Low Power, Wide Dynamic Range Carbon Nanotube Vacuum Gauges

Dr. Anupama B. Kaul & Dr. Harish M. Manohara

Jet Propulsion Laboratory
California Institute of Technology
Anu.Kaul@jpl.nasa.gov

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Personnel

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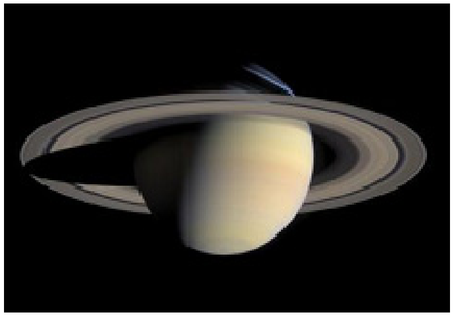
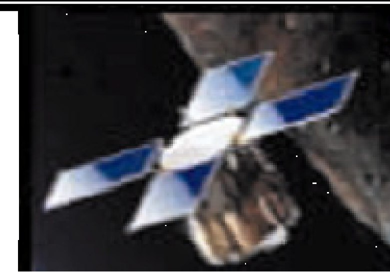
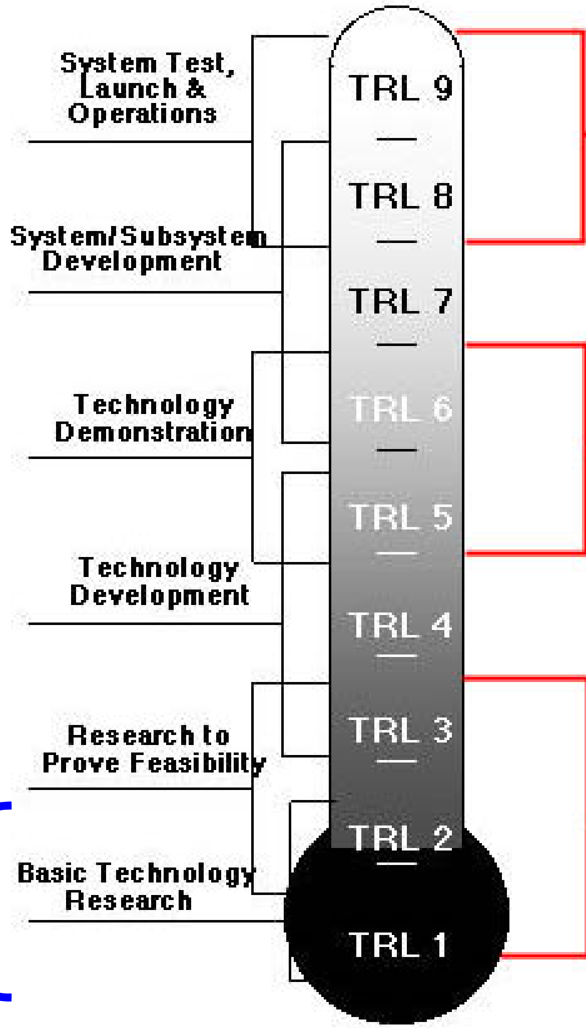
NASA Technology Development







Flight technologies

Mid-level technologies

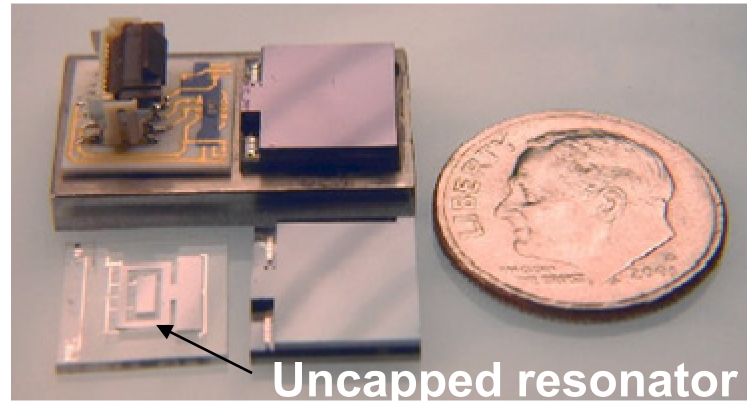
Early level technologies

This work:
Feasibility study of CNT-based vacuum gauges

Vacuum Gauge Applications

Vacuum-Packaged Structures

- Vacuum electronics: MMW and THz vacuum electronics (e.g. amplifiers, oscillators, traveling wave tubes)
- Microgyroscopes: e.g. as resonators
- RF MEMS: e.g. switches, filters

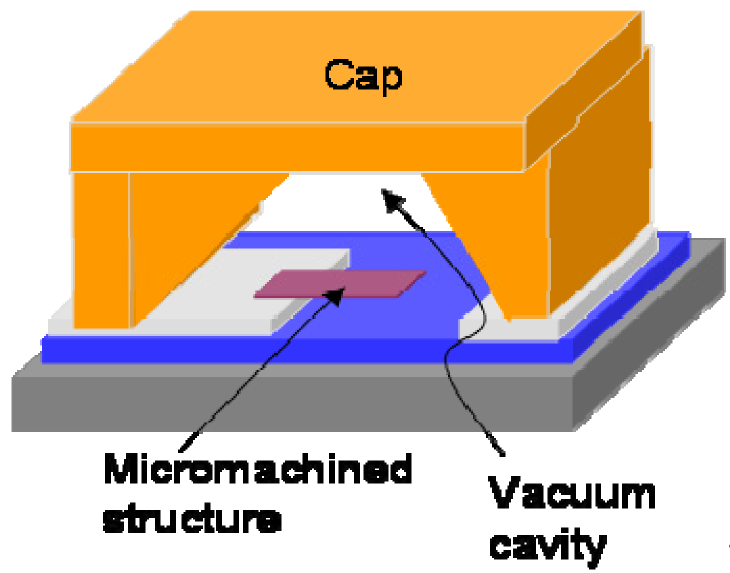


Sparks, Ansari, Najafi,
IEEE Trans. Adv. Packaging, vol. 26, 2003

Vacuum Micro-cavities

- Device performance within micro-cavity usually affected by quality of vacuum
- Small-volumes → large pressure changes from thermal excursions and gas desorption
- Miniature vacuum gauges integrated with micro-cavities can non-invasively monitor local pressures to characterize device performance over product lifetime

Vacuum Micro-cavity



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Conventional Pressure Sensors

Thermal Conductivity Gauges

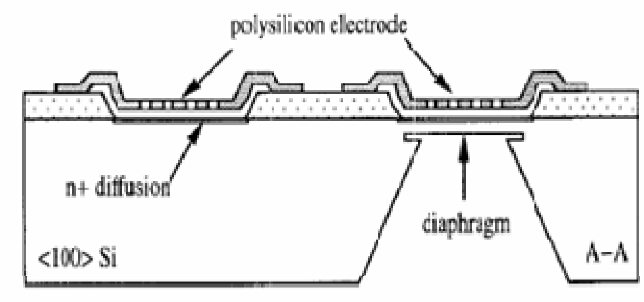
- Examples: Pirani, ion gauges
- Sensitivity good at UHV conditions
- Large volume, high power: invasive
- Difficult to integrate with micro-cavities

MEMS-based pressure sensors

- Mechanisms: capacitive, piezoresistive, optical determination of membrane deflection, resonance frequency shift, alpha particle source, radio-isotope based pressure sensors
- Low power, small volumes
- Wide-dynamic range challenging

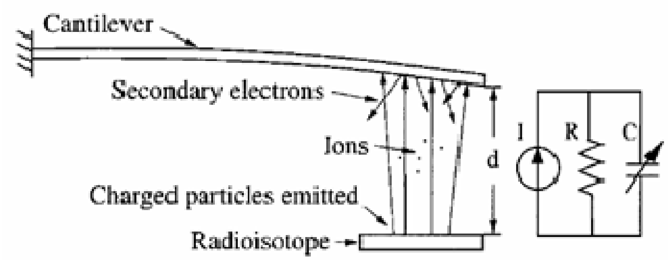
Proposing: Carbon nanotube based thermal conductivity vacuum gauge

Examples: diaphragm-based for capacitive, piezoresistive sensing



Mastrangelo, Zhang, Tang, *J. MEMS*, vol. 5, 1996

Recently: Radio-isotope charged cantilever for sensing



A. Lal *et al. J. Appl. Phys.* vol. 92, 2002.
H. Li and A. Lal, *Proc. of the 12th Int. Conf. on Solid State Sensors, Actuators and Microsystems*, vol. 1, 2003.

Carbon Nanotube Pressure Sensors

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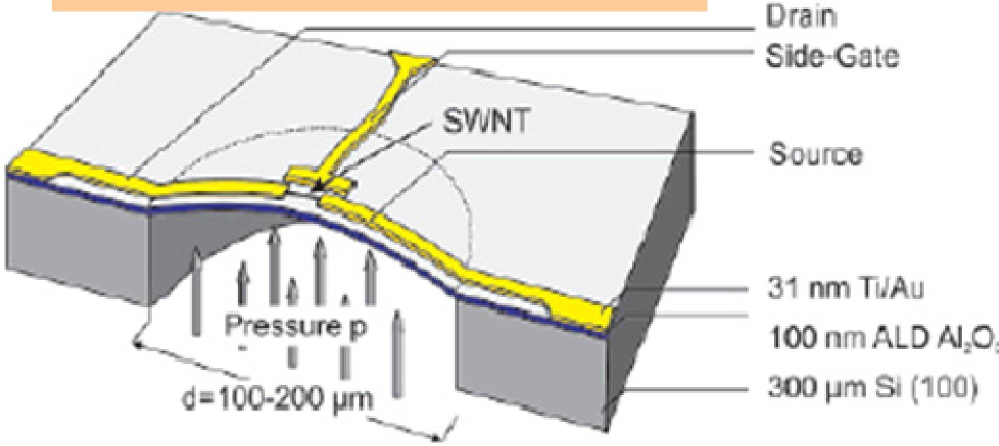
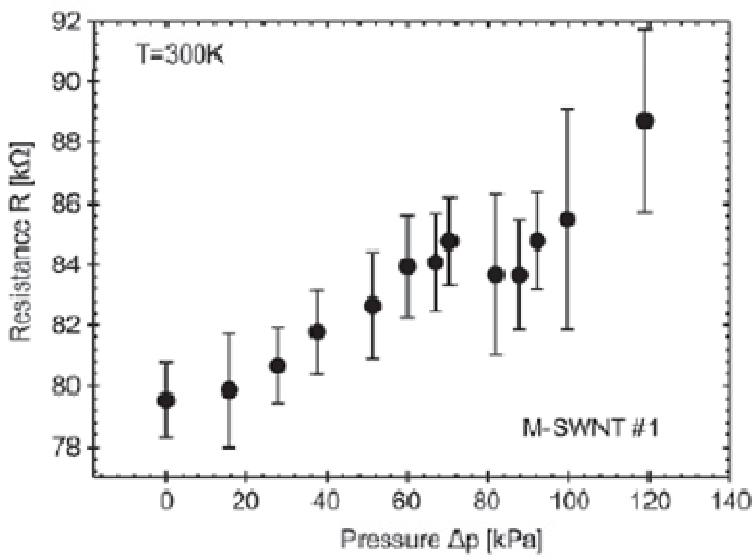
Piezoresistive Mechanism





C. Stampfer, C. Hierold, *et al. Nano Lett.*, vol. 6, 2006.

Piezoresistive Mechanism

- Metallic SWNTs on ALD-deposited membranes of Al_2O_3
- Pressure differential causes membrane to bulge, inducing strain in overlying SWNT

This Work: Thermal Conductivity Mechanism

- Heat transferred to gas function of pressure for sensor held at fixed bias

$$E_{Total} = E_{substrate} + E_{radiation} + E_{gas}$$

- Small dimension of CNT and large TCR values enables greater pressure sensitivity
- Non-intrusive: low power, small-size, promising for micro-cavities

Device Fabrication Facilities

JPL's Microdevices Laboratory: 12,000 square feet class 10 cleanroom

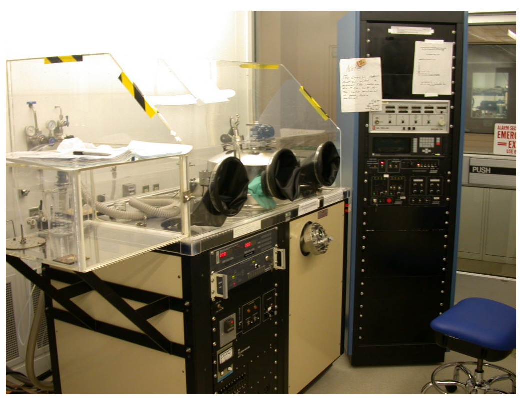
Contact lithography units

JEOL E-beam aligner
Canon Excimer Laser DUV Stepper

Conventional
RIE etchers (CF_4/BCl_3)

ICP etchers: high density, low pressure plasma (chlorine and fluorine chemistries)

Dektak profilometer

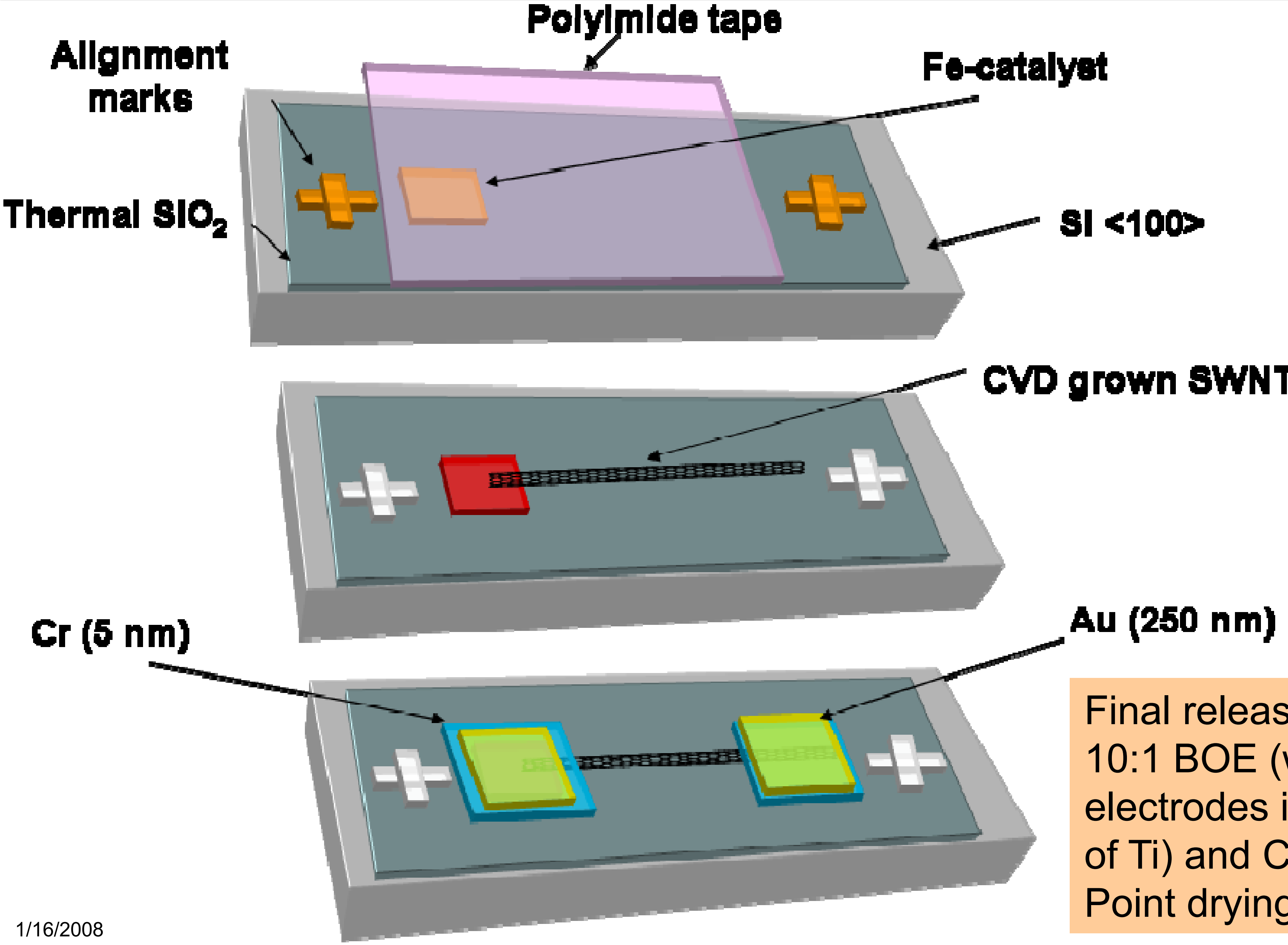


E-beam evaporator (Pt, Ti, Au electrodes)

