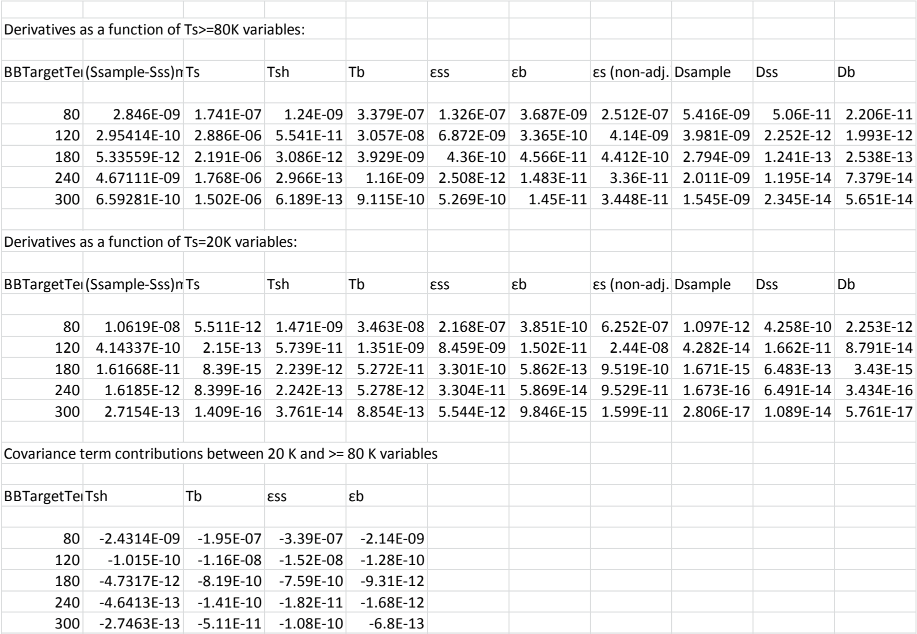


Derivatives as a function of Ts>=80K variables:

BBTargetTe	(Ssample-Sss)n	Ts	Tsh	Tb	εss	εb	εs (non-adj.	Dsample	Dss	Db
80	2.846E-09	1.741E-07	1.24E-09	3.379E-07	1.326E-07	3.687E-09	2.512E-07	5.416E-09	5.06E-11	2.206E-11
120	2.95414E-10	2.886E-06	5.541E-11	3.057E-08	6.872E-09	3.365E-10	4.14E-09	3.981E-09	2.252E-12	1.993E-12
180	5.33559E-12	2.191E-06	3.086E-12	3.929E-09	4.36E-10	4.566E-11	4.412E-10	2.794E-09	1.241E-13	2.538E-13
240	4.67111E-09	1.768E-06	2.966E-13	1.16E-09	2.508E-12	1.483E-11	3.36E-11	2.011E-09	1.195E-14	7.379E-14
300	6.59281E-10	1.502E-06	6.189E-13	9.115E-10	5.269E-10	1.45E-11	3.448E-11	1.545E-09	2.345E-14	5.651E-14

Derivatives as a function of Ts=20K variables:

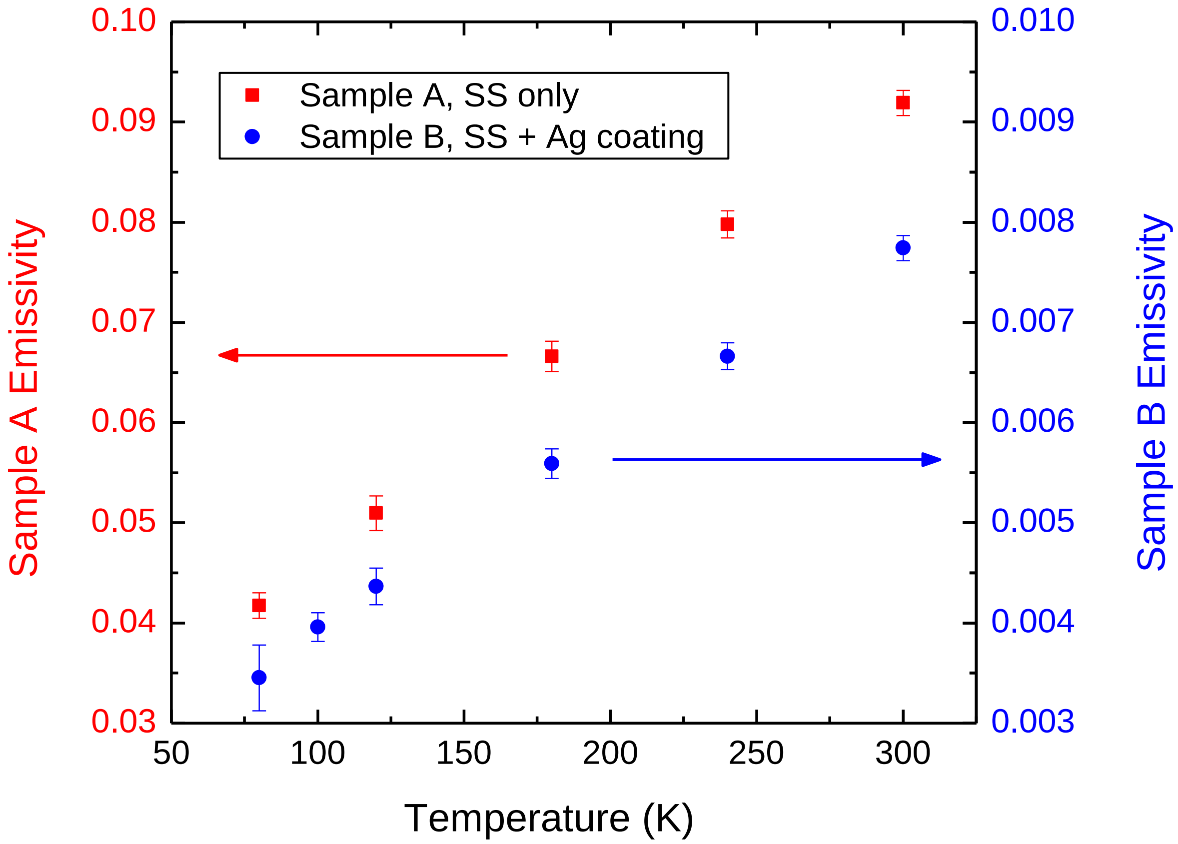
BBTargetTe	(Ssample-Sss)n	Ts	Tsh	Tb	εss	εb	εs (non-adj.	Dsample	Dss	Db
80	1.0619E-08	5.511E-12	1.471E-09	3.463E-08	2.168E-07	3.851E-10	6.252E-07	1.097E-12	4.258E-10	2.253E-12
120	4.14337E-10	2.15E-13	5.739E-11	1.351E-09	8.459E-09	1.502E-11	2.44E-08	4.282E-14	1.662E-11	8.791E-14
180	1.61668E-11	8.39E-15	2.239E-12	5.272E-11	3.301E-10	5.862E-13	9.519E-10	1.671E-15	6.483E-13	3.43E-15
240	1.6185E-12	8.399E-16	2.242E-13	5.278E-12	3.304E-11	5.869E-14	9.529E-11	1.673E-16	6.491E-14	3.434E-16
300	2.7154E-13	1.409E-16	3.761E-14	8.854E-13	5.544E-12	9.846E-15	1.599E-11	2.806E-17	1.089E-14	5.761E-17

Covariance term contributions between 20 K and >= 80 K variables

BBTargetTe	Tsh	Tb	εss	εb
80	-2.4314E-09	-1.95E-07	-3.39E-07	-2.14E-09
120	-1.015E-10	-1.16E-08	-1.52E-08	-1.28E-10
180	-4.7317E-12	-8.19E-10	-7.59E-10	-9.31E-12
240	-4.6413E-13	-1.41E-10	-1.82E-11	-1.68E-12
300	-2.7463E-13	-5.11E-11	-1.08E-10	-6.8E-13

example of uncertainty spreadsheet with contributions from measurement parameters

