A New Generation of NIST Laser Power Standards Using Microfabrication Techniques and Carbon Nanotubes

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Laser Sources and Detectors

PHYSICAL MEASUREMENT LABORATORY

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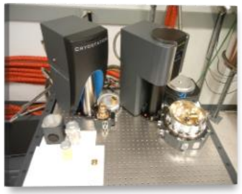
Develop next generation of sources and detectors for laser power and energy measurements traceable to NIST

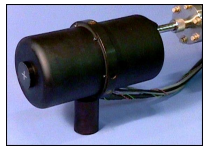
NIST
National Institute of Standards and Technology
U.S. Department of Commerce

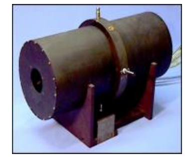
Extending Wavelengths, Power Range, Performance

Carbon Nanotube Cryogenic Radiometer*

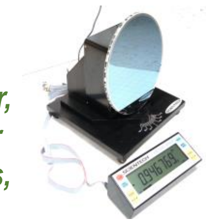


Electrical Standards

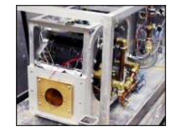




Smaller, higher powers, faster (msec)



Radiation Pressure Meter



Flowing water power meter

Room Temp Electrical Substitution Radiometer

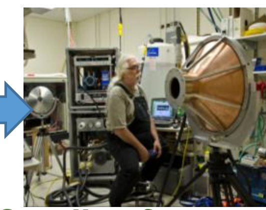
Chip-scale; fast smaller, portable, faster (tens of seconds)

Cryogenic Radiometer
0.4 µm to 2 µm
100 µW to 1 mW

C Series Calorimeter
0.4 µm to 2 µm
50 µW to 10 mW
10 mJ to 30 J

K Series Calorimeter
0.4 µm to 20 µm
1W to 1000 W
300 J to 3000 J

BB Calorimeter
1.06 µm to 10.6 µm
100 W to 200 kW
10 k J to 6 MJ



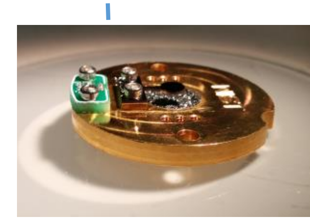
Smaller, faster (tens of seconds)

THz wavelengths

Q Series Calorimeter
1.06 µm
0.5 J to 15 J total

QUV Calorimeter
248 nm
0.5 J to 15 J total

QDUV Calorimeter
193 nm
0.5 J to 15 J total

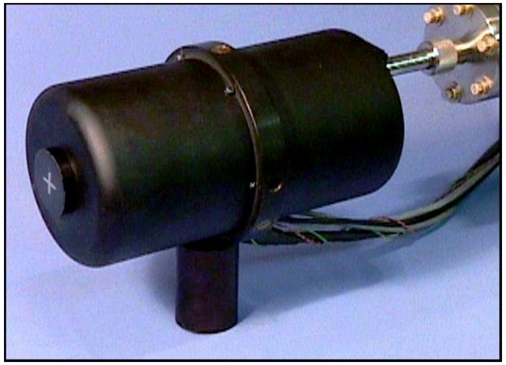


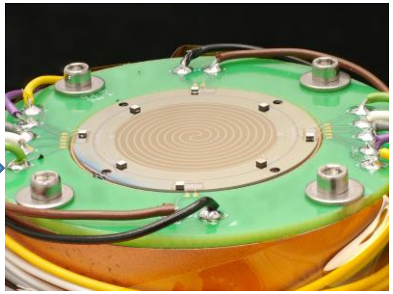
Planar Hyperblack Absolute Radiometer

***Malcolm White, "Characterization of New Planar Cryogenic Radiometric Standards under Development at NIST", Thursday morning in *Advancements in Radiometric Calibration State-of-the-Art* session**

Application: Room Temperature Laser Power Standard

- Current C-series standards are used for UV-NIR CW laser power calibrations
  - 10 mW – 1 W
  - 325 nm – 1.93 mm
- C-series calorimeters have slow responses (~15 min), are limited to laboratory use, and are legacy instruments whose designers have all retired
- Develop a carbon nanotube micro-fabricated bolometer as a C-series replacement that is faster (~2 min), automated and portable.