Other key equipment:

-dc magnetron sputtering system
-CVD furnace (CNT growth)
-Fe e-beam evaporator (catalyst layer)
-PECVD dielectric deposition
-AFM, SEM (CNT imaging, characterization)

Formation Process



Final release in 10:1 BOE (with Cr electrodes instead of Ti) and Critical Point drying

Catalytic CVD Growth of CNTs

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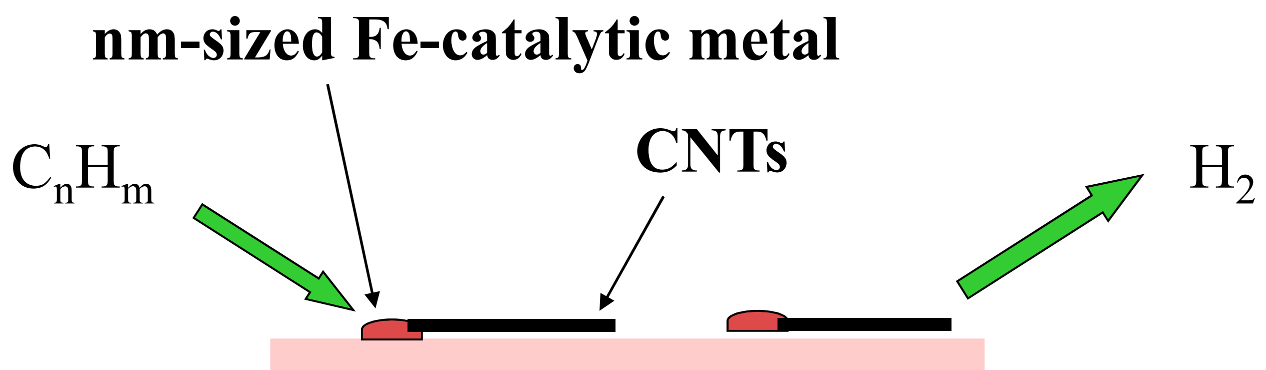
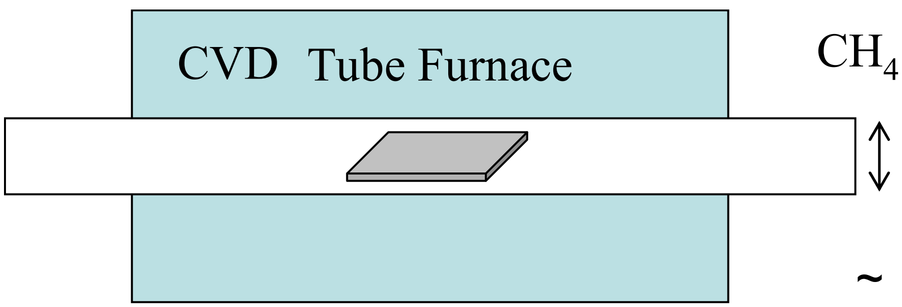
High solubility of C in catalyst at high temperatures

$$\text{Hydrocarbons } (C_nH_m) \xrightarrow[\text{600-800 C}]{\text{Fe (or Ni, Co, etc.)}} C(s) + H_2$$



