

Characterisation of New Planar Cryogenic Radiometric Standards under Development at NIST

Malcolm White

CALCON 2016

National Institute of Standards and Technology

325 Broadway, Boulder, Colorado 80305-3228

[*m.white@boulder.nist.gov*](mailto:m.white@boulder.nist.gov)

August 22 – 25, 2016

Logan, Utah



Physical Measurement Laboratory
Applied Physics Division

Laser Sources and Detectors Group

Leader: John Lehman



Calibration Services:

Matthew Spidell
Josh Hadler
David Livigni
Igor Vayshenker

Radiometry R&D:

Michelle Stephens
Thomas Gerrits
Ivan Ryger
Nathan Tomlin
Malcolm White
Chris Yung
Solomon Woods (G)

High Power Applications:

Paul Williams
Josh Hadler
Brian Simonds

TeraHertz:

Erich Grossman



Development of next generation sources and detectors for laser power and energy measurement traceable to NIST

- Key enabling technologies – CNT's & silicon micro-machining
- Oxygen plasma treatment to reduce CNT reflectance
- Implementation of CNT chip-scale radiometric detectors
 - cryogenic for fibre telecoms and THz wavelengths
 - design, fabrication and characterisation of PBR chips
- Same technology for radiometers for Space (LASP)

➤ NIST has initiated a program called NIST on a Chip.

The goal is to develop compact, fast and easy to use radiometric standards, spanning the wavelength spectrum from the ultraviolet to the THz region in a single detector, with utility beyond that of the laboratory environment, including space based remote sensing applications.

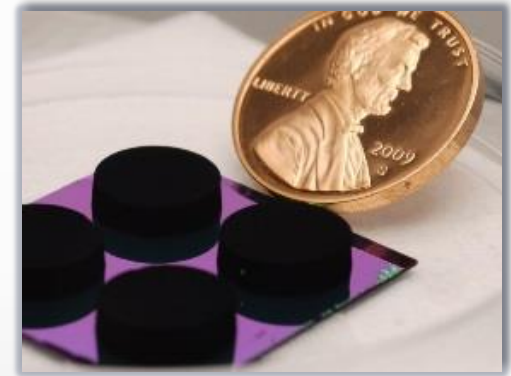
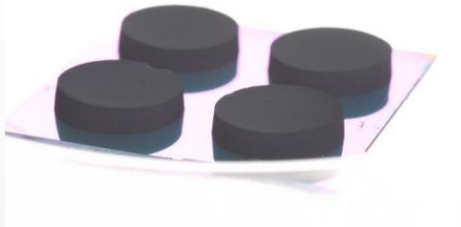
This has been implemented using VACNT arrays, grown on Si substrates, prepared using silicon micro-fabrication techniques, to produce highly efficient, broad spectrum, planar (2D) radiometric detectors.

Key Enabling Technologies - VACNT's

The NIST Sources and Detectors group is developing next generation cryogenic and room temperature laser power standards, based on two key enabling technologies:

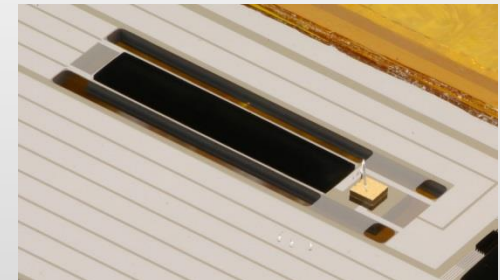
- vertically aligned CNT arrays with near unity absorptivity out to at least 50 μm
- state of the art silicon micro-fabrication techniques

**Vertically aligned arrays
grown by CVD on silicon**

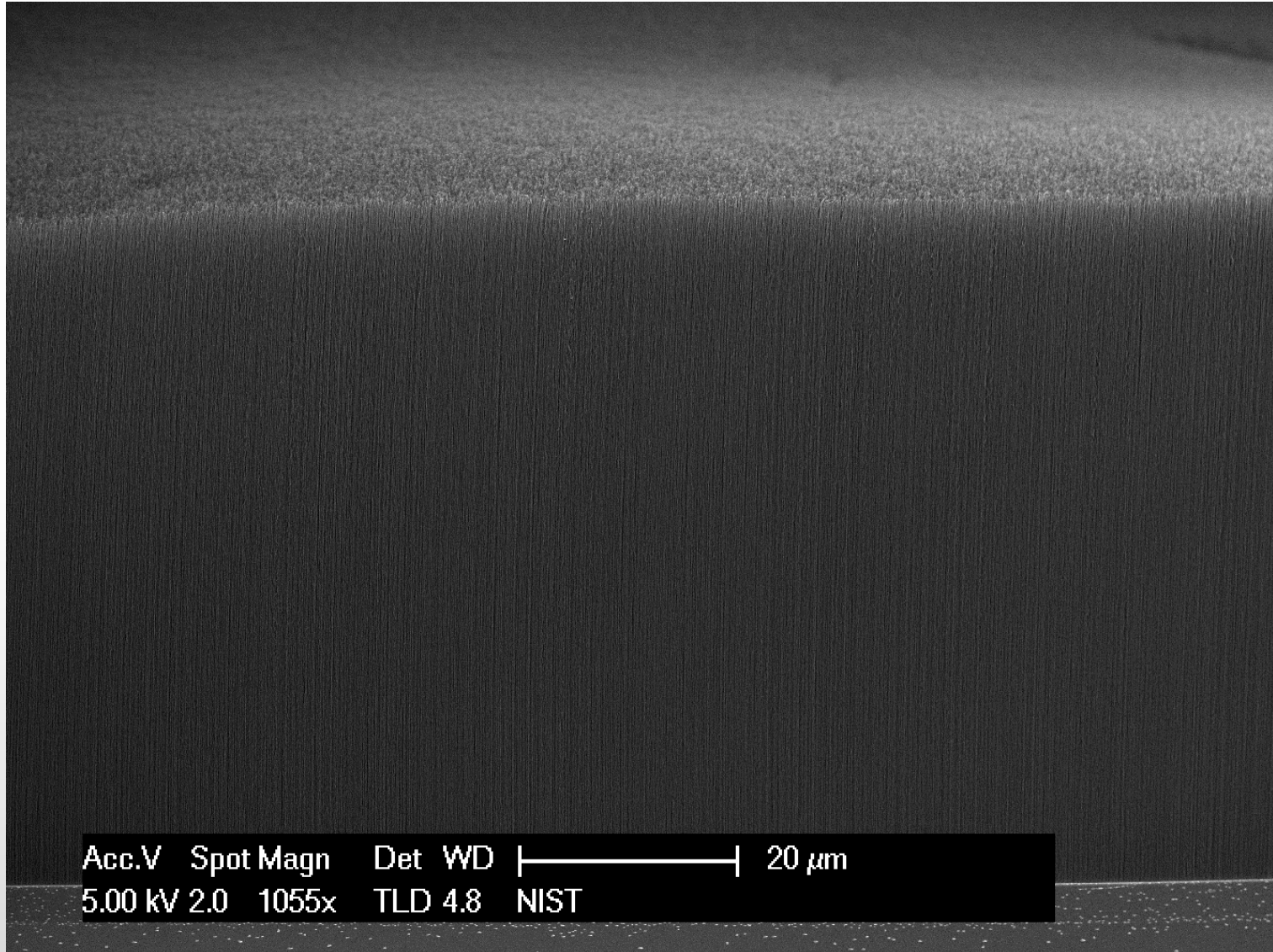


The core technology has been demonstrated by developing:

- cryogenic OFPM's and THz radiometers
- a room temperature radiometer – in process
- carbon nanotube detectors for space applications



Enlarged View of VACNT's



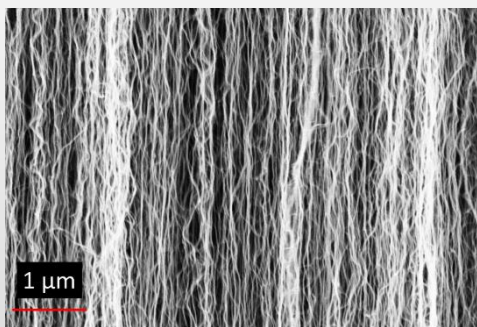
NIST Nanotube Furnace in Cleanroom

NIST has just purchased a cold-wall PE-CVD furnace which will allow us to grow our own nanotubes, giving us complete control over the deposition process and turnaround time.

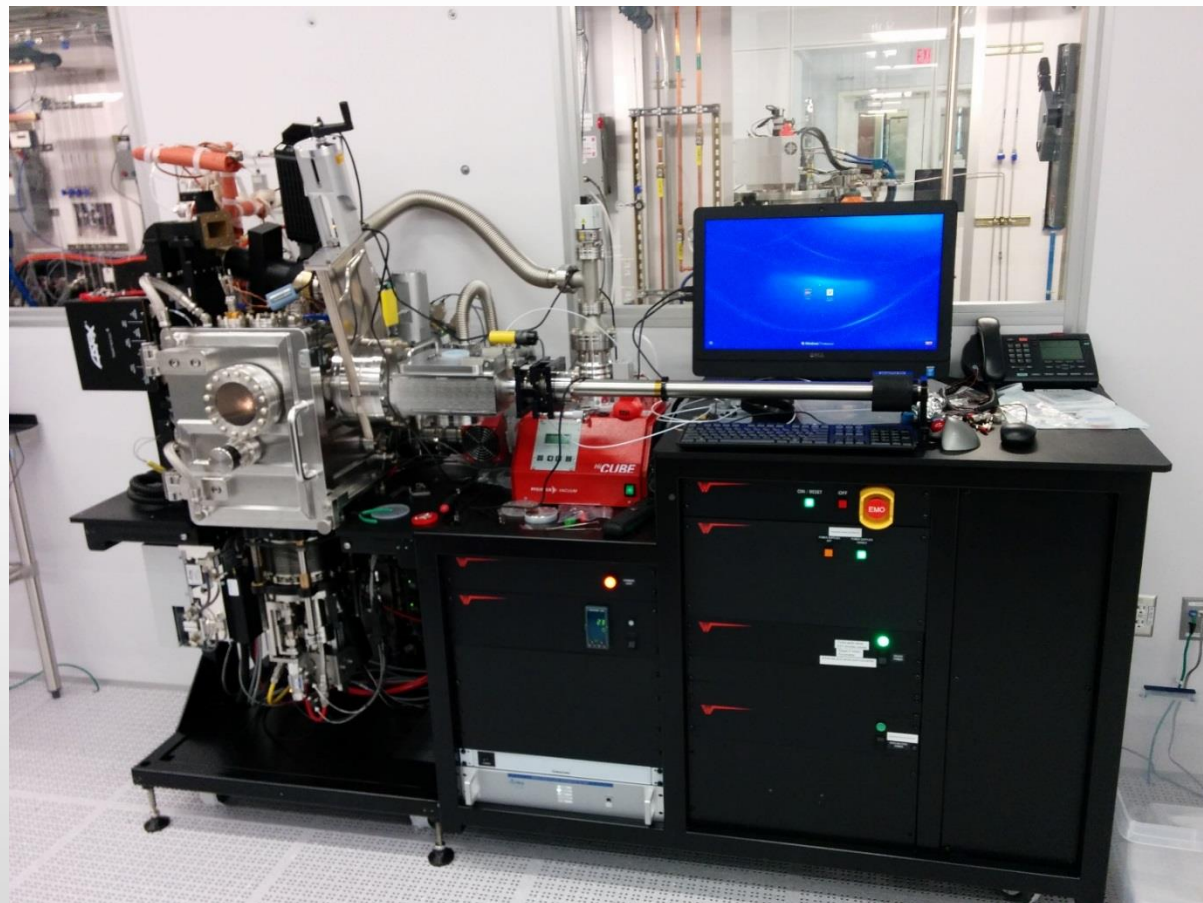
Initial trials have grown tubes 350 μm long



Pyro-electric detector

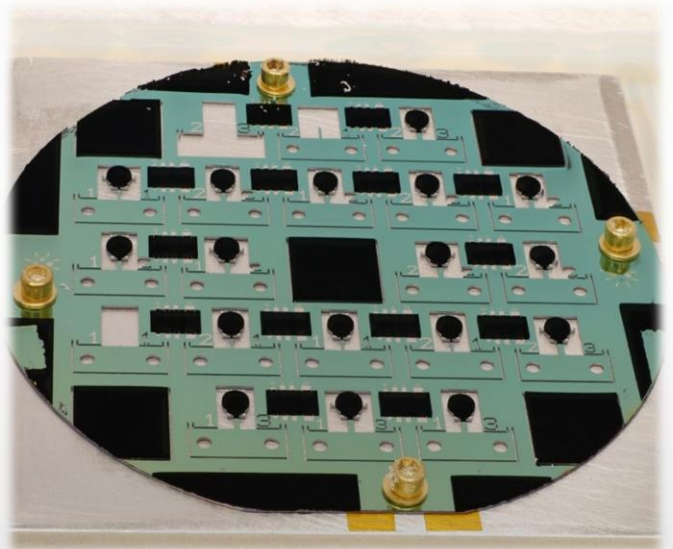
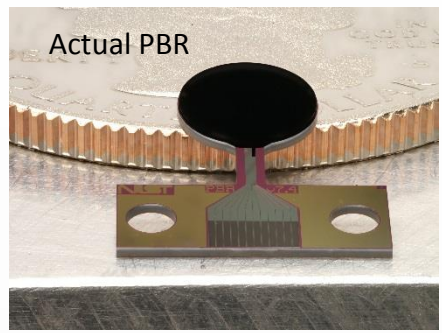


VACNT's



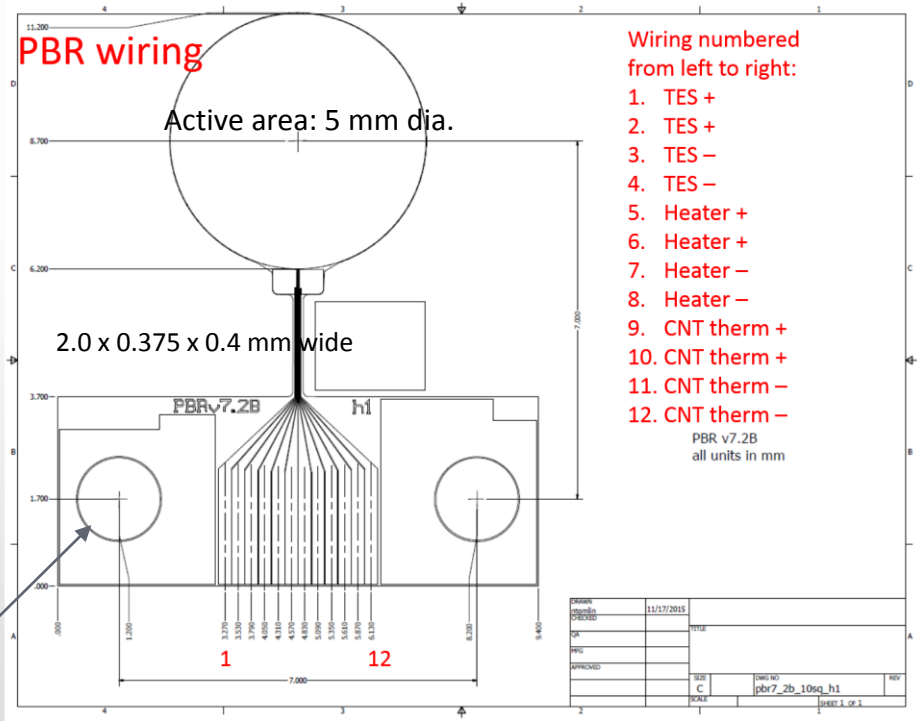
Key Enabling Technologies - Si Micro-machining

- Mo bilayer superconducting TES temp sensor, 6 K (0.9 K)
- Vanadium s/c wiring to mitigate joule heating losses, 13 K (5.4 K)
- Tungsten heater, 1100 Ω
- CNT absorber deposited within tungsten heater



20 detectors and individual witness samples from each 75 mm wafer

7.0 mm hole spacing



Credit: Nathan Tomlin, NIST

N.A. Tomlin, M. White, I. Vayshenker, S.I. Woods, J.H. Lehman, *Metrologia* **52**, 2, 376 – 383 (2015). <http://dx.doi.org/10.1088/0026-1394/52/2/376>

O₂ Plasma Treatment to Decrease Reflection

- We observed a linear decrease in VACNT height with plasma exposure due to crust ablation.
- This resulted in decreased reflection, improved absorption.

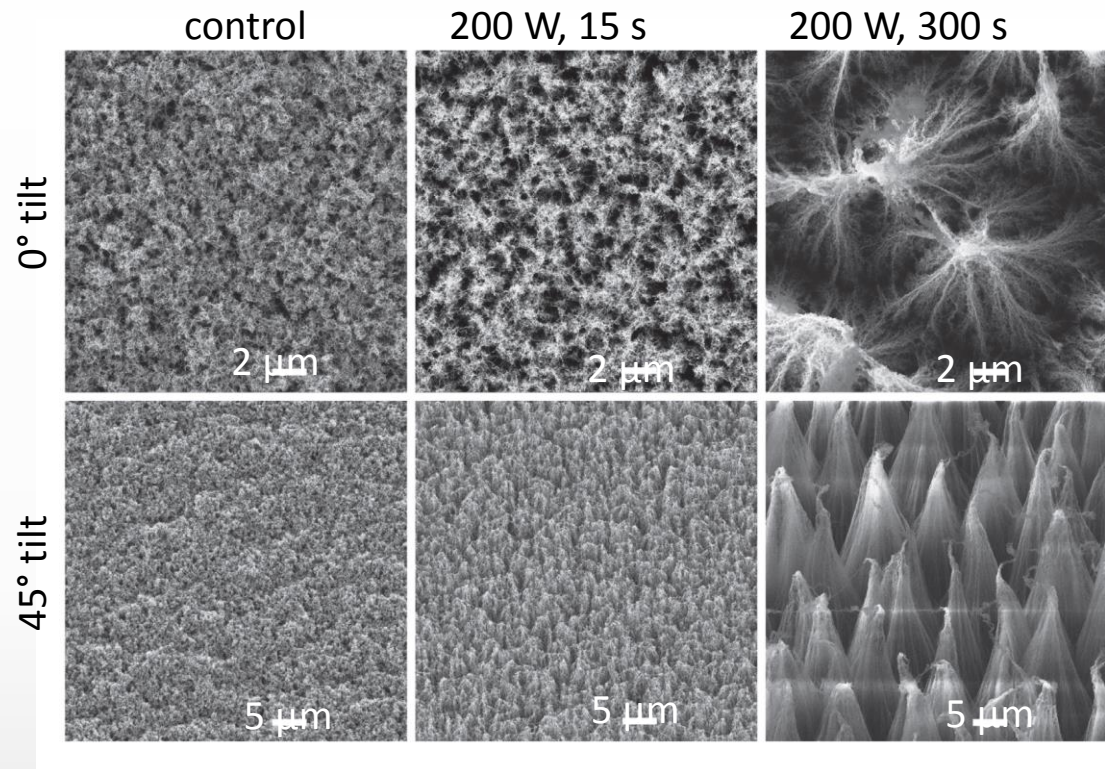
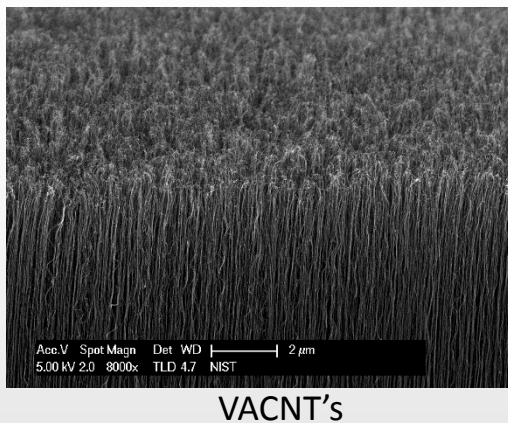


Fig. 1 – HIM images of three VACNT samples at 0 and 45° tilt from normal incidence. The depth of field is approximately equal to the field of view, which is 10 times the scale bars.

Oxygen plasma treatment is shown to reduce the reflectance of VACNT by altering the surface morphology, characterised by crust ablation and tip formation. The crust has a higher reflectance than the bulk, since it is more dense and uniform.

N.A. Tomlin, A.E. Curtin, M. White, J.H. Lehman, *CARBON* **74** 329 – 332 (2014). <http://dx.doi.org/10.1016/j.carbon.2014.03.040>

Total Hemispherical Reflectance - NIR

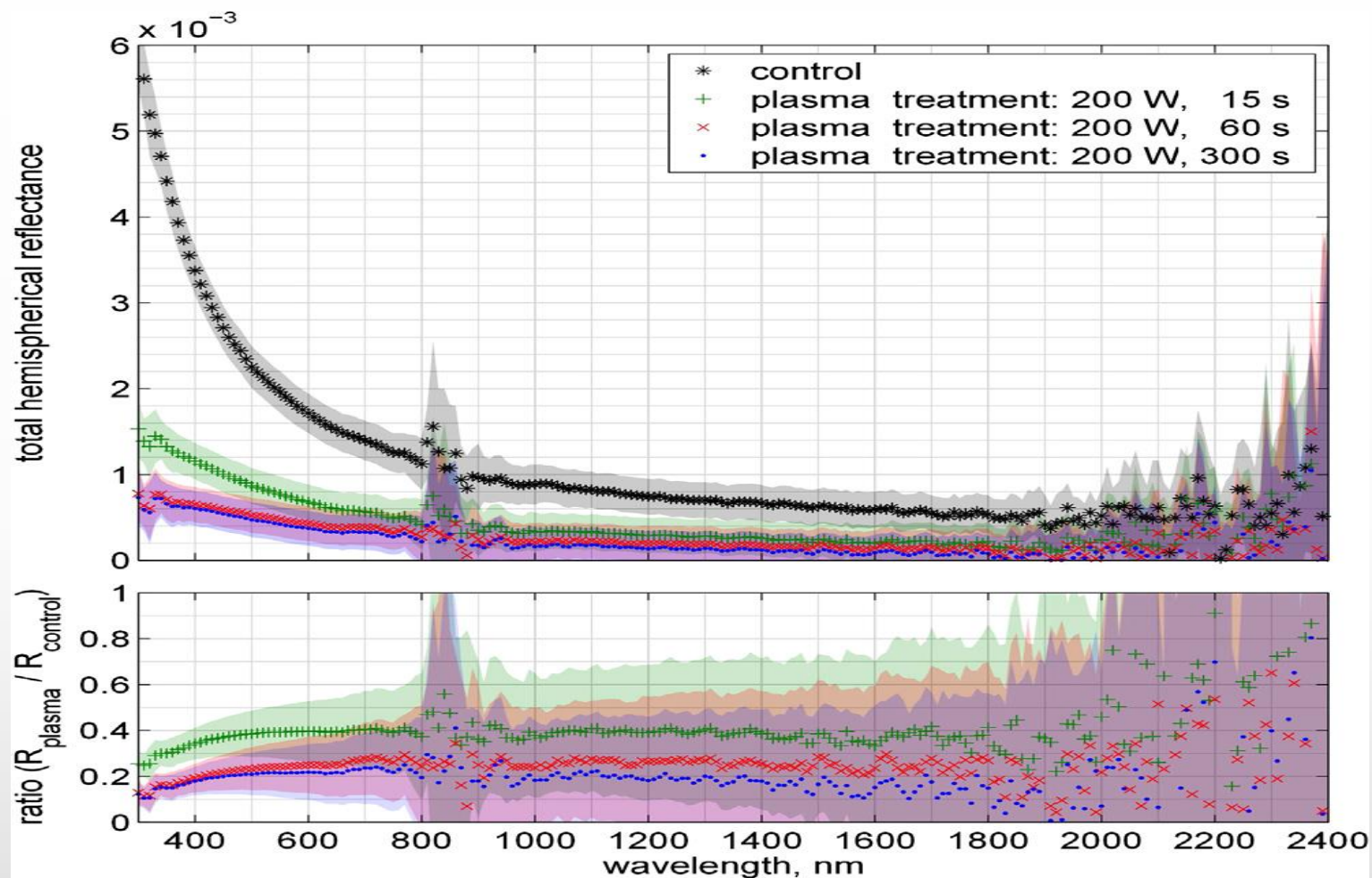
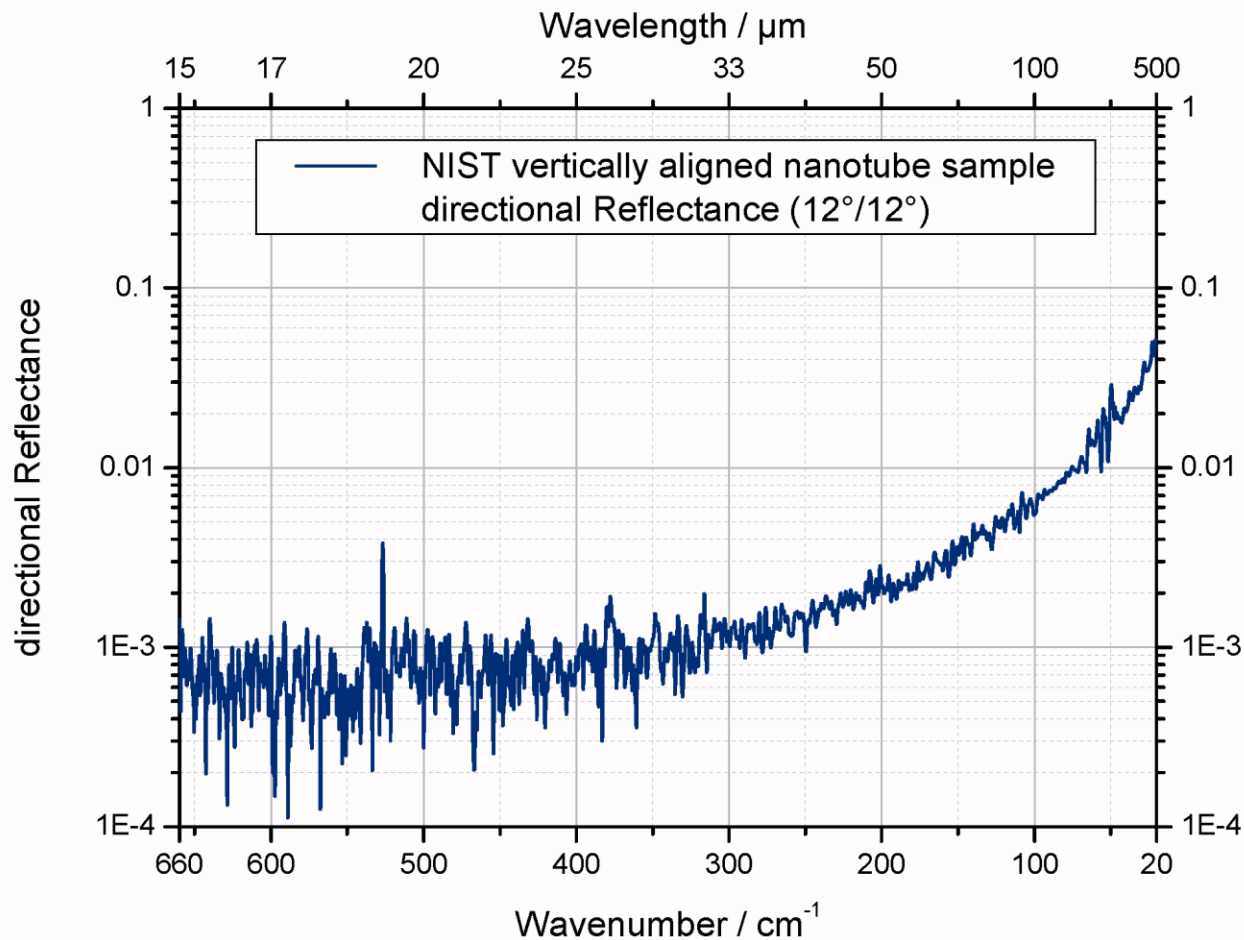


Fig. 2 – (Top) Total hemispherical reflectance with uncertainty ($k = 2$) shown by shaded regions. (Bottom) Ratio of total reflectance between plasma-treated samples and control sample