Emissivity of Stainless Steel and Silver



* Silver-coating lowers the emittance of the shield by more than a factor of 10

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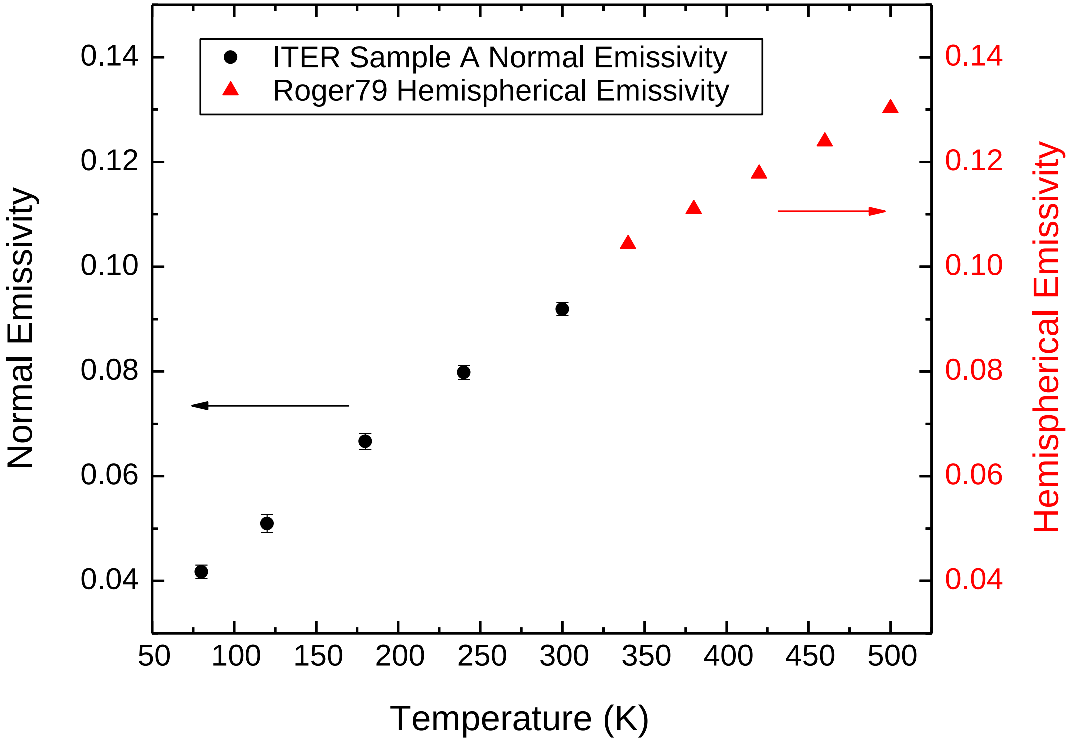
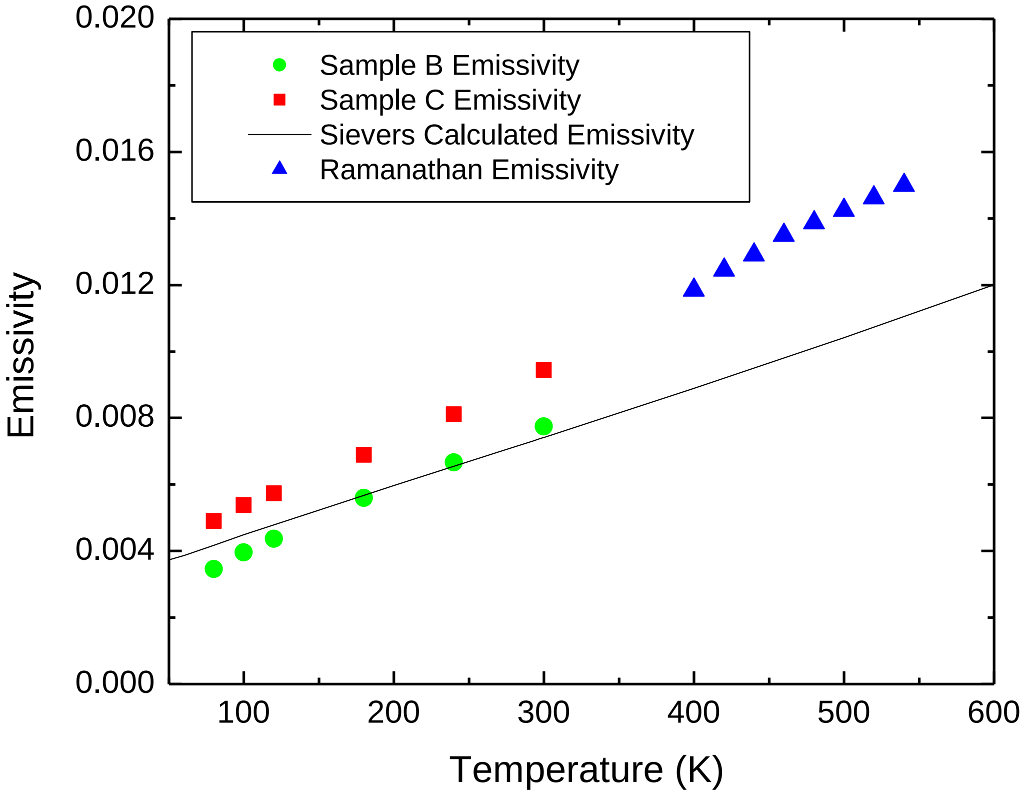
Comparison of SS304L Data with Previous Measurements



* There is no simple relationship between hemispherical and normal emissivity for a “poor” conductor such as stainless steel, but they are nearly equal for metals which exhibit dc resistivities similar to stainless steel.

- Sievers A J 1978 Thermal radiation from metal surfaces *J. Opt. Soc. Am.* 68 (11) 1505-16
- Roger C R, Yen S H and Ramanathan K G 1979 Temperature variation of total hemispherical emissivity of stainless steel AISI 304 *J. Opt. Soc. Am.* **69** (10) 1384-90

Comparison of Silver Data with Previous Measurements



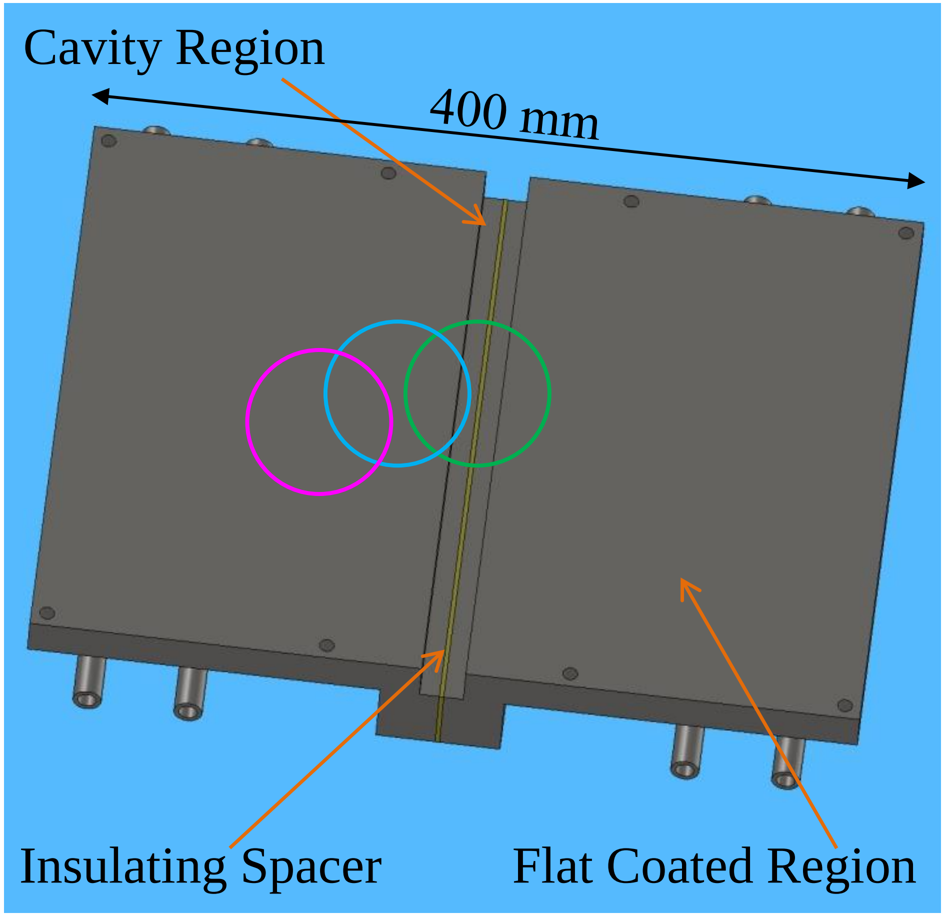
- Sievers AJ. 1978 Thermal radiation from metal surfaces. *J. Opt. Soc. Am.* 1978; **68**(11):1505-16.
- Ramanathan KG and Yen SH. High-temperature emissivities of copper, aluminum and silver. *J. Opt. Soc. Am.* 1977; **67**(1):32-8.

Emissivity of Multiple Sample Regions in One Cooldown

Take data with shutter in three different positions on the sample plate:

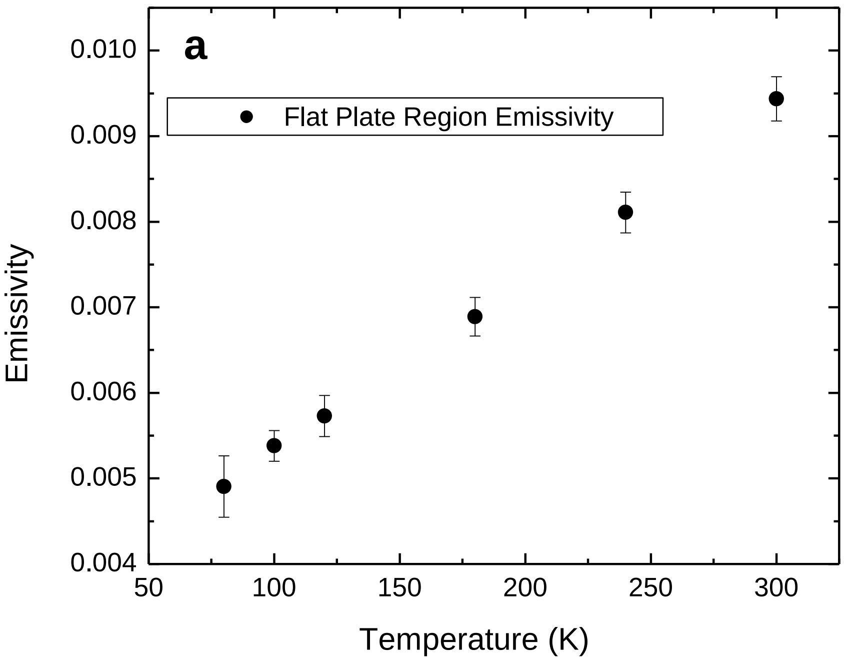
green circle: includes insulating spacer, cavity and flat region
blue circle: includes cavity and flat region
magenta circle: includes only flat region

Use geometrical data and appropriate configuration factors to extract emissivity contribution of each separate region (insulating spacer, cavity, flat region)

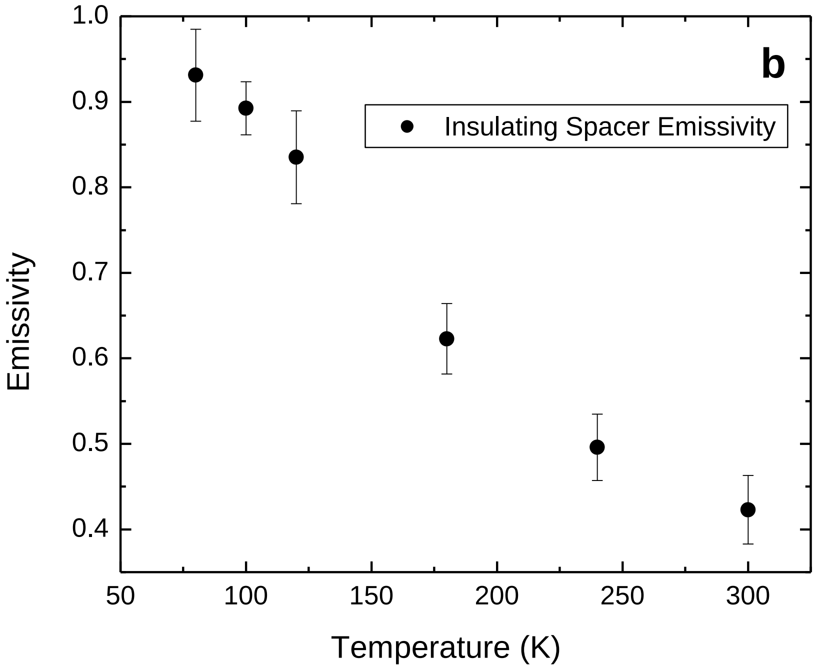


Emissivity of Silver-Coating and G10

“Repaired” Silver-Coating

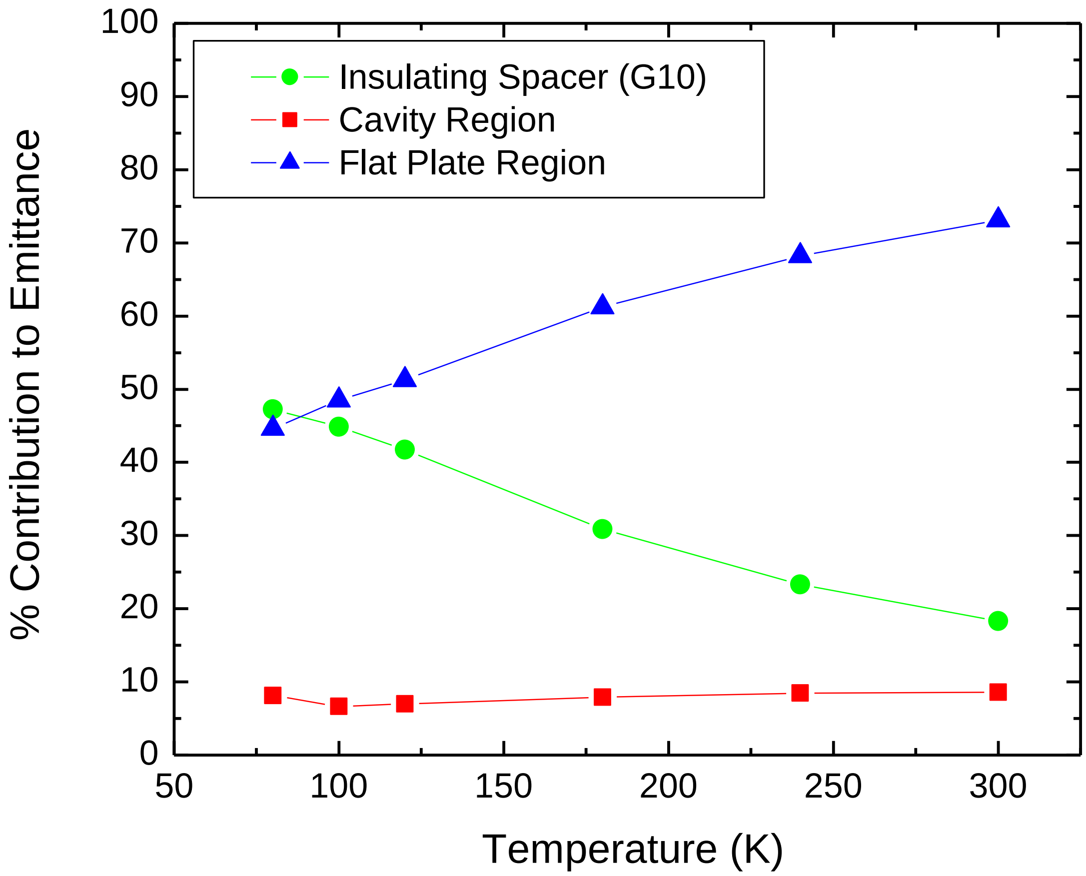


G-10 Spacer



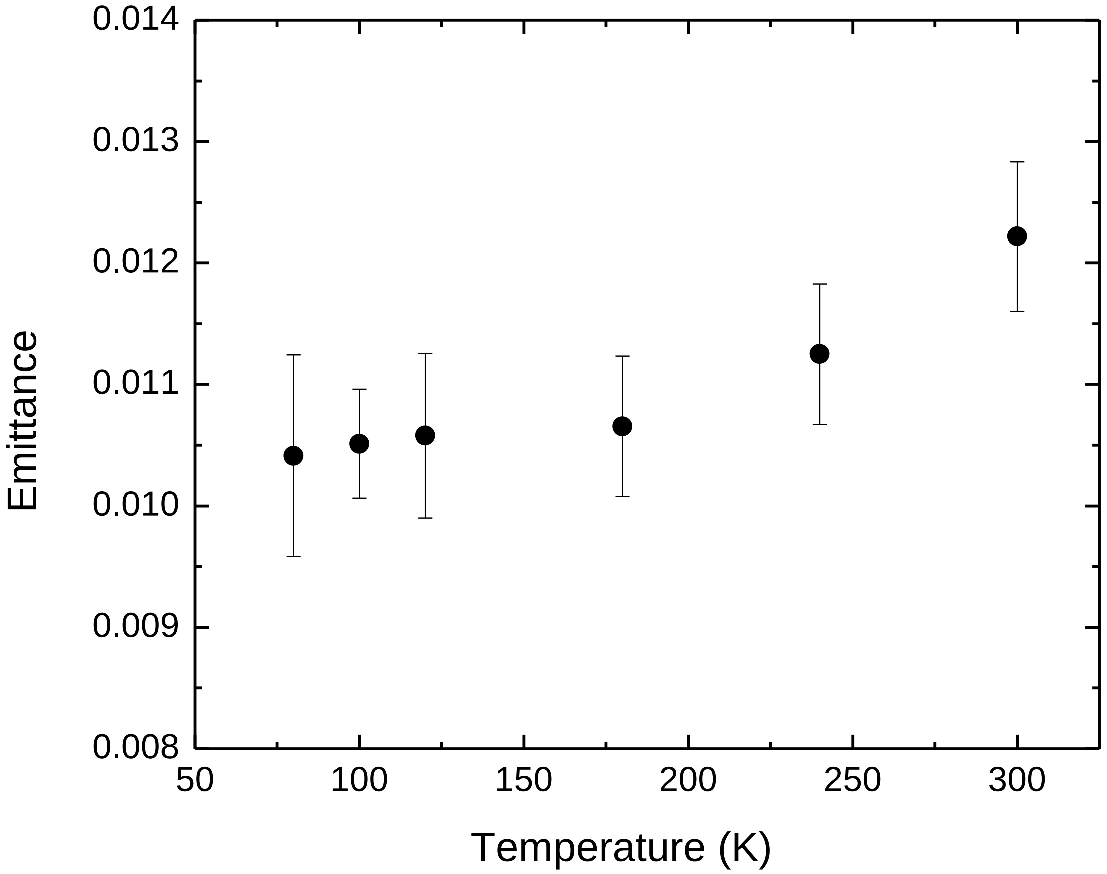
* These emissivities were extracted from the data taken on the Sample C assembly

Relative Emittance of Thermal Shield Assembly Regions



* The G10 insulating spacer accounts for nearly half the emittance at 80 K, even though it only covers about 0.5 % of the surface area.

Emittance of the Entire Thermal Shield Assembly



Emittance of the entire Sample C assembly is around 1 % and relatively flat as a function of temperature from 80 K to 300 K.

Conclusions and Further Plans

- We have developed a method to measure the normal emissivity of highly reflective samples from 80 K to 300 K with uncertainty as low as 0.0002 (k=1)

- We can measure the emissivity of different regions of a sample during a single cooldown and calculate the emittance of an assembly from 80 K to 300 K

- Insulators in a cryogenic thermal shield assembly should be surface-metallized or otherwise hidden to minimize emittance

- We could develop a system to rotate samples within our cryogenic chamber in order to measure directional emissivity as a function of angle