SWNT Synthesis Conditions

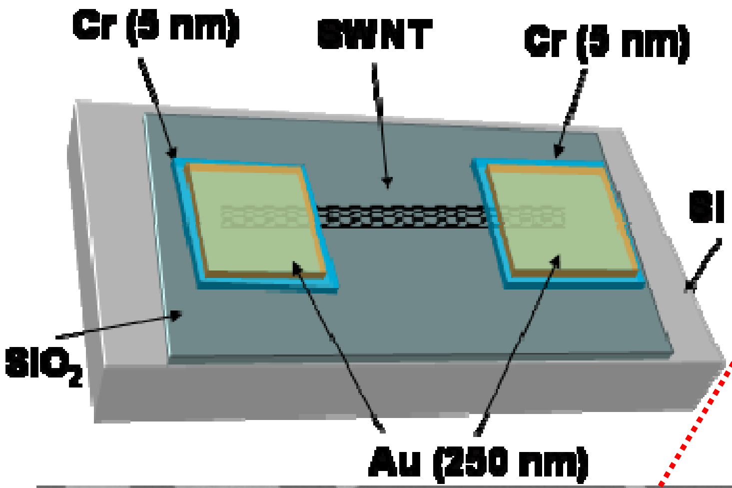


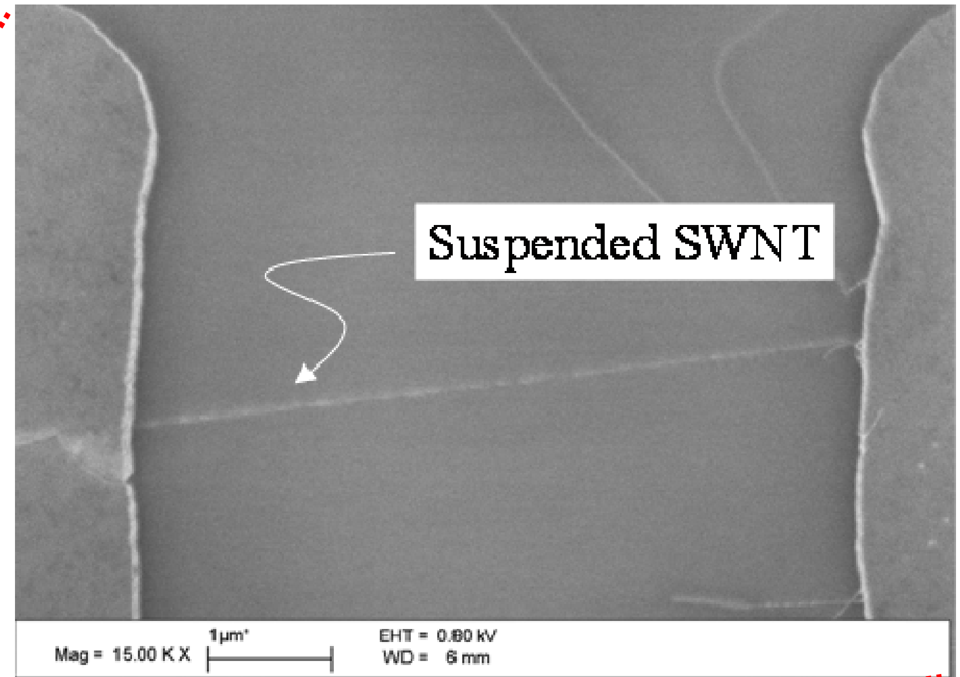
CH_4 1500 sccm & H_2 50 sccm @ 850 ºC

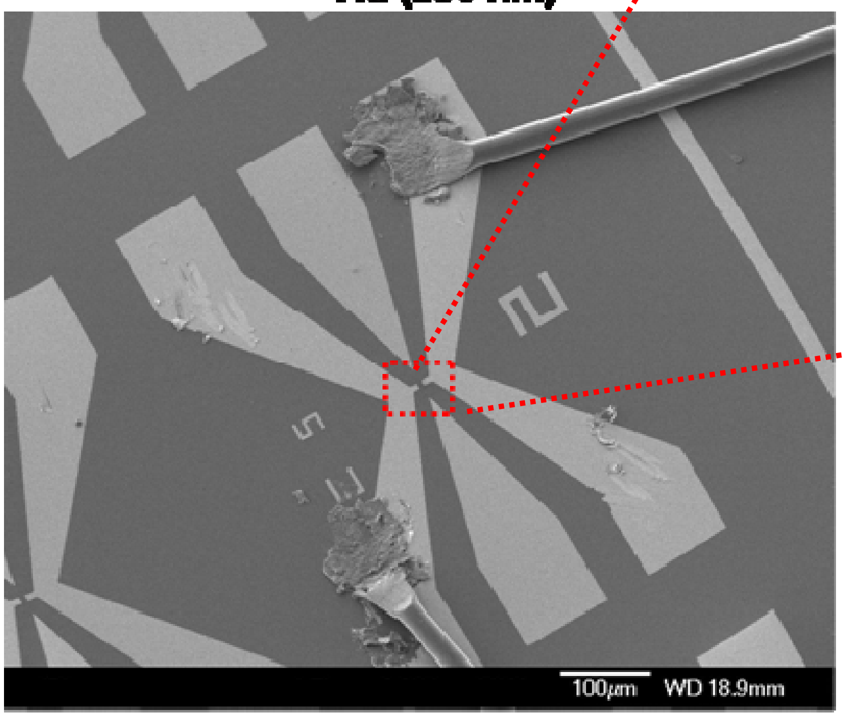
2” Quartz Tube

~ 1 nm thick Fe-catalyst film

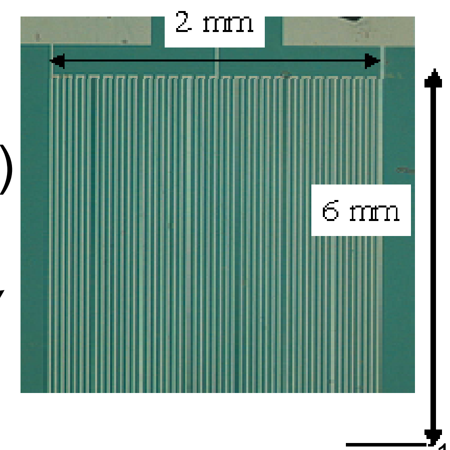
Fabricated Devices







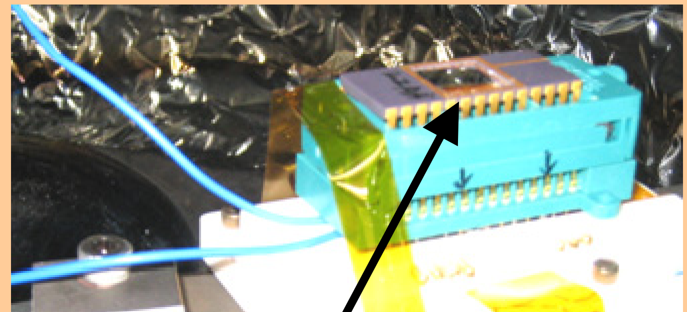
Au/Ti (35 nm/ 2 nm) thin film meander fabricated for comparison



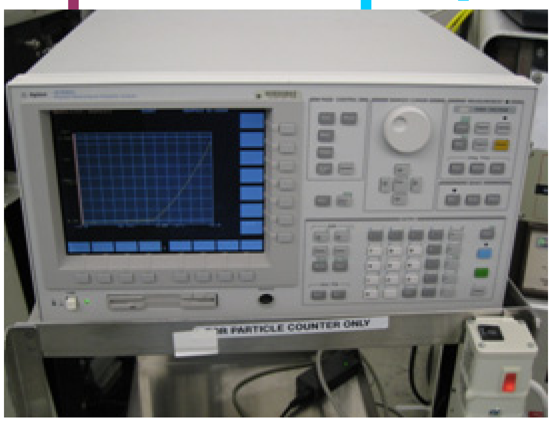
Measurement Set-up

Computer interface to parameter analyzer using ICS data acquisition software; current sampled at 1 sec intervals at fixed bias voltage

Bell-jar



Devices wire-bonded on chip-carrier



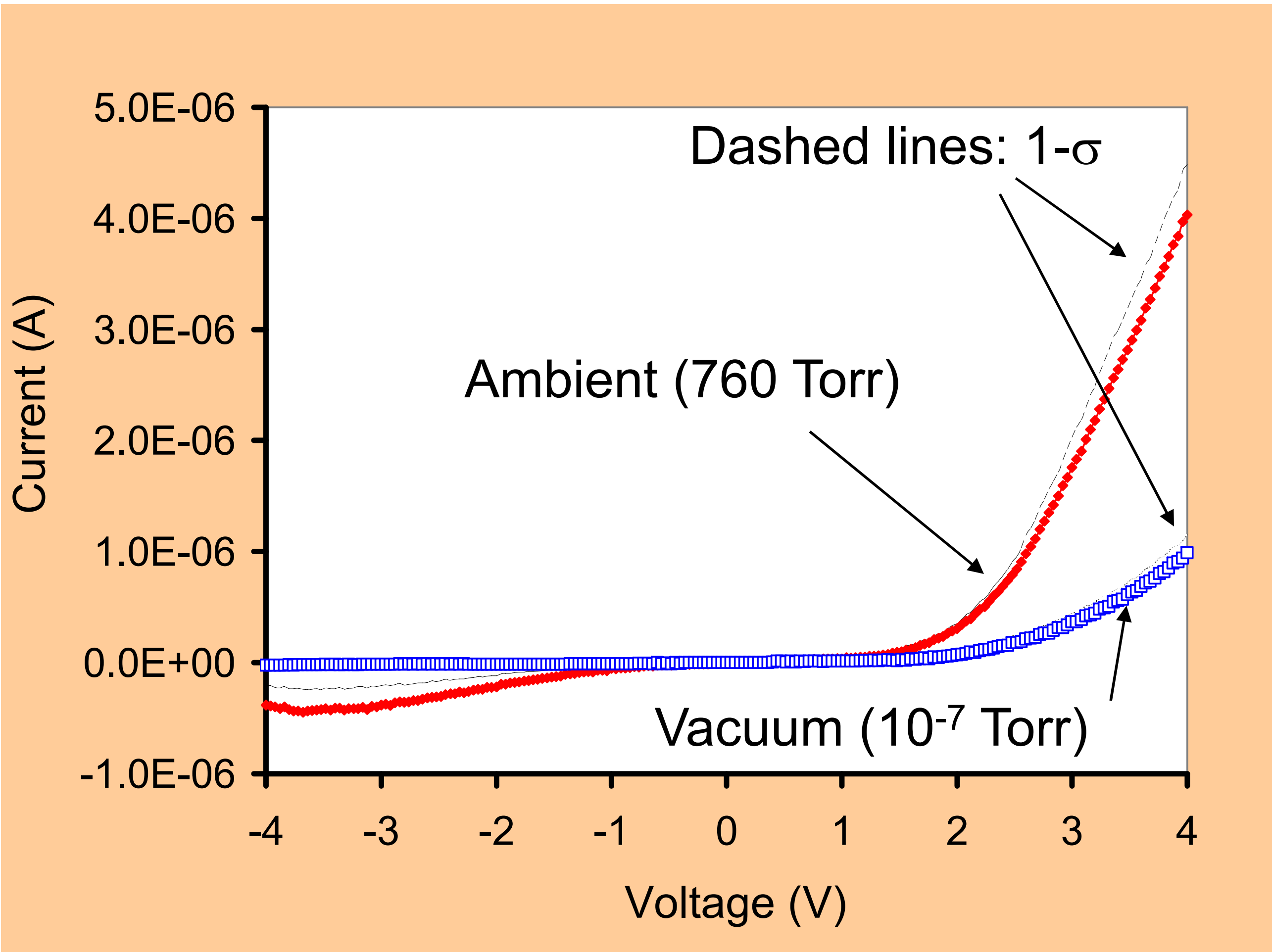
HP 4156C Parameter Analyzer

coaxial cables

Electrical feed through for 2-terminal measurements

- Wheatstone bridge not required, sensitivity ~10 pA on analyzer
- Mechanical pump (760 Torr – 35 mTorr); pressure measured with convectron gauge
- Ultimate pressure ~ 10^{-7} Torr (cryo pump); pressure measured using ion gauge

Static Pressure Response



- Reduced conductance at low pressures
- Change in I detected from 760 Torr – 10^{-7} Torr
- Good repeatability
- Stable over weeks, contacts do not change irreversibly despite high resistances
- Earlier Ti-contacted CNTs less stable with pressure cycling (possibly due to propensity of Ti for oxidation)

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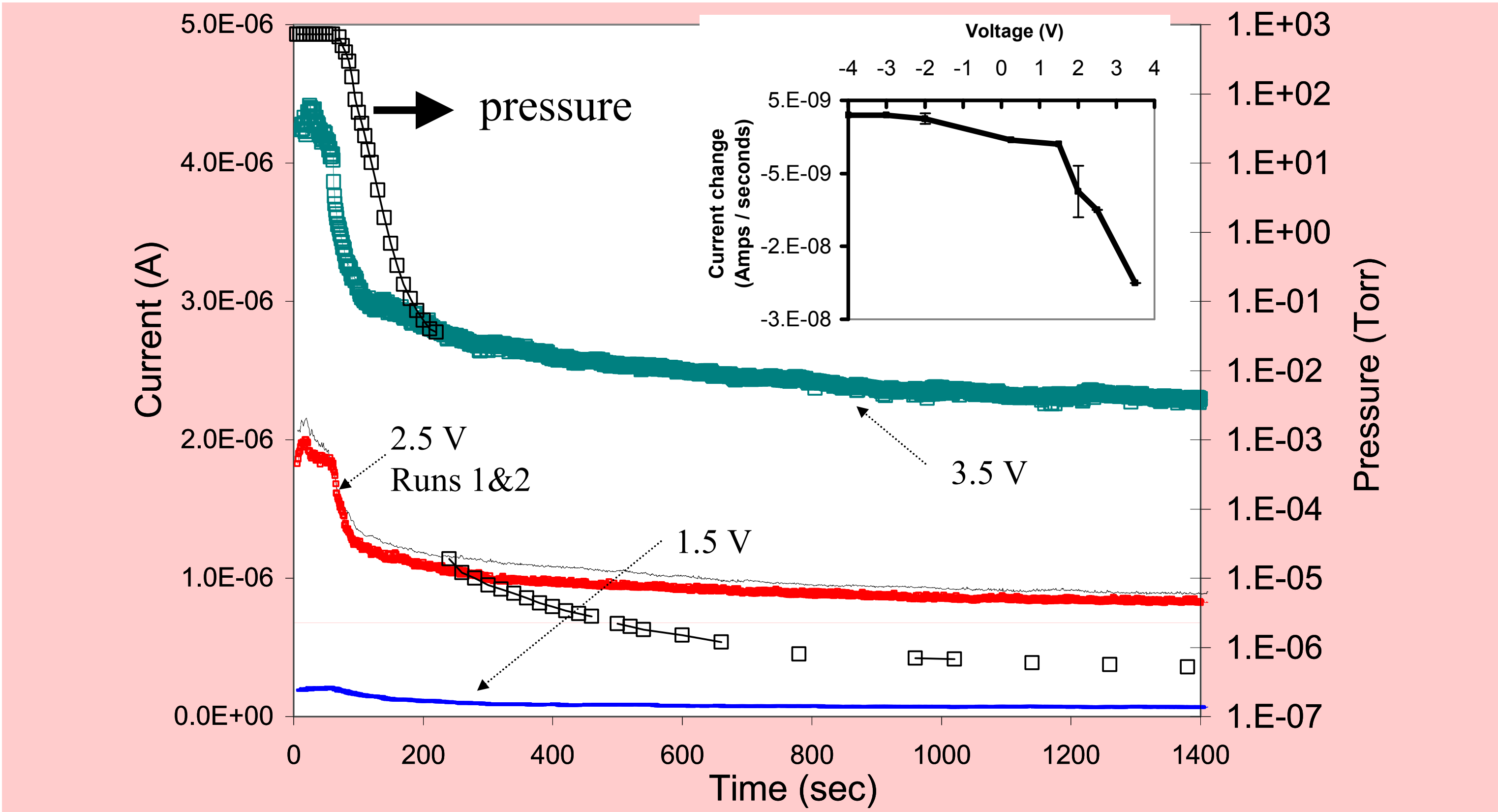
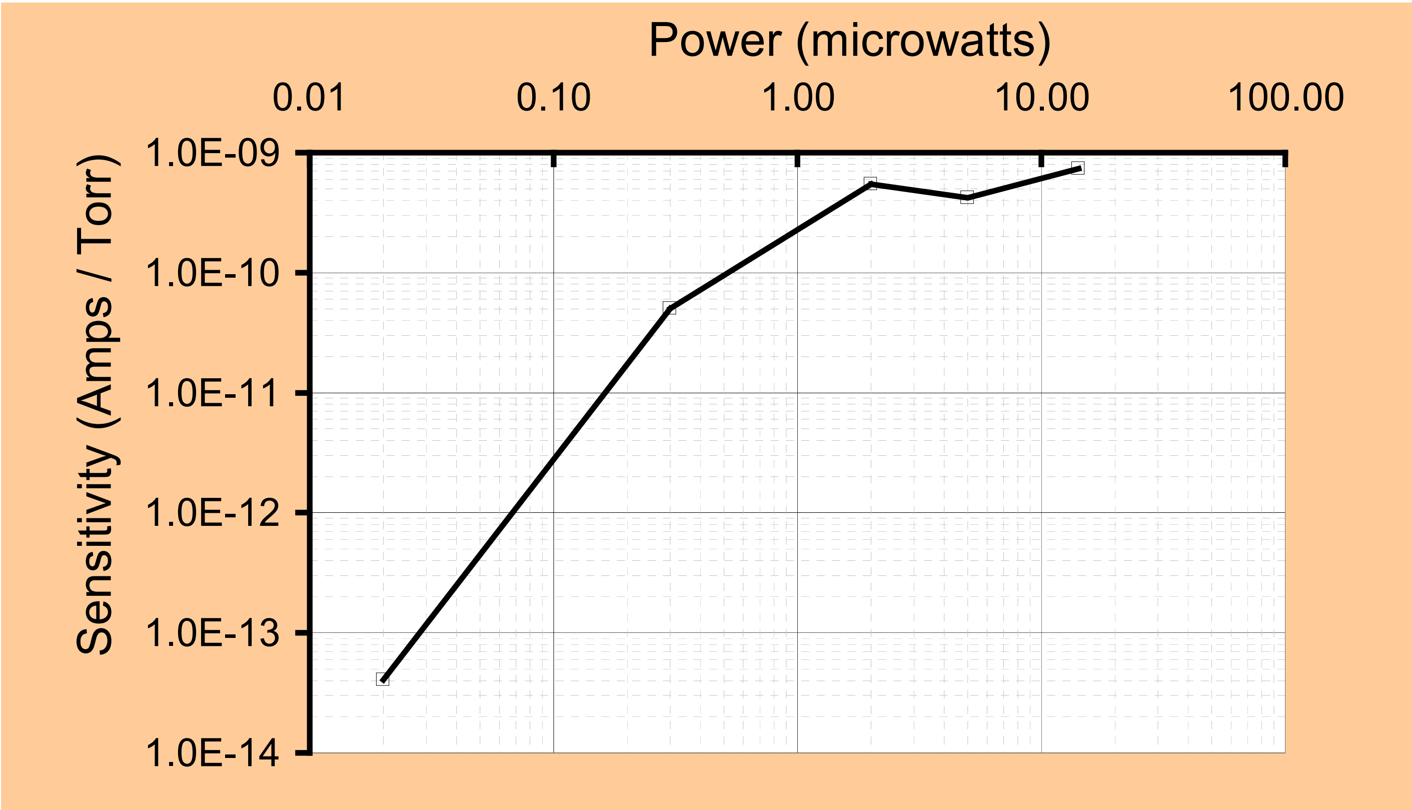
Dynamic Pressure Response



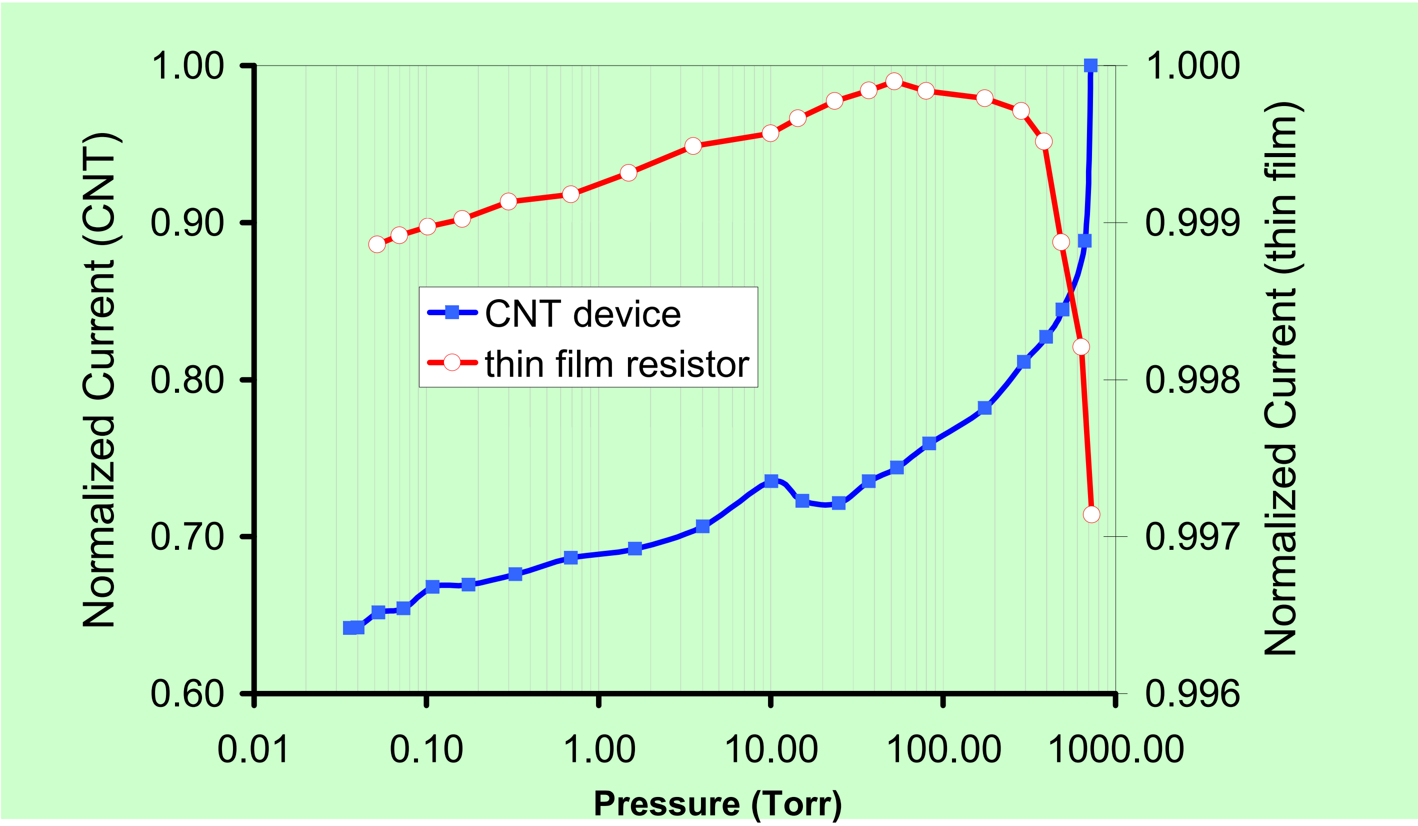
- Conductance decreases rapidly initially (760 Torr - 1 Torr)
- Response less sensitive at lower pressures; molecular collision rates higher at higher pressures → greater cooling, greater current change
- Cooling causes current to decrease as a result of –ve TCR
- As bias voltage increased, rate of current change increases in linear regime (inset)

Pressure Sensitivity with Power



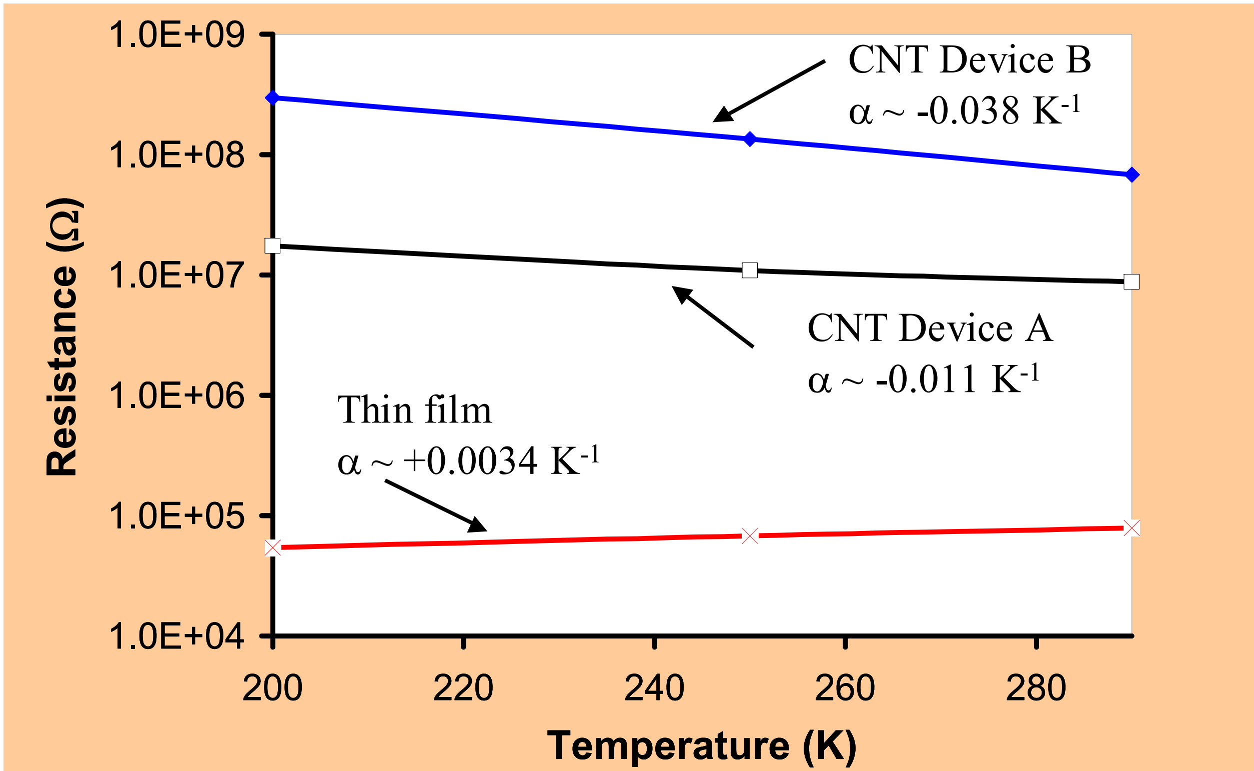
- Sensitivity increases with power (temperature changes higher)
- At powers as low as 20 nW, measurable sensitivity ~ 40 fA/Torr
- Increases up to ~ 1 nA/Torr at ~ 14 μW
- Earlier thermal conductivity gauges show similar behavior but at > mW-levels of power

