# Engineered Carbon Nanotube and Graphene for Sensors, Actuators and Nanoelectronics

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Stevens Institute of Technology

# Outline

I. Carbon Nanotube

1. Quantum Dots for RT SETs
2. Nanoactuator

II. Graphene

1. Electron Gyroscope
2. Field Emission

# Nanoelectronics Team

- Dr. Frank Fisher
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  Assistant Professor
  Department of Physics
  Stevens Institute of
  Technology

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  Moscow

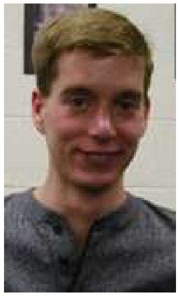
- Students
  - Kitu Kumar, Stevens (ME)
  - Yao-Tsan Tsai, Stevens (ME)
  - Nan Ai, Stevens (Physics)
  - Qiang Song, Stevens (Physics)
  - Erin Lynn Cochran, UI

# Nanoactuator, E-Gyro, FE Teams

- Dr. Seung Seob Lee
  Professor
  Department of Mechanical Engineering
  KAIST

- Dr. Stefan Strauf
  Assistant Professor
  Department of Physics
  Stevens Institute of Technology

- Dr. Chris Search
  Assistant Professor
  Department of Physics
  Stevens Institute of Technology



- Postdocs/Students
  - Dr. Onejae Sul, Stevens (ME)
  - Dr. Seongjin Jang, Stevens (ME)
  - Kitu Kumar, Stevens (ME)
  - Dr. Seok Woo Lee, KAIST

# Acknowledgements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- US Army Armaments Research Development and Engineering Center
- Exchange student program by Brain Korea 21
- Stevens Startup funds

MECHANICAL ENGINEERING
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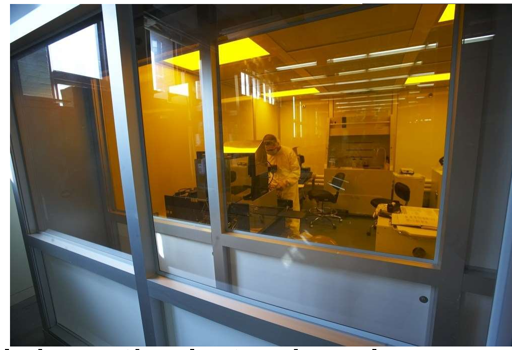
# Stevens Micro Devices Laboratory

STEVENS
Institute of Technology

Director: E. H. Yang

Core faculty members: C.H. Choi, F. Fisher, S. Manoochehri, K. Pochiraju, and Y. Shi

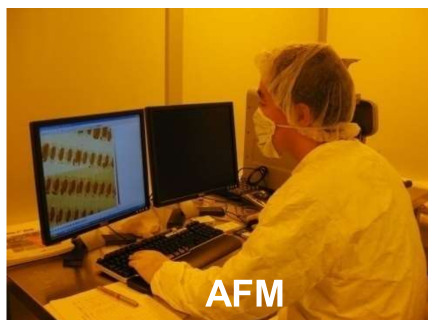
- Initial funding provided by US Army/ARDEC
- Additional equipment funding:
  - AFM, thermal evapoartor (internal funding)
  - NSF MRI: Nanomanipulator/SEM (awarded; PI Fisher)
  - NSF MRI: ICP etcher (awarded; PI Shi)




Gown room

- Stevens faculty, students, and researchers work collaboratively to develop novel devices in the areas of MEMS and nanotechnologies.


Photo room

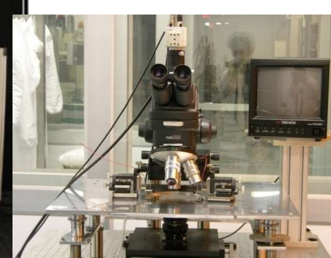

AFM


SEM/Nanomanipulator


Mask Aligner (MA6)


Spin Coater


ICP Etcher


Probe Station


XeF2 etcher


Deep Reactive Ion Etching

# I-1. Carbon Nanotube: Quantum Dots

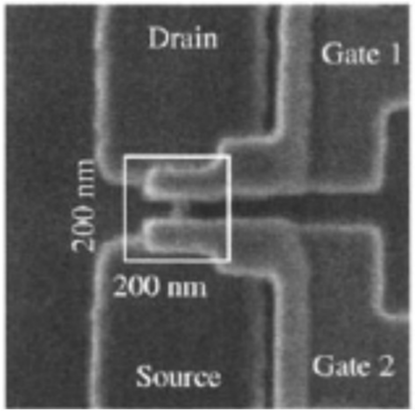
- Nanoelectronics: Working at the length scale of 1-100 nm, in order to create materials, devices and systems with fundamentally new properties and functions because of their nanoscale size. (www.nano.gov)

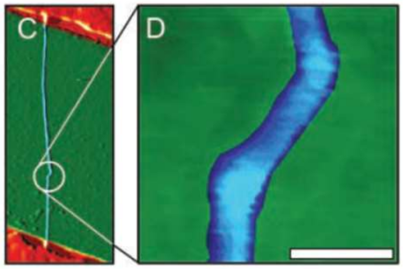
- Low dimensional materials such as Carbon Nanotube (CNT) and Graphene are considered strong candidates.
- Single Electron Device controls the movement of individual electrons; comprise quantum dot (QD) and tunnel junctions.

# Why CNT SET?

$$E_c = \frac{e^2}{C_\Sigma} + \Delta E_L >> KT$$

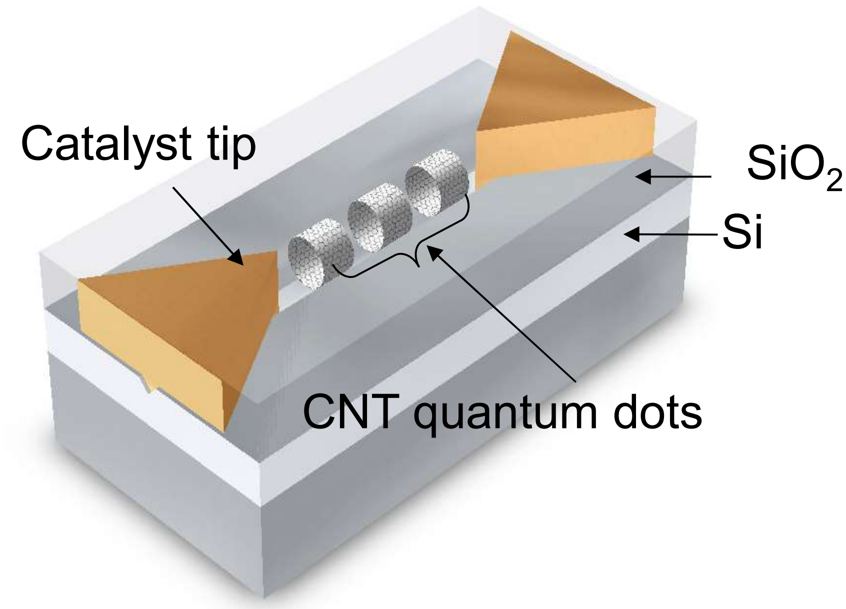
- For practical room-temperature operation ($\sim 10kT = 259$ meV), $d$ should be ~1nm for Si.
- **Core of the problem:** Charge and island size fluctuation ➔ The larger the island AND the larger the charging energy, the more effectively will spurious charges be suppressed.
- **In CNT, