Low Power, Wide Dynamic Range Carbon Nanotube Vacuum Gauges

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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

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


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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

Piezoresistive Mechanism

- Metallic SWNTs on ALD-deposited membranes of Al₂O₃
- 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

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 (CF₄/BCl₃)
- Dektak profilometer
- E-beam evaporator (Pt, Ti, Au electrodes)
- ICP etchers: high density, low pressure plasma (chlorine and fluorine chemistries)

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)

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Formation Process

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

High solubility of C in catalyst at high temperatures

Hydrocarbons (C\textsubscript{n}H\textsubscript{m}) \xrightarrow{Fe \text{ (or Ni, Co, etc.)}} C(s) + H\textsubscript{2}

600-800 °C

nm-sized Fe-catalytic metal

C\textsubscript{n}H\textsubscript{m} \rightarrow \text{CNTs} \rightarrow H\textsubscript{2}

SWNT Synthesis Conditions

CH\textsubscript{4} 1500 sccm & H\textsubscript{2} 50 sccm @ 850 °C

\uparrow 2” Quartz Tube

\sim 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

- 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
- 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)
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)
• 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 $\alpha$ +ve: cooling during pumpdown $\Rightarrow$ $R \downarrow$ & $I \uparrow$
- TCR of CNT devices $\alpha$ -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 $\Rightarrow$ 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

\[ E_{\text{Total}} = E_{\text{substrate}} + E_{\text{radiation}} + E_{\text{gas}} \]

- 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\textsubscript{2} underneath
- Released device continued decrease in current down to 10\textsuperscript{-5} Torr
Effect of Power at High Vacuum

- Effect of power on unreleased and released CNT devices, thin film resistor gauge
- Net current change $\Delta I$ measured for pressure change from $5 \times 10^{-6}$ Torr to $8 \times 10^{-7}$ Torr
- $\Delta I$ insignificant for thin film meander in this high vacuum regime
- Released CNT device has the highest $\Delta I \sim 550$ nA compared to $\sim 150$ nA for unreleased CNT device at $\sim 6 \mu W$
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 μm²
• 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
Role of H₂
• H₂ required to minimize amorphous carbon, but too much H₂ 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

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.)