Comparison to Thin Film Meander



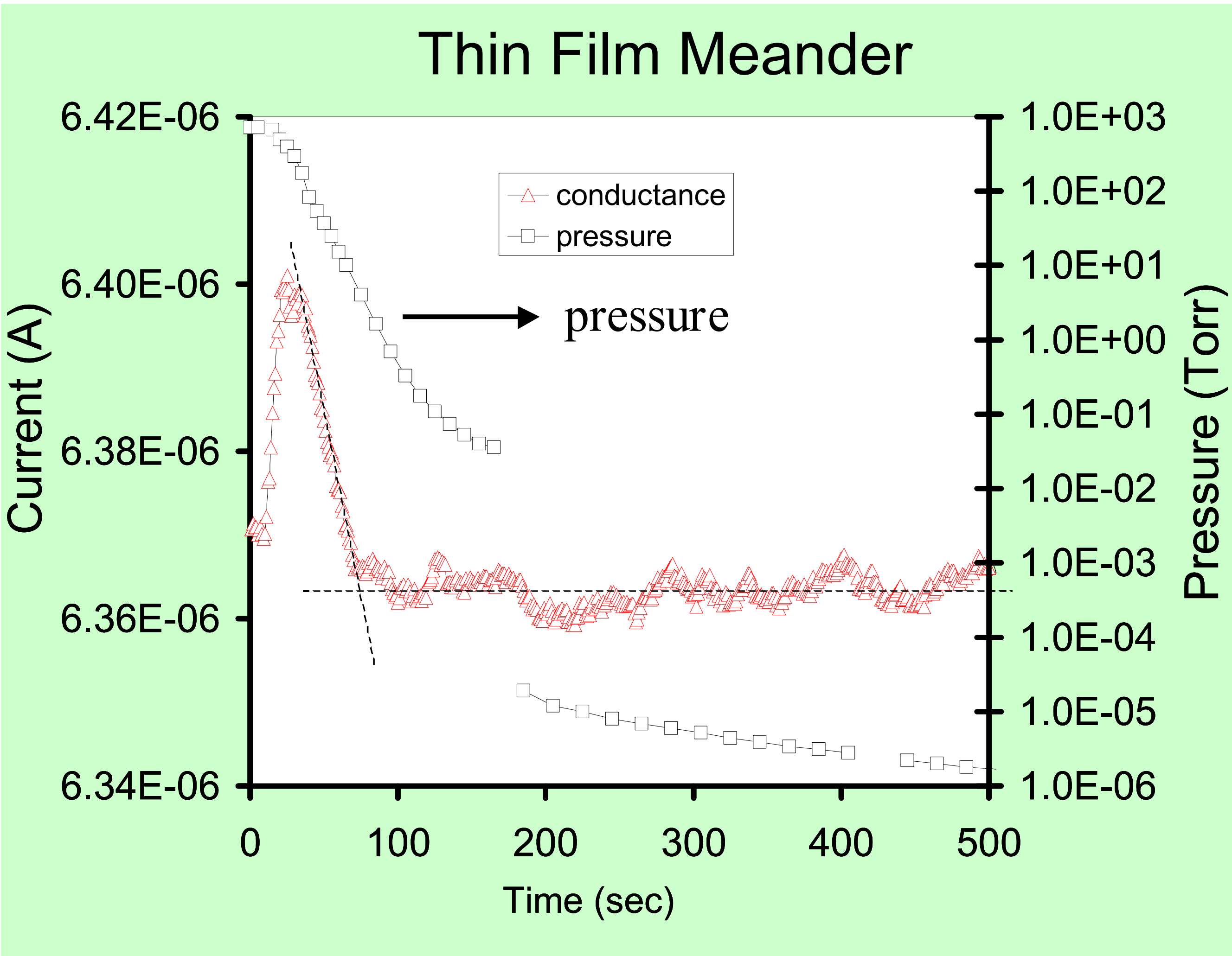
- Thin film resistor and CNT device biased at a few watts of power
- Thin film resistor < 1% change in current and has 3 regimes:
  a) Current increases from 760 Torr to ~ 200 Torr, b) plateaus, and c) decreases below about 20 Torr
- CNT device: current decreases down to ~ 40 mTorr with ~ 35% change

Temperature Coefficient of Resistivity



- TCR of thin film resistor +ve: cooling during pumpdown → R ↓ & I ↑
- TCR of CNT devices –ve: opposite pressure response, as observed
- CNT device has a higher surface area to volume ratio, more sensitive
- Magnitude of TCR also higher for CNT devices → enhanced sensitivity
- Prior work indicates large variation in TCR of CNTs (experimentally and theoretically)
- Large TCR could be attributed to tunnel barriers at contacts, defects in tubes

High Vacuum Regime: Meander



- Sensitivity disappears after ~ 90 sec or ~ 10 Torr due to constant background signal.
- These devices operated at low power (< 10's μW)
- Radiative losses dominant at higher temperatures (> 200 deg. C).

High Vacuum Regime: CNT Gauge

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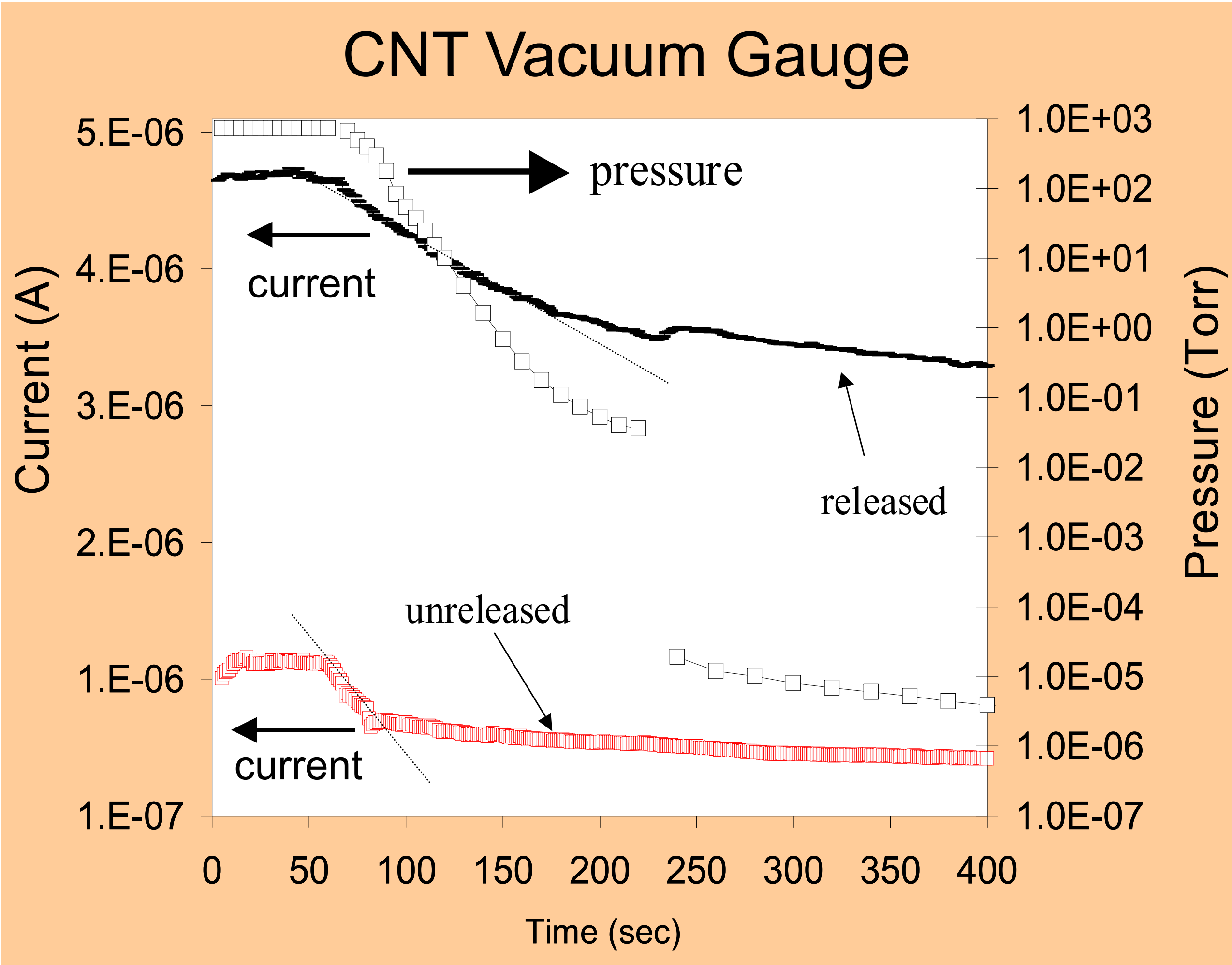
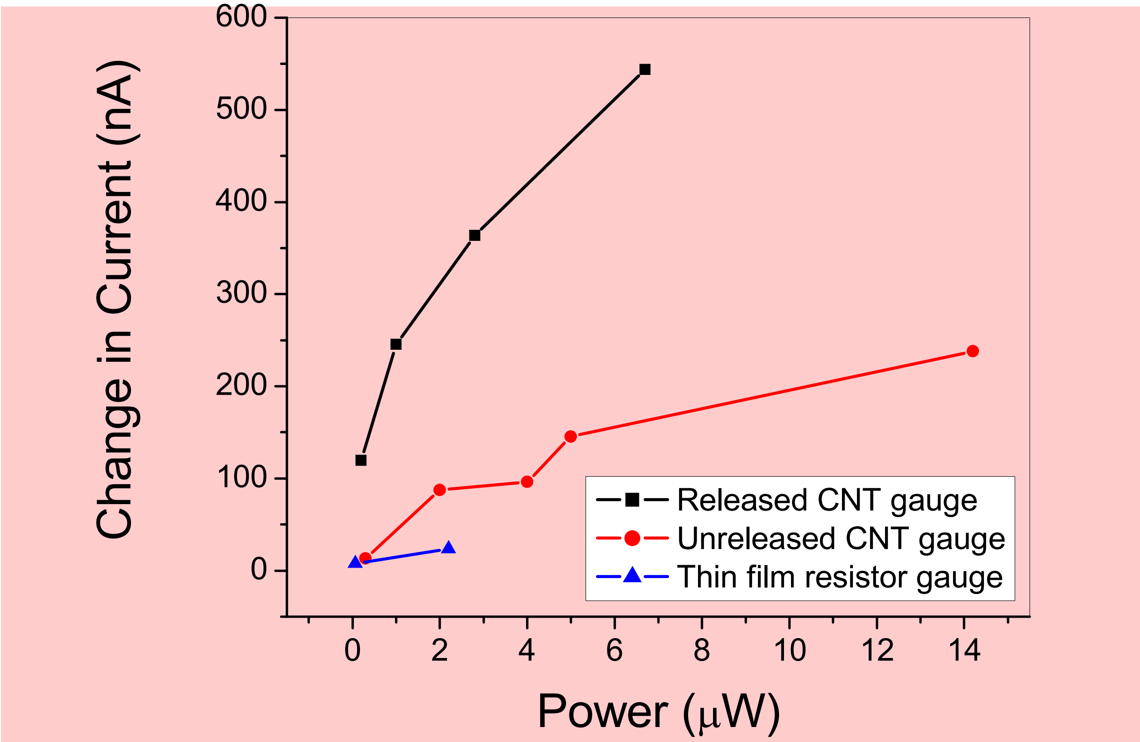
$$E_{Total} = E_{substrate} + E_{radiation} + E_{gas}$$



- Losses through substrate important at low pressures
- By removing substrate, more heat propagated by gas
- CNT devices released in 10:1 BOE and critical point drying to remove thermal SiO_2 underneath
- Released device continued decrease in current down to 10^{-5} Torr

Effect of Power at High Vacuum



- Effect of power on unreleased and released CNT devices, thin film resistor gauge
- Net current change ΔI measured for pressure change from 5 x 10^{-6} Torr to 8 x 10^{-7} Torr
- ΔI insignificant for thin film meander in this high vacuum regime
- Released CNT device has the highest ΔI ~ 550 nA compared to ~ 150 nA for unreleased CNT device at ~ 6 μW

Summary and Future Work

Summary

CNT vacuum gauges:

• have a broad range of pressure response from 760 - 10^{-6} Torr
• Sensitivity ~ 100's nA in high vacuum regime (10^{-6} Torr) and increases with power and substrate removal
• have –ve and a large magnitude of TCR (up to 0.0038 K^{-1})
• can be operated at low power (nW – μW)
• have an active device region footprint of < 10 μm^2
• are non-intrusive due to small size and passive operation
• have compatible fabrication requirements for their integration with micromachined structures for micro-cavity applications

Future Work

• Further work is necessary to fully characterize effect of tube characteristics (chirality, length, transparency at the contacts) on pressure response of devices

Backups

CVD Synthesis of SWNTs

Role of H_2

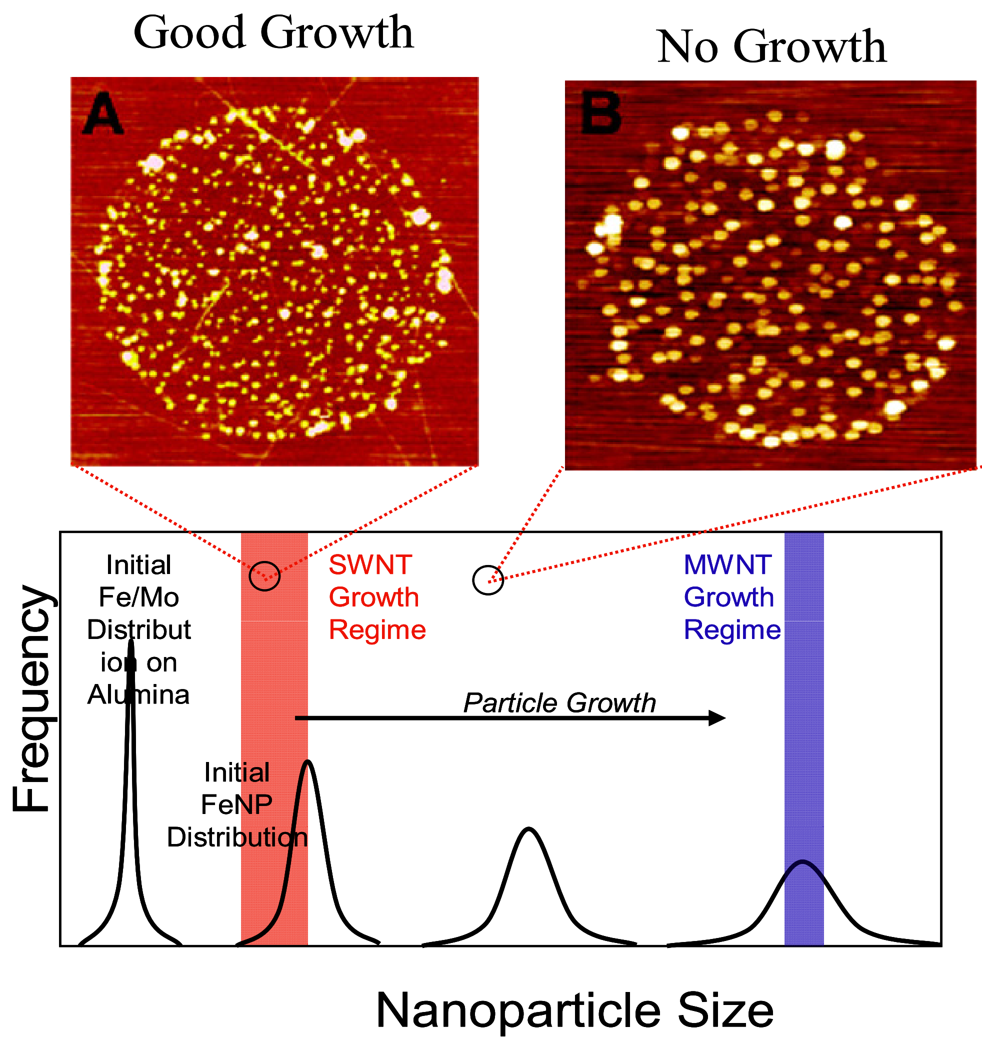
• H_2 required to minimize amorphous carbon, but too much H_2 or hot annealing causes particle fusion and inhibition of SWNT growth

• Particle growth desirable for larger MWNTs

Gas flow dynamics

• Stagnant zone in boats results in variable gas mixing leading to variable SWNT yield

• Flat top holders with laminar flow give consistent results



Wong, E.W. *et al., Chem. Mater.*, **17** (2005) 237-241

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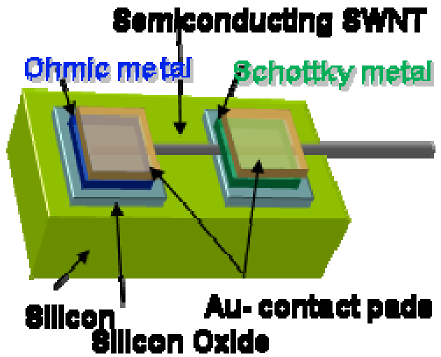
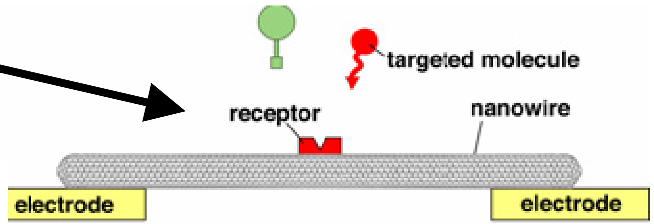
JPL CNT Device Efforts

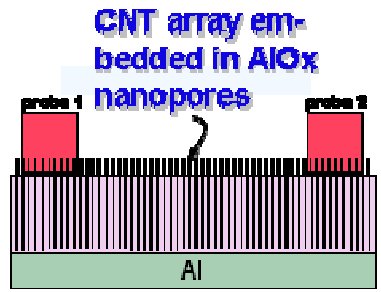
- CNT schottky diodes for THz detectors (H. Manohara, E. Wong et al.)



- CNT field emitters for THz sources (H. Manohara, M. Bronikowski)
- Nanowire-based chemical spectrometer for molecular ID (B. Hunt, E. Wong, M. Bronikowski, et al.)



- CNT mechanical resonators for RF signal processing (B. Hunt, L. Epp et al.)



- CNT switches (A. Kaul, E. Wong, et al.)