FIR Reflectance - PTB Measurement



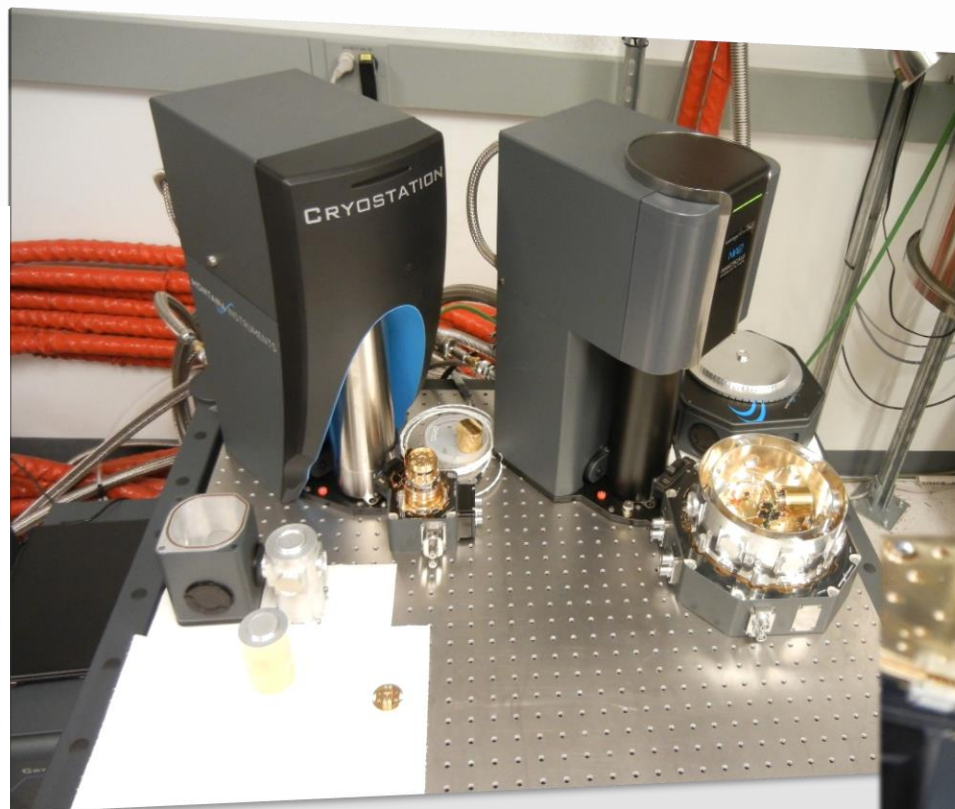
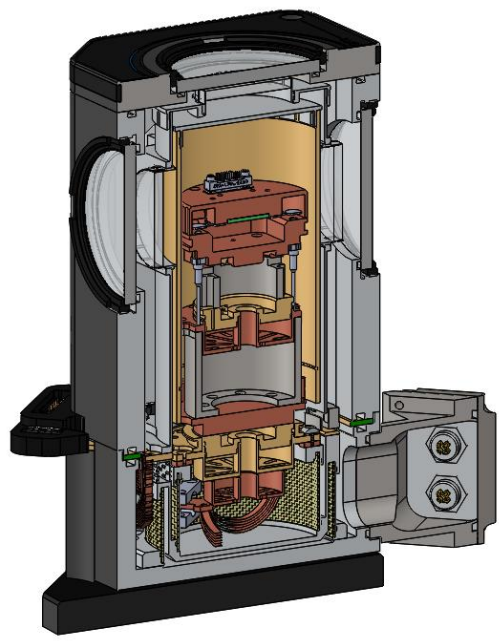
PTB, Department 7.3, M. Kehrt, C. Monte

3D cavities, a very typical geometry – absorptance $> 0.9999\%$; planar 2D design using carbon nanotube absorbers, absorptance of > 0.9995 in visible and NIR when oxygen plasma treated, decreasing to 0.999 at 30 μm .

NIST Table Top Cryogenic Systems using CNT Absorbers

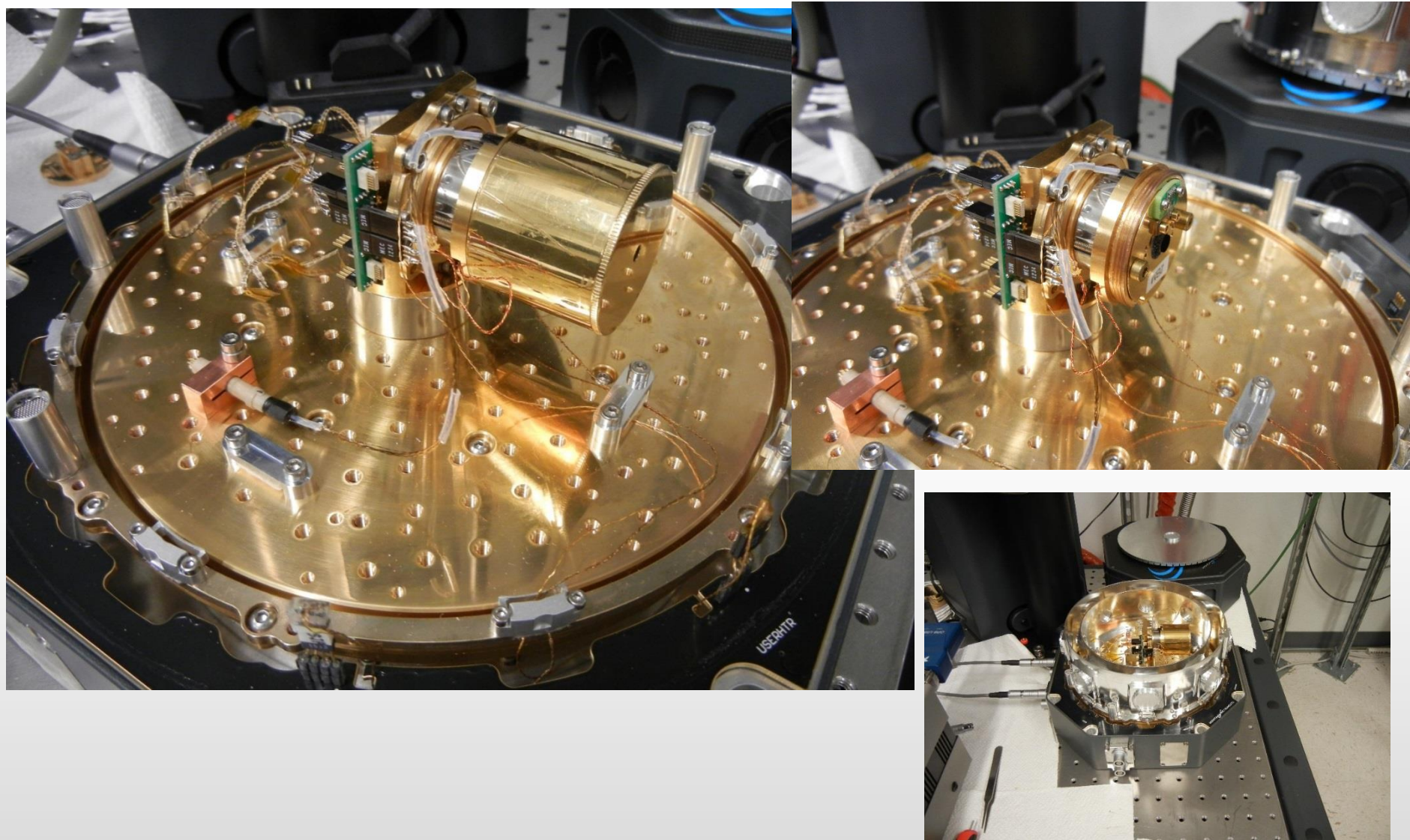
Cryogenic CNT detector is the basis for our table top fibre coupled and THz open beam radiometers, 10's μW to 1 mW

- 850 nm
- 1310, 1550 nm
- 119, 394 μm



These radiometers will become our respective primary standards

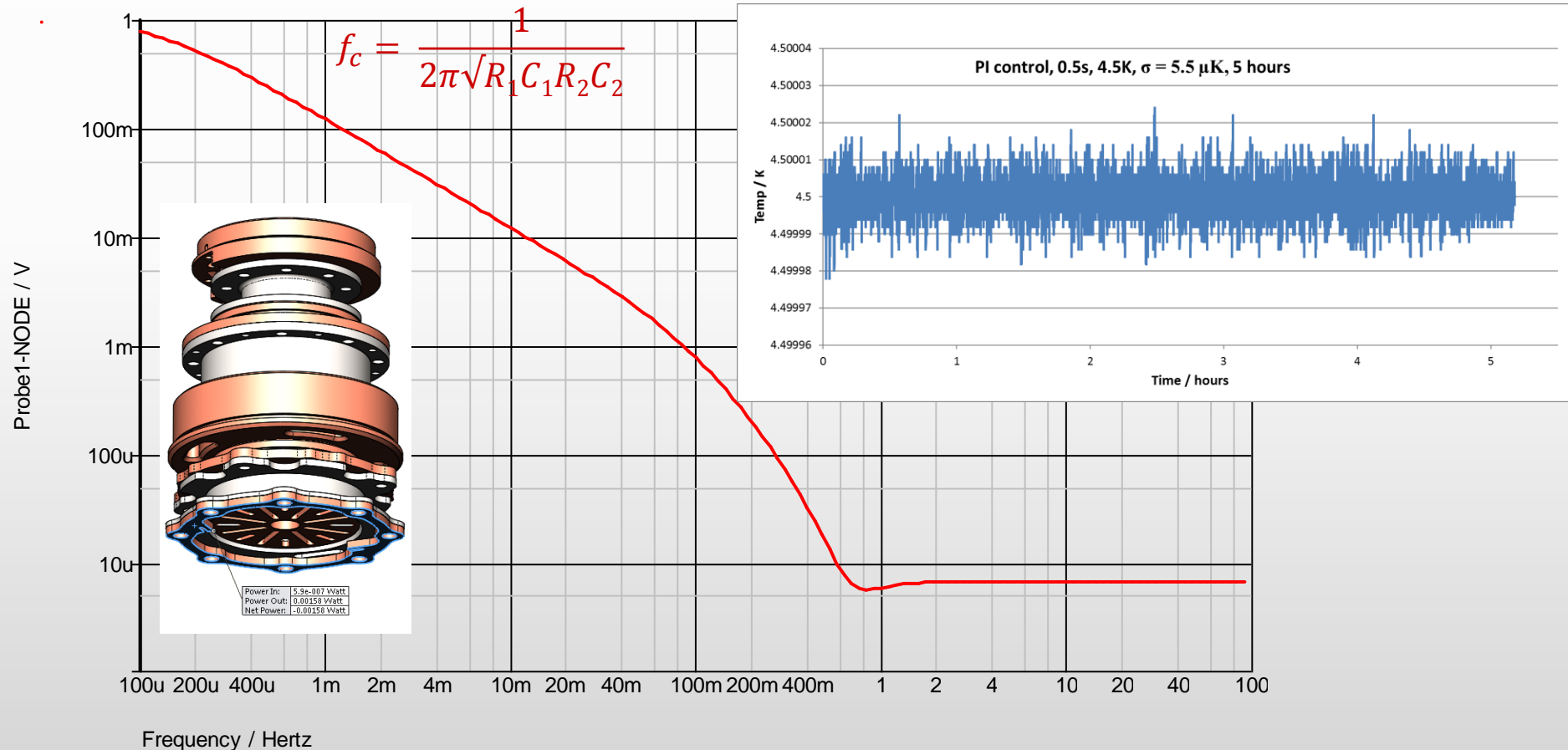
System - 4 K THz Absolute Radiometer



J. Lehman, A Steiger, N.A. Tomlin, M. White, M. Kehrt, I. Ryger, M. Stephens, C. Monte, I. Mueller, J. Hollandt, M. Dowell Submitted Optics Express 2016

Thermal Stability of Platform - RC Filters

The cyclical thermal fluctuations of cryocoolers at 1.4 Hz present a unique challenge to the researcher trying to establish a stable temperature platform of the order 1 μK with low noise. Here, thermal stability is achieved with a 2 pole Butterworth filter. Each pole has the same time constant optimising performance. The electrical analogue is the 2 pole Butterworth filter.



Thermal Stability of Platform - RC Filters

A thermal filter is comprised of a thermal impedance R (K/W) and a thermal capacitor C of heat capacity J/K, with time constant in seconds. Temperature fluctuations are reduced at 10 dB / decade for a single pole filter.

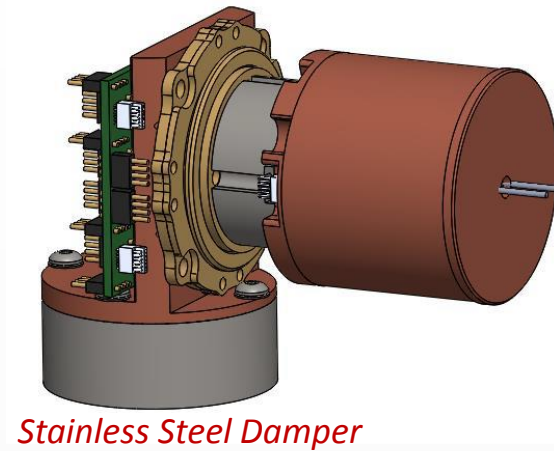
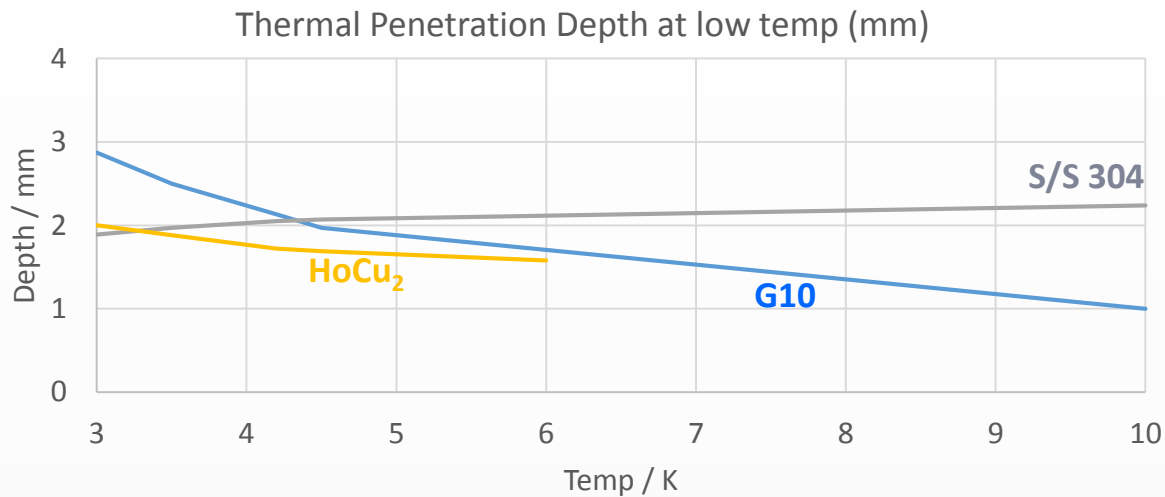
Example: the 3 dB cutoff frequency f_c for a filter with an RC of 180s is 0.88 mHz. This attenuates the 1.4 Hz temp. fluctuation of the cryocooler by 1000x or 30 dB.

$$f_c = \frac{1}{2\pi RC} = 0.884 \text{ mHz} \Rightarrow 10 \times \log \frac{1.4}{8.84 \times 10^{-4}} = 32 \text{ dB}$$

Temp (K)	Cu RRR 150	Pb	Stainless Steel 304	HoCu ₂
3.0	0.00052	0.0035	0.0117	0.161
3.5	0.00069	0.0057	0.0134	0.199
4.2	0.00098	0.0106	0.0158	0.253
4.5	0.00112	0.0145	0.0169	0.277
10.0	0.00768	0.1557	0.0411	0.257

Volumetric heat capacity (J/cm³-K) of typical materials at cryogenic temps. From NIST cryogenic database.

Thermal Stability of Platform - Dampers



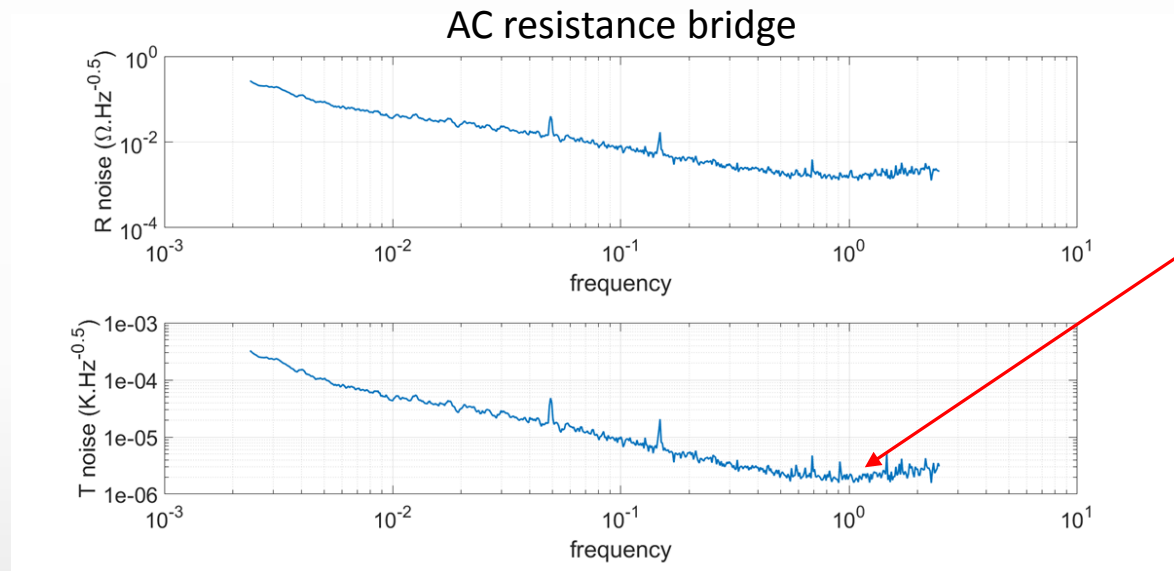
Thermal penetration depth (mm) of common materials at cryogenic temps, $f = 1.4$ Hz

For a periodic time dependent thermal fluctuation it can be shown that $\Delta T(x) = \Delta T(0)\exp(-x/\delta_{th})$ where x = distance, δ_{th} the thermal penetration depth, α the thermal diffusivity of the material and f the frequency of the driven temp. oscillation.

δ_{th} is the thermal penetration depth – it is that depth at which the amplitude of the disturbance has dropped to $1/e$ of its initial value $\delta_{th} = \sqrt{\frac{\alpha}{\pi f}}$

Thermal Stability of Platform – Effectiveness of Filtering

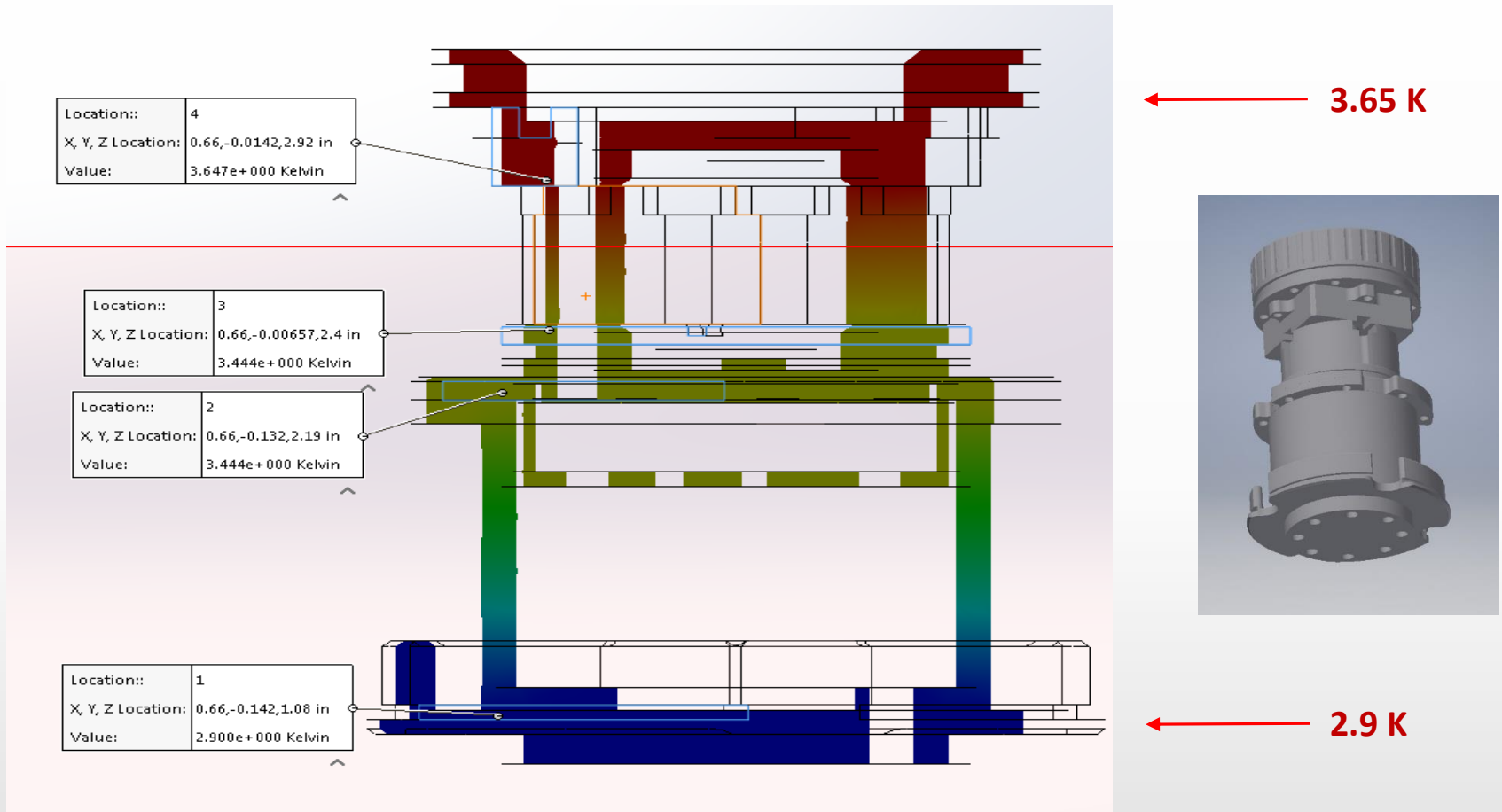
Temperature and Resistance noise spectrum of thermal system. Measurements taken with a commercial AC resistance bridge, sampled at 5Hz, bridge excitation 10 mV, constant current.



No excitation at 1.4 Hz.

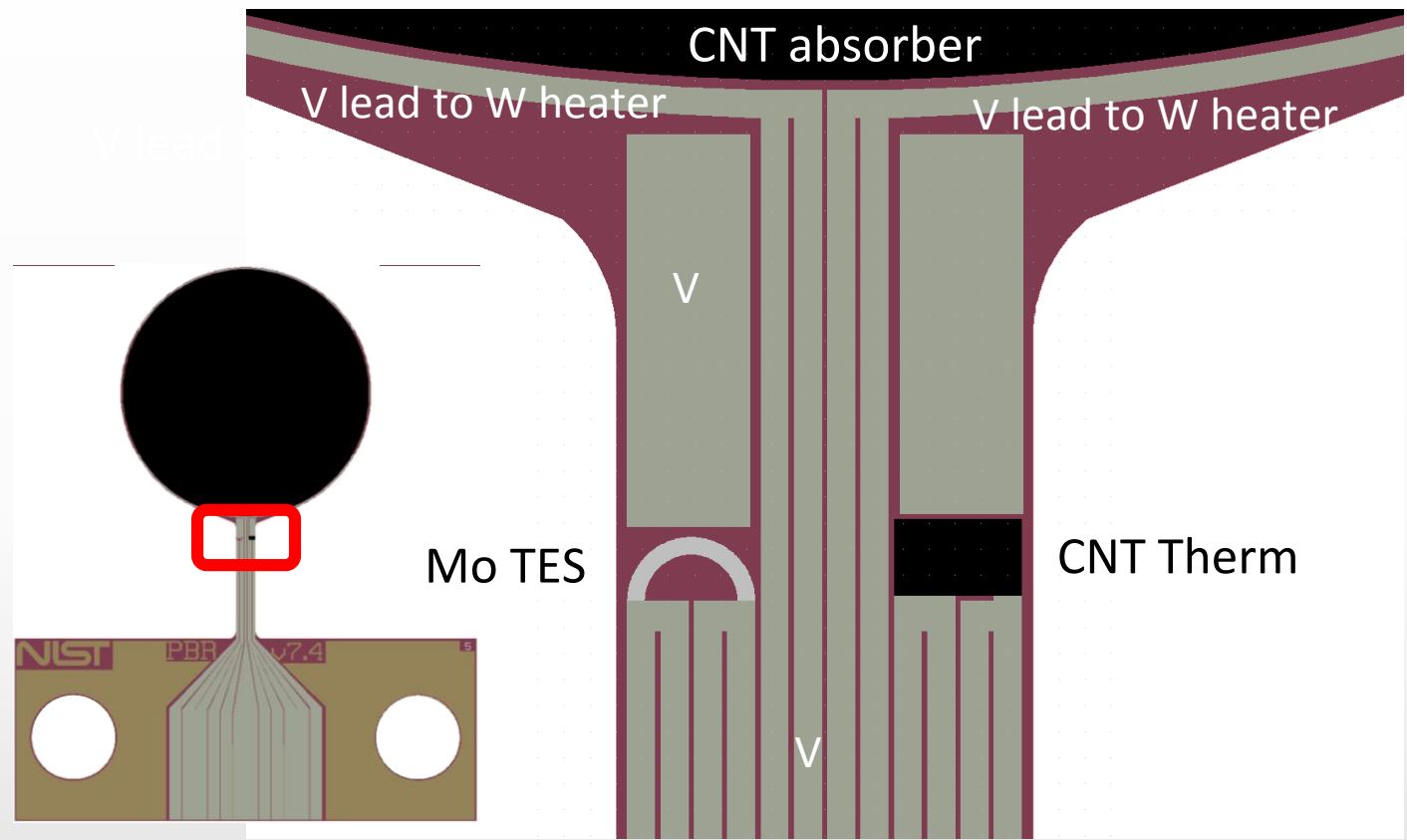
There is effectively 50 dB attenuation of the 1.4 Hz signal; from 200 mK pk-pk to 2 μK . No excitation visible at 1.4 Hz. Thermal damping is very effective.

Thermal Profile - 1 mW Input Power



1 mW input power: 0.55 K temp rise across 1st stage RC filter, 0.2 K across 2nd stage

Design - Closeup of Patterning

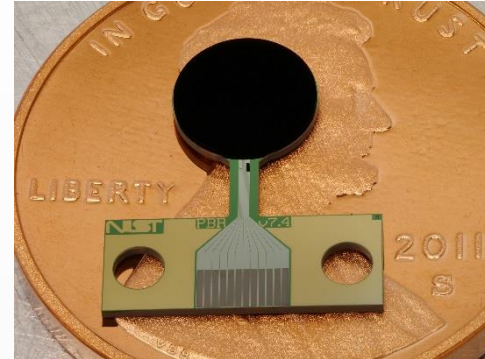
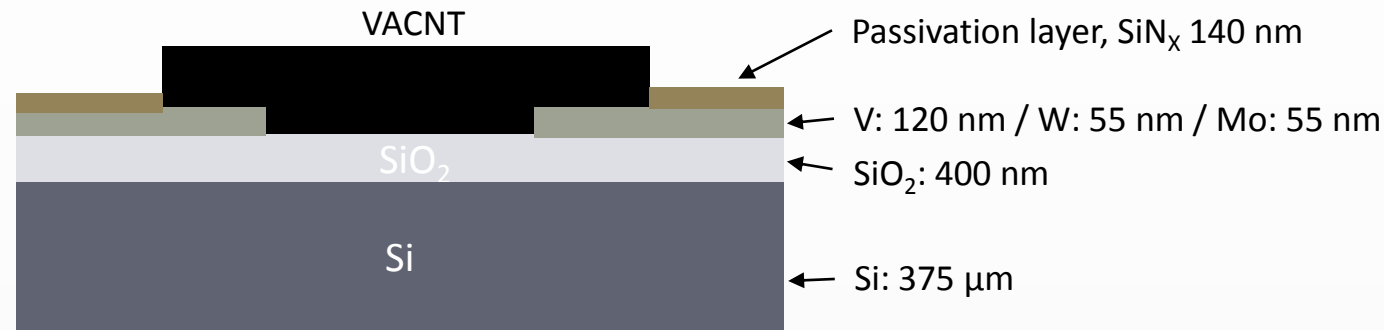


Credit: Nathan Tomlin, NIST

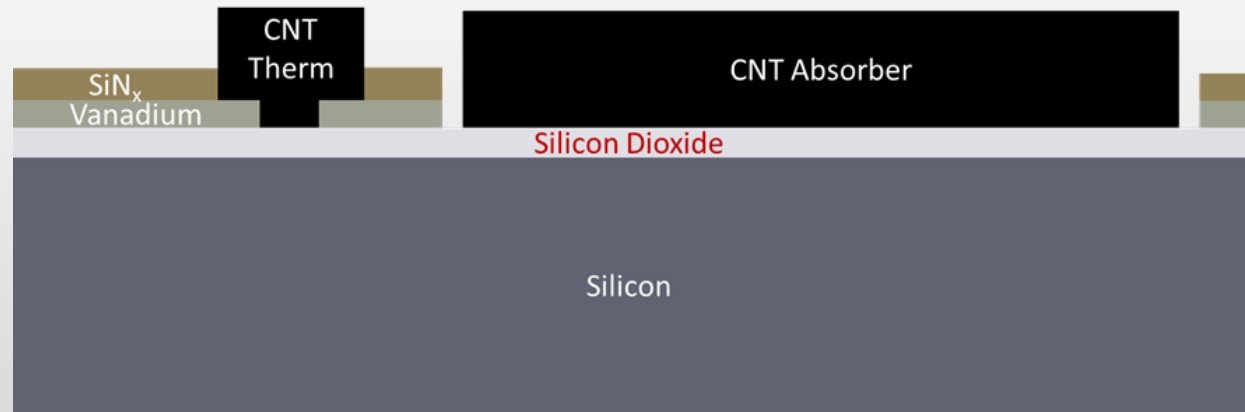
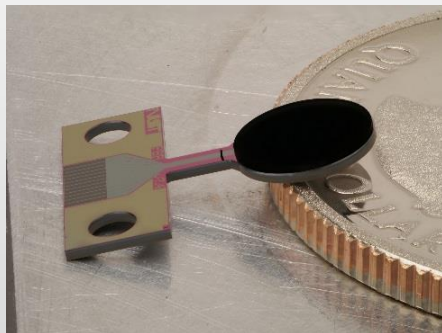
Position of Mo TES, CNT thermometer and Vanadium leads to the Tungsten heater

Design - PBR Structure

Side view showing the various layers of the planar radiometer fabricated in the NIST cleanroom



Side view of lithographic pattern

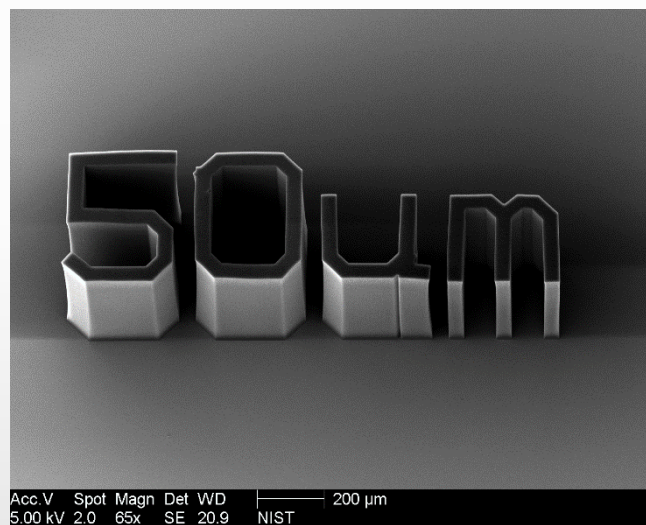


Extended side view

Characterisation - Reflectance Measurement

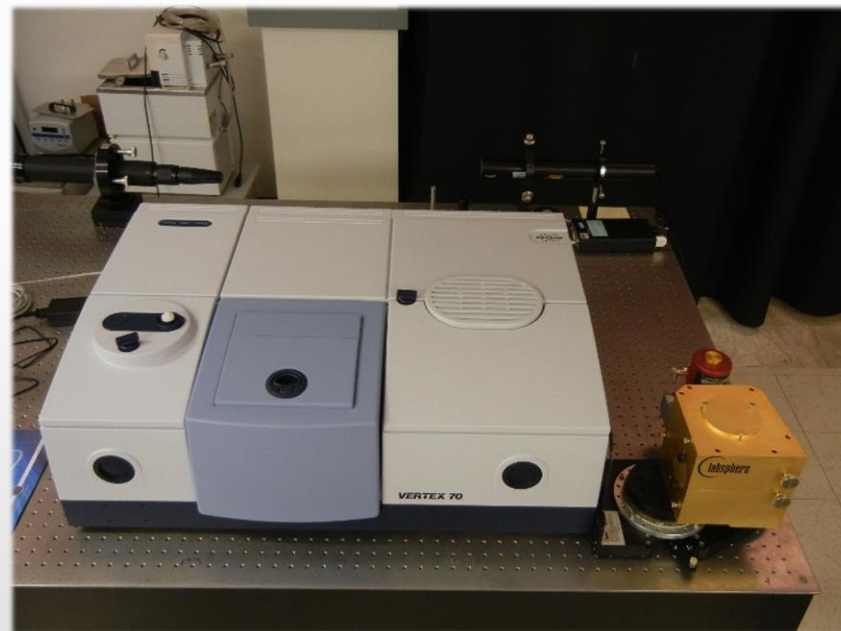
In conjunction with NIST Gaithersburg we are developing the capability to characterise the reflectance of CNT's from 400 nm to 25 μm , limited principally by the IR detectors available, sources and the blackness of the nanotubes themselves.

We are using a Lambda 1050 system for the visible and NIR and an FT system for the MIR.



Credit: Chris Yung, NIST

Nanotubes can be easily patterned



It is not easy to measure reflectance at the 0.1 % level

Characterisation - Physical Properties System

The Physical Properties Measurement System (PPMS) gives us the ability to characterise our chips within the hour. A new sample is mounted on a puck and loaded via a probe to the sample space and cooled to 4K. Measurements are collected automatically as the system cools.



Photo courtesy Quantum Design

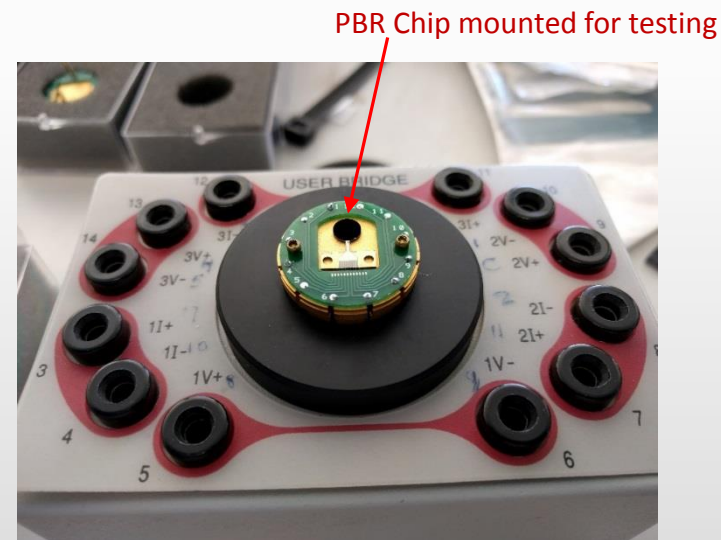
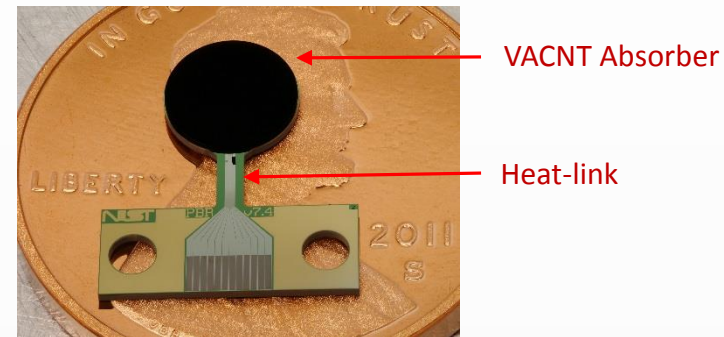
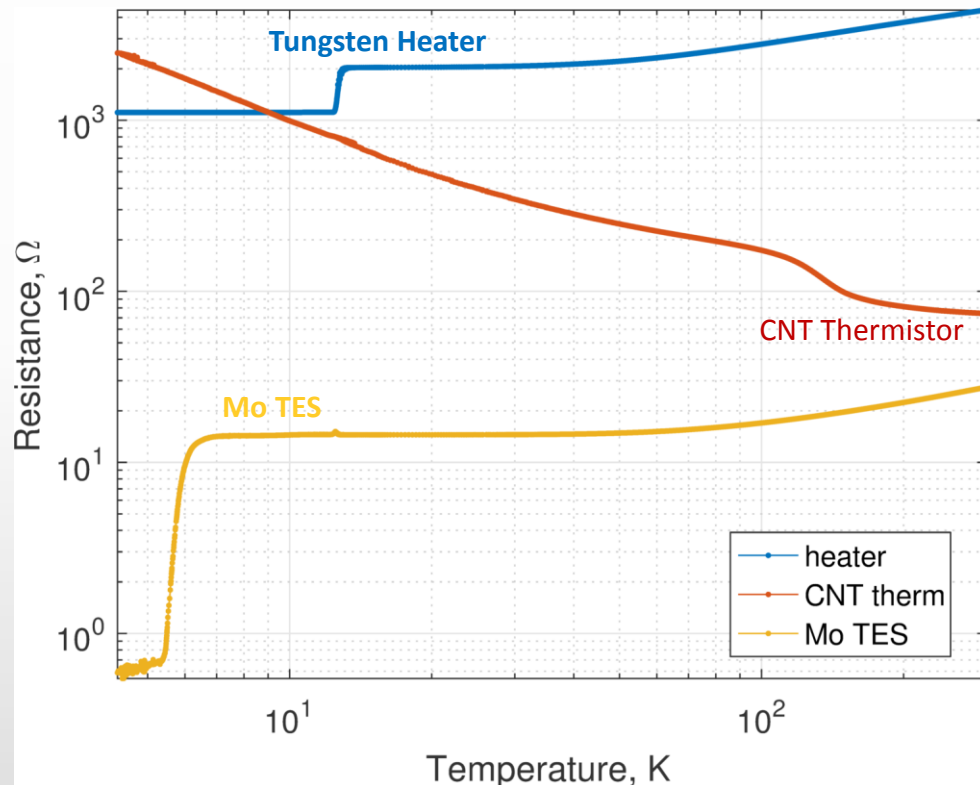