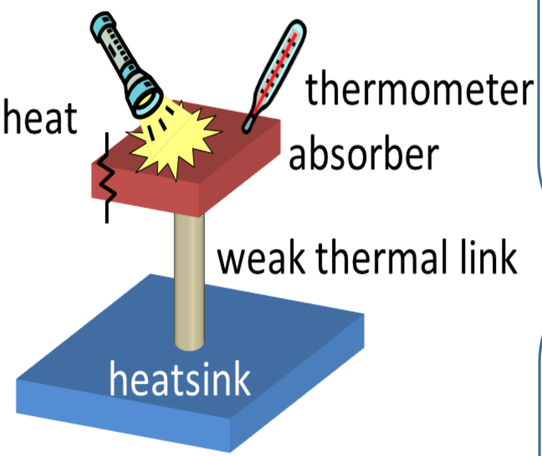


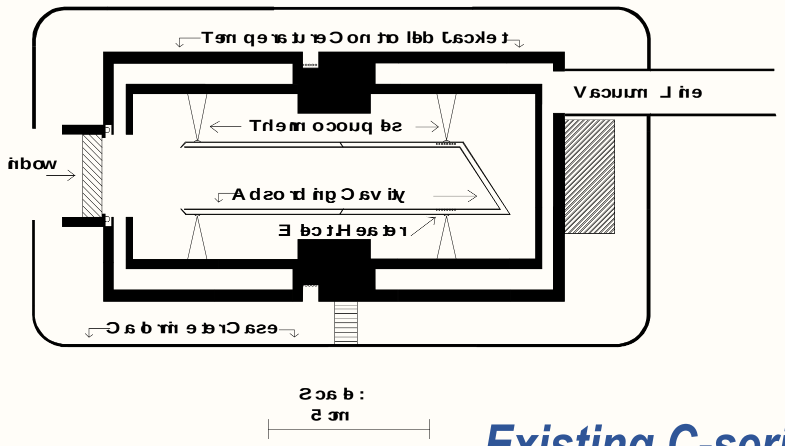


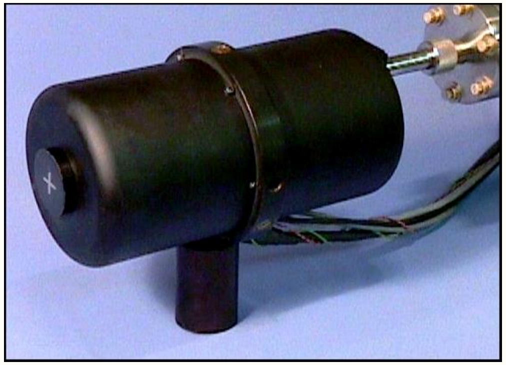
Requirement	Minimum	Goal
Power Range	10 μW - 100 mW*	1 μW - 200 mW *
Wavelengths	325 nm - 1.93 mm	244 nm - 1.93 mm
Laser Beam Size	2 cm	2 cm
Time to Equilibrate	120 sec	60 sec

*Use of beamsplitter in test arrangement increases range by 10X

Calorimeter/Bolometer Concept





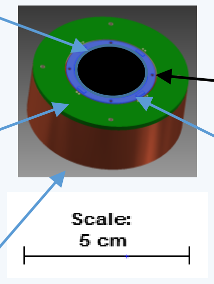


Existing C-series calorimeter

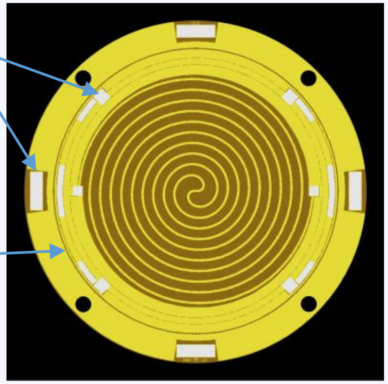
Planar, VACNT absorber

Printed circuit board

Temperature controlled heat sink



Thermistors (8)



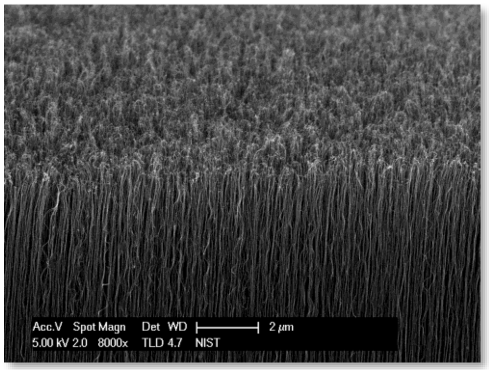
Micromachined weak heat link

Vacuum line, window not shown

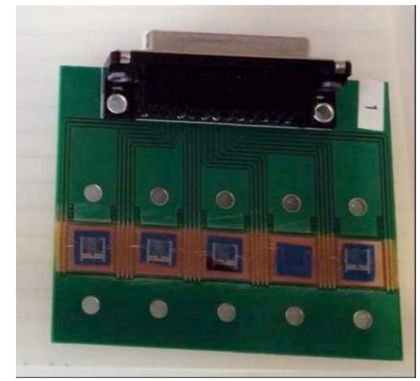
Prototype #1, 'Next-Gen C'Bolometer

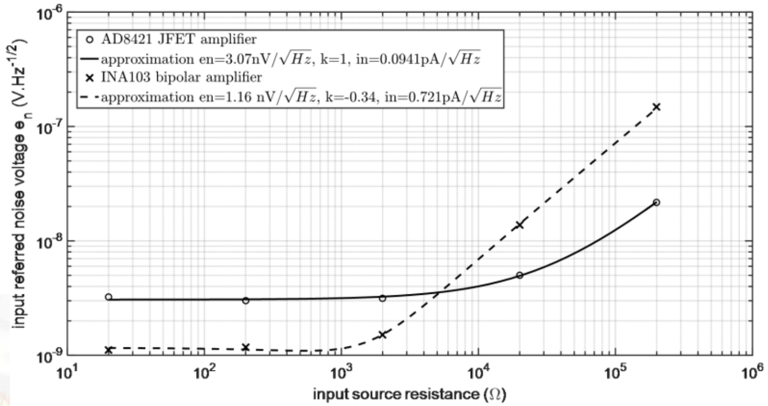
Advancing Technologies for Next Generation Standards

Understand impact of process parameters on Vertically Aligned Carbon Nanotube (VACNT) growth

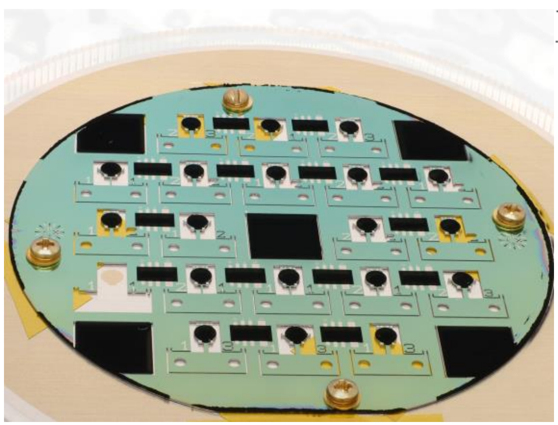


Fabrication and measurement of low-noise thermistors, room temp to 4°K, compatible with nanotube growth



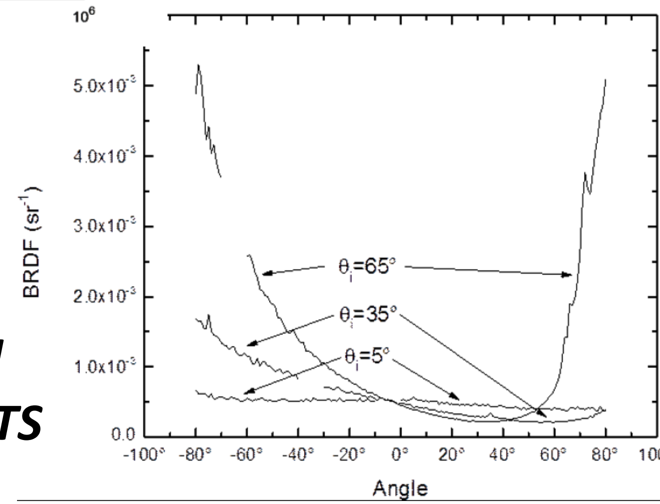


Low-noise electronics for bolometers



Integrate VACNTs with microfabricated electronics

Characterize Optical Performance of VACNTS



VACNT Growth at NIST

- **Multiple depositions/growths are possible in one day.**
  - **Controllably grow *vertically aligned* CNTs (VACNTs) of desired height.**
  - **Reliably grow CNTs on various substrates – Si, SiO_2, SiN_x**

Investigating

- ***Impact of process parameters on VACNT height, density***
- ***Growth of VACNTs on metals: W, Mo, Au***
- ***Micro-fabricated thermistors with VACNT growth***

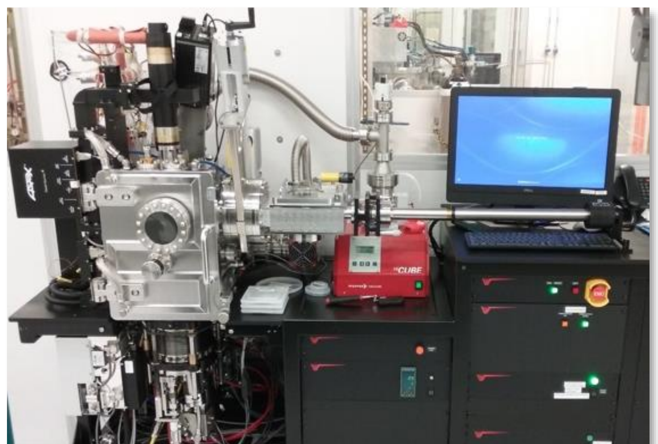
Sputter tool – catalyst deposition

Bi-layer catalyst:

Aluminum oxide ~10 nm

Iron ~2 nm

PECVD system – CNT growth



Temperature: 700 – 800 C

Pressure

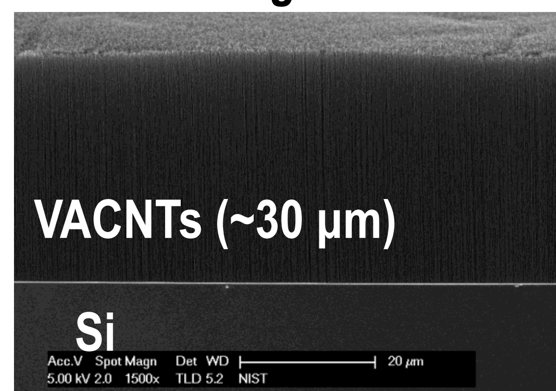
Gas: CH_4 or C_2H_4

Ratios: Ar : H_2 : CH_4 : C_2H_4

Flow rates

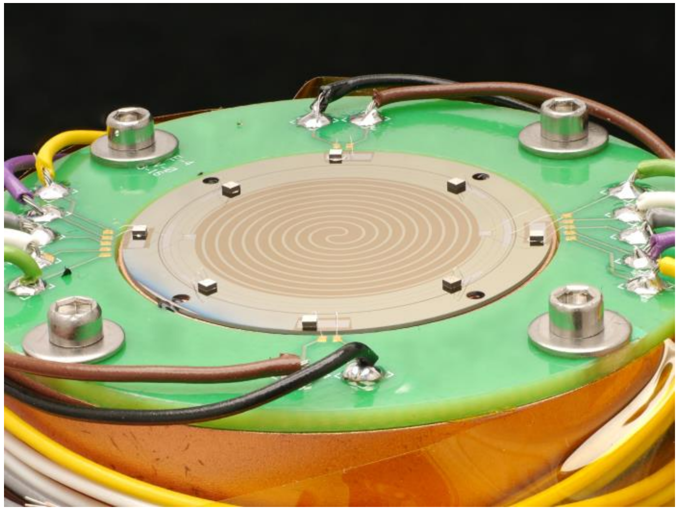
Plasma power

DC bias

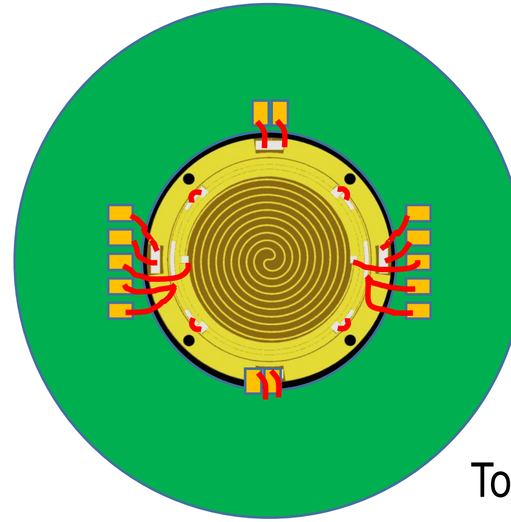


SEM side view of VACNTs grown at NIST

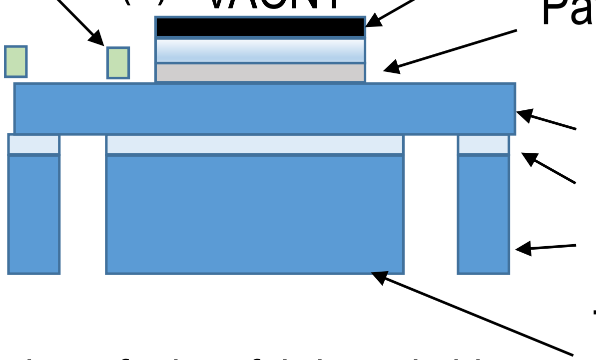
Prototype #1, Micro-fabricated Chip



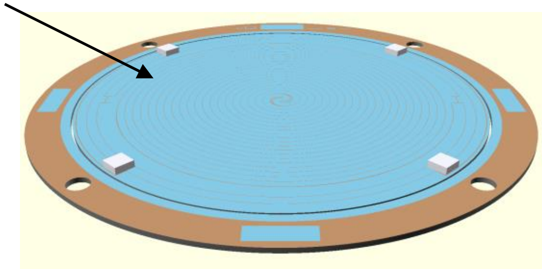
First prototype does not include VACNTs



Top view



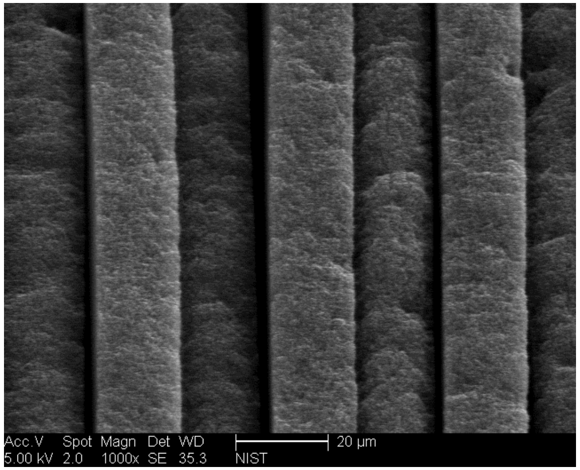
Side view of micro-fabricated chip. Not to scale.



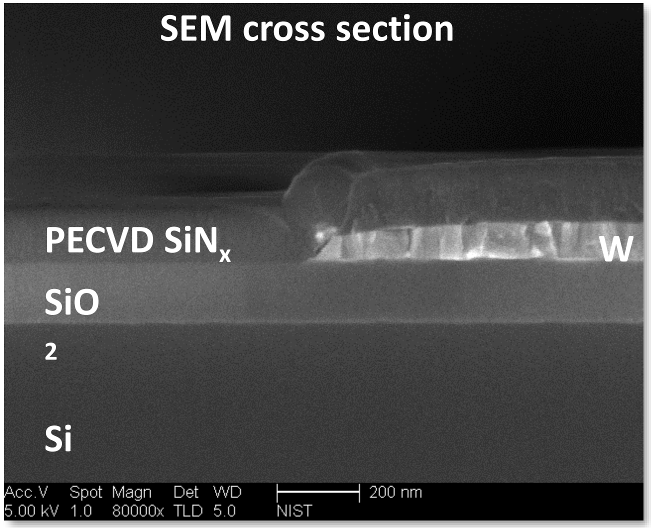
VACNTs (not shown) grown over tungsten heater

VACNT Growth on Passivated Tungsten

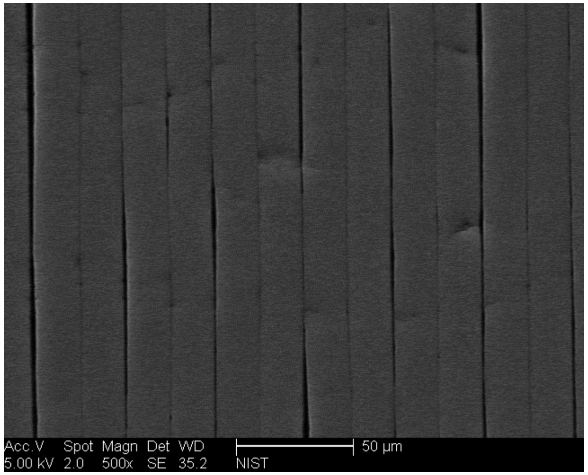
- VACNT growth height can vary significantly depending upon substrate and sub-substrate
  - VACNT growth tested on passivated (aSi, SiO_2, SiN_x) tungsten wires (10 µm)
  - Best results on SiN_x



<u>Before optimization</u>: VACNTs 10 µm shorter on W

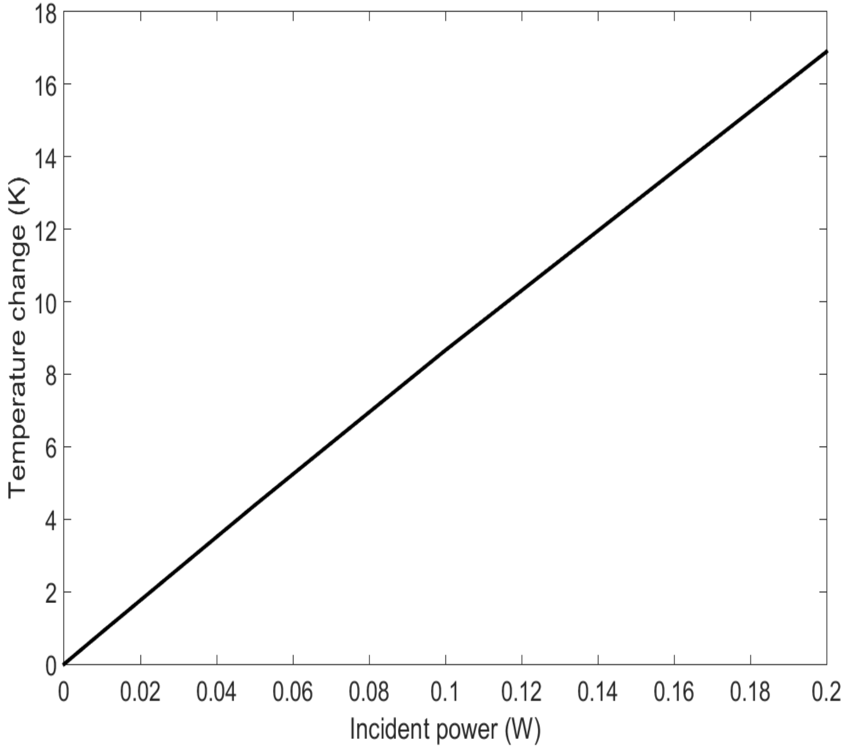


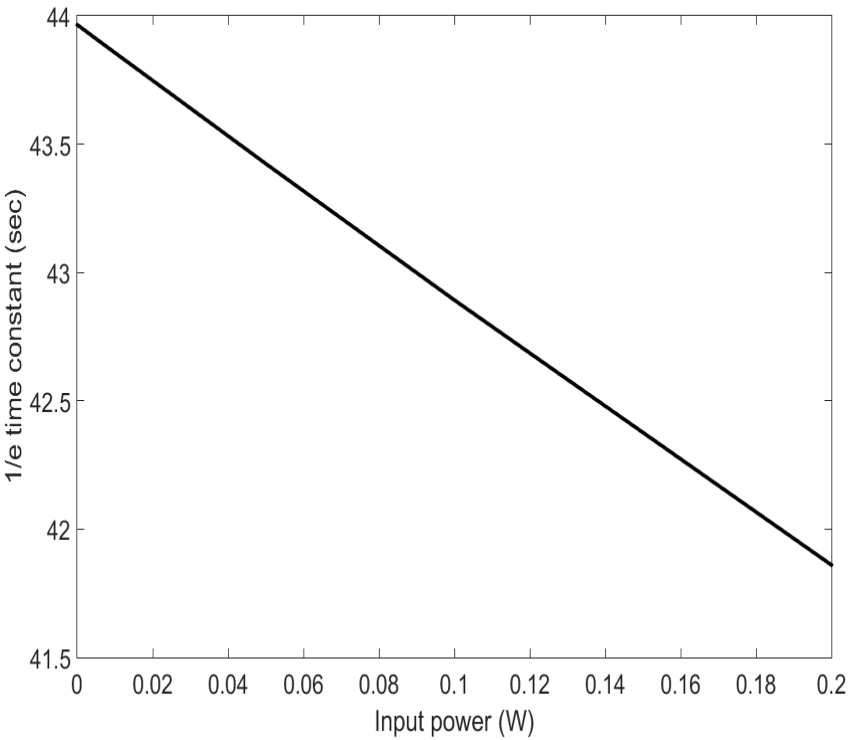
Step coverage of SiNx/W before growth



<u>After optimization</u>: Nearly planar VACNT growth

Expected Performance: Response Time and Temperature

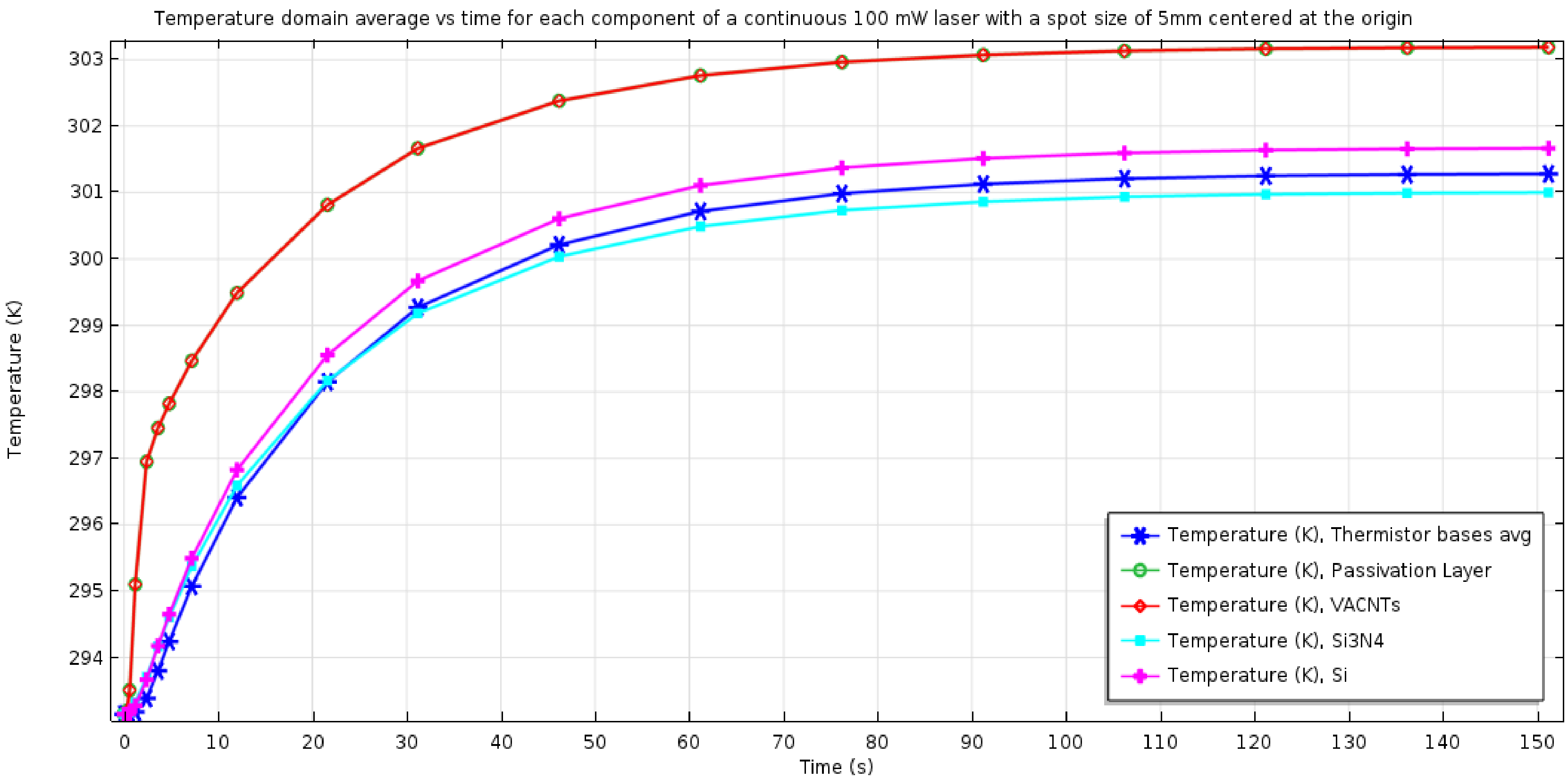