Credit: Chris Yung, NIST

Mount for testing new chips at 4K

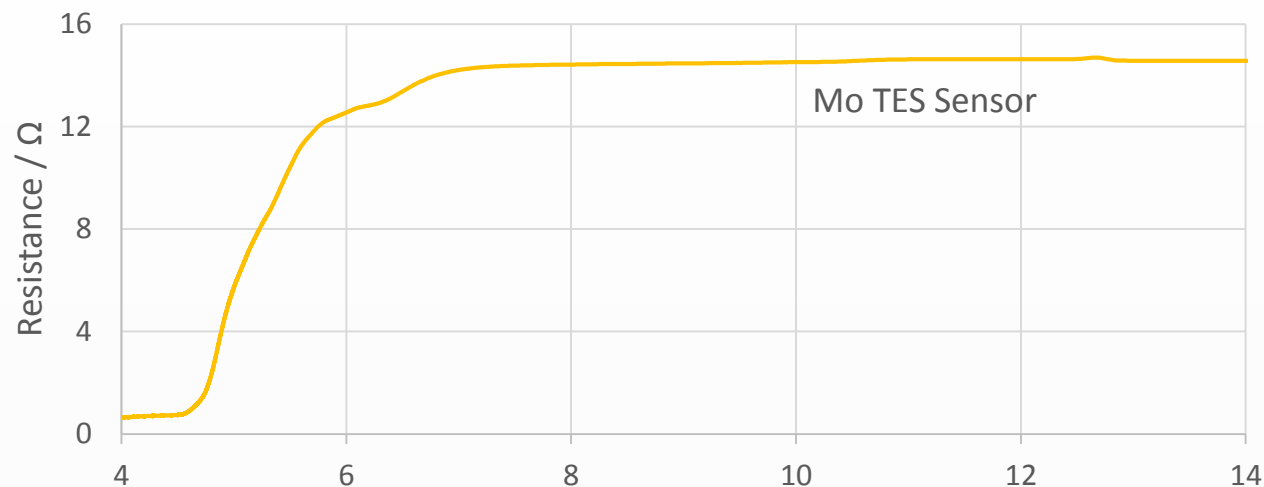
Characterisation - Chip Radiometer

We have used both the PPMS and 4K radiometer to characterise the performance of the PBR chips. In particular the Mo superconducting sensor and the Vanadium wiring along the heatlink, and the temperature dependence of the CNT sensor.

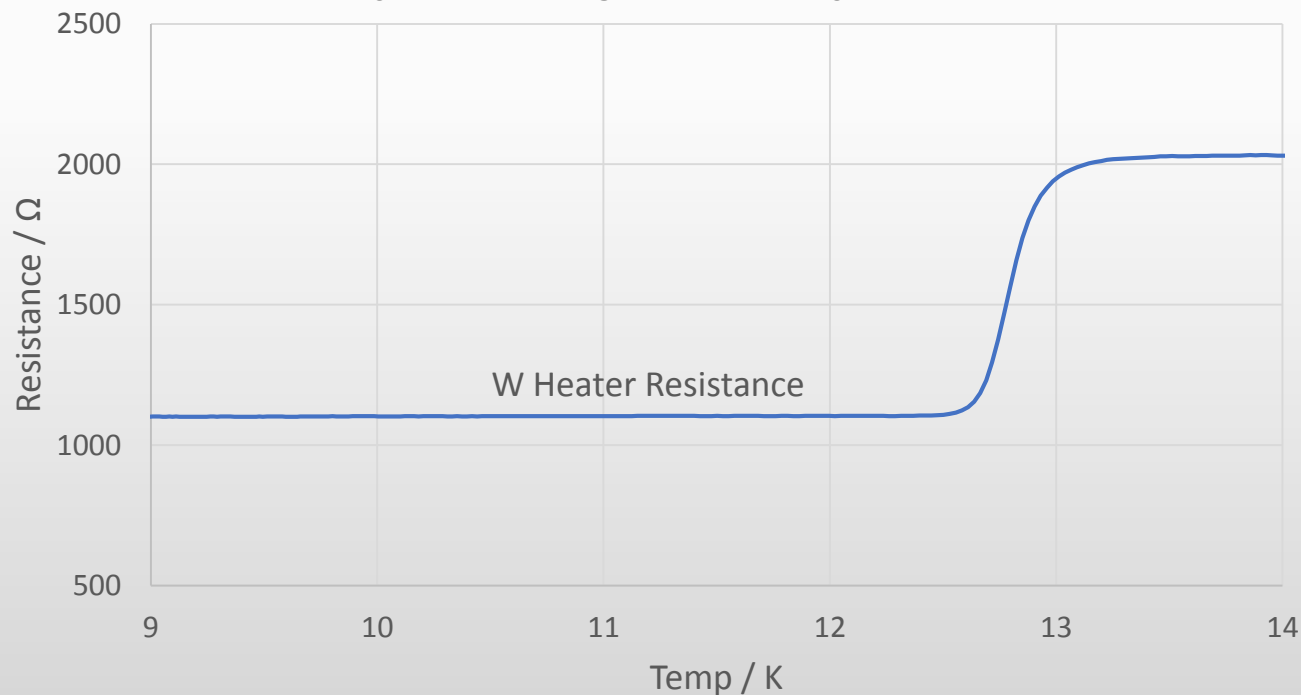


Fully functional micro-fabricated chip with VACNT absorber, Mo sensor and tungsten heater

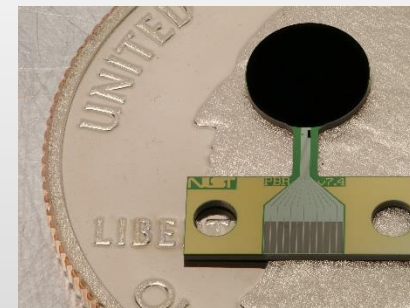
..... and in More Detail



- Mo transition at 6 K
- 10 Ω / K
- Cernox > 100 Ω / K
- Thermal link impedance 1430 K/W



- Vanadium wiring transition at 13K
- W heater 1100 Ω



- ❖ NIST has initiated a program called “*NIST on a Chip*” with the intent of establishing chip-scale standards in compact, portable packages.
- ❖ Carbon nanotube array technology has enabled the development of near perfectly absorbing planar detectors, manufactured using micro-fabrication techniques.
- ❖ Collaboration with *LASP* developing the application of carbon nanotube technology to space based detectors.
- ❖ The development of a detector paradigm that has led to the establishment of a new family of laser power standards, with application to remote sensing observations.

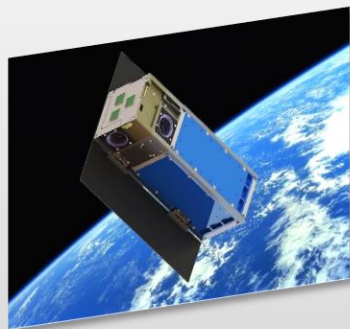


Photo courtesy Blue Canyon Technologies, Boulder

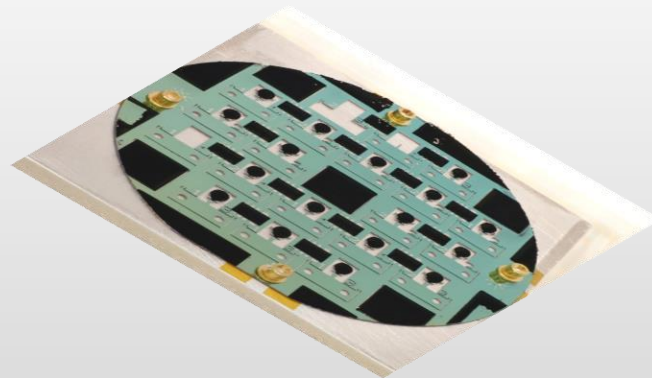


Photo courtesy Nathan Tomlin, NIST Boulder

THANK YOU