MATLAB model of chip predicts heat rise and time constant

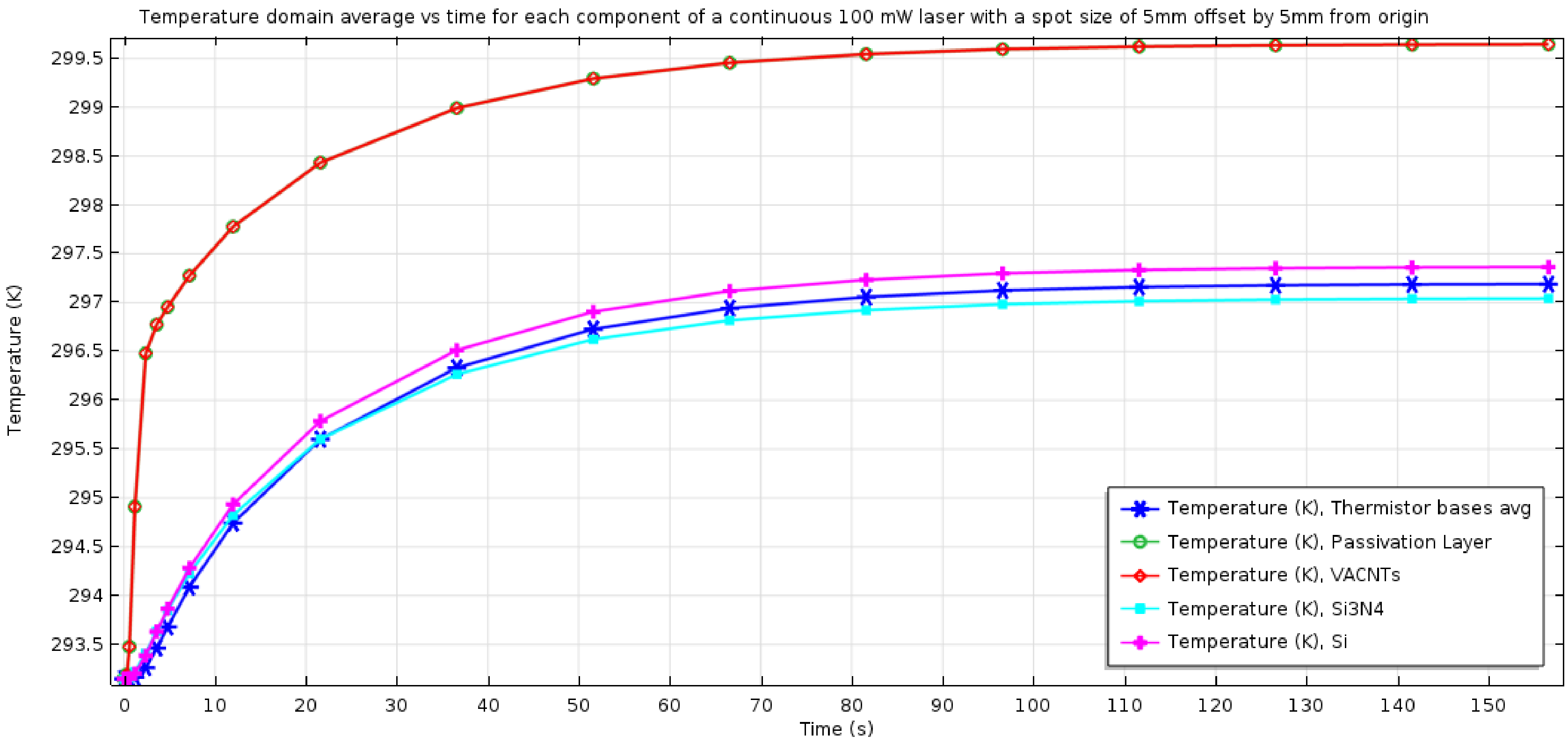
Performance depends on materials properties of chips after nanotube growth

Expected Performance: Laser centered



COMSOL model of chip predicts temperature of individual layers and impact of mis-alignments in laser

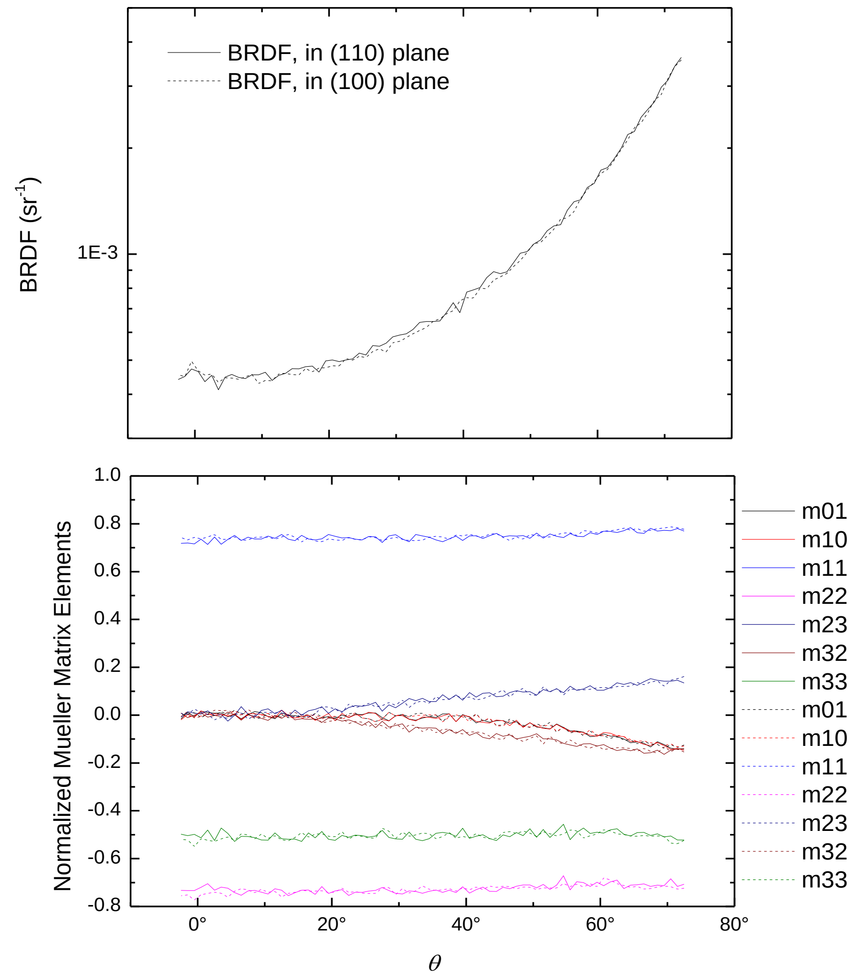
Expected Performance: Laser offset



For 100 mW laser with 5 mm spot size offset by 5 mm measured dT may vary by up to 5 degrees. Heat link to be modified to reduce effect in next prototype

Expected Performance: Nanotube Scatter/Reflectivity

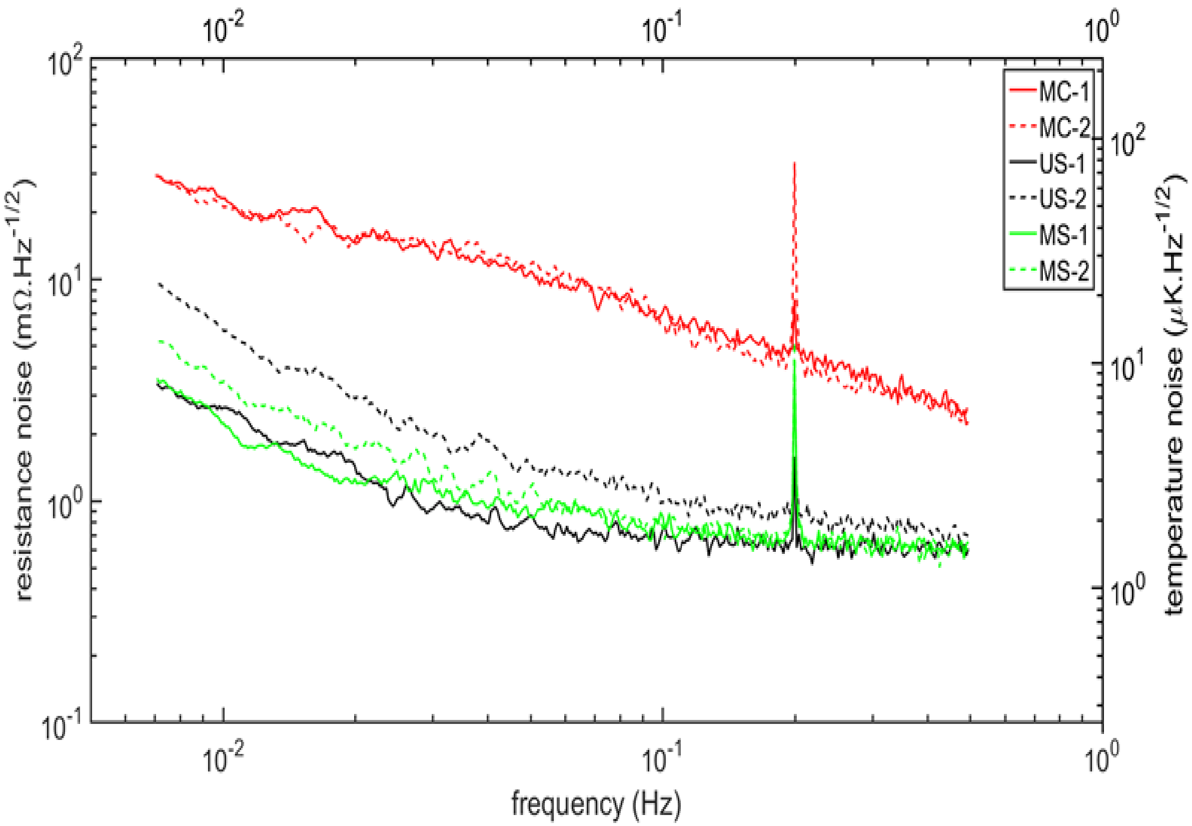
- VACNT total hemispherical reflectances have been measured ~ 0.07% for visible wavelengths[1], ~ 0.1% for 5-10 μm, flat to 50 μm
- Surface preparation can change reflectance[2]
- Collaborating with Laboratory for Atmospheric and Space Physics, University of Colorado and with Optical Radiation Group, NIST Gaithersburg to characterize VACNT scatter/reflectivity as function of surface preparation



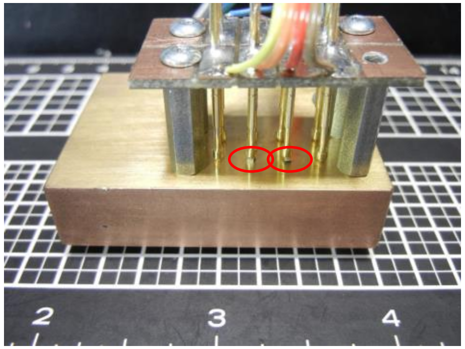
VACNT BRDF and Mueller Matrix elements at 633 nm, Thomas Germer, Sensor Science Division, NIST Gaithersburg

[1]Z. Yang, L. Ci, J. A. Bur, S. Lin, and P. M. Ajayan, Nano Lett. 8, 446 (2008); C. J. Chunnilall, J. H. Lehman, E. Theocharous, and A.Sanders, Carbon 50, 5348 (2012).
[2]Tomlin, N.A., Curtin, A.E., White, M., Lehman, J.H., Carbon 74, 329 (2014)

Thermistor Noise Measurements



- Thermistor noise will limit resolution of temperature measurement
- Able to measure noise of both commercial and in-house thermistors[1]



[1]Ivan Ryger, Dave Harber, Michelle Stephens, Malcolm White, Nathan Tomlin, Matthew Spidell, John Lehman, "Noise characteristics of thermistors: measurement methods and results of selected devices" in preparation.

Next Steps

- Key technologies
  - Continued development of VACNT growth capabilities
  - Continued development of micro-fabrication capabilities
  - Investigate low-noise thermistor fabrication compatible with VACNT growth
  - Continue characterization of optical properties of nanotubes
- Room temperature standard
  - Measure time constants of Prototype 1 and compare with model
  - Measure temperature readout noise of Prototype 1 and compare with component-level measurements
  - Modify heat link design based on measurements and modeling
  - Build and test Prototype 2 with VACNTs

Space Qualification of VACNTS

All measurements to date show that VACNTs are safe and compatible with space flight

- National Physical Laboratory (NPL), Surrey NanoSystems, EnerSys ABSI:

"VANTA coatings are chemically inert, have a high resistance to vibration and shock, and exhibit excellent environmental, thermal and outgassing stability. VANTA coatings are, therefore, good candidates for Earth Observation applications and in particular as coatings for blackbody cavities and baffles."[1,2]

- Applied Physics Laboratory (APL), L-1 Standards and Technology, Blue Canyon Technologies (RAVAN mission)

Vibration test of VACNTs showed no change, https://esto.nasa.gov/forum/estf2016/PRESENTATIONS/Swartz_A6P4_ESTF2016.pdf

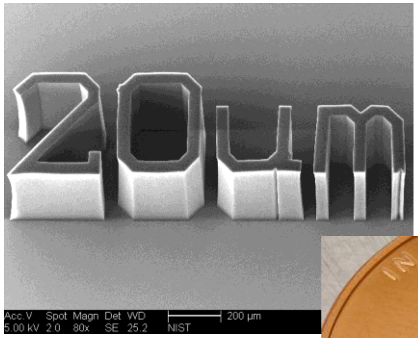
- Ball Aerospace, General Nano – Compact Infrared Radiometer in Space http://www.veelotech.com/news-feed/2016/5/12/nasa-satellite-manufacturer-select-veelo-blac-for-prestigious-invest-program
- PTB – Release rate tests of single CNTs and agglomerate from CNT coated surfaces with a measured with a very high sensitivity and found to be below asbestos fiber threshold values
- NIST "paint shaker test" - https://www.youtube.com/watch?v=2t4VQgB9VN0
- Laboratory for Atmospheric and Space Physics (LASP) – plans for tests as part of IIP/ACT work

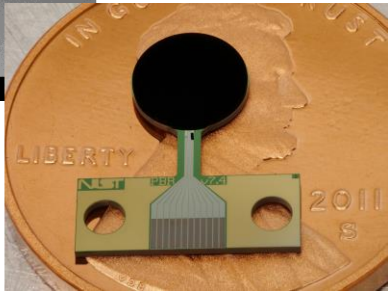
[1] ***VANTA = Vertically Aligned Nanotube Array***

[2] ***Theocharous, E. et al., "The partial space qualification of a vertically aligned carbon nanotube coating on aluminium substrates for EO applications", Opt. Exp. 22 (2014), 7290.***

Summary

- New family of micro-fabricated carbon nanotube laser radiometric standards are under development at NIST
- Advancing technical capabilities
  - Impact of process parameters on VACNT growth
  - Integration of VACNT growth with micro-electronics
  - Optical characterization of carbon nanotubes
  - Low-noise thermistors/temperature measurement
  - COMSOL modeling of multi-layer chips





- Applications to room temperature and cryogenic operation
  - Please attend “Characterization of New Planar Cryogenic Radiometric Standards under Development at NIST”, Malcolm White, Thursday morning in Advancements in Radiometric Calibration State-of-the-Art session
- Applicable to incoherent radiation and space missions
- Interested in new collaborations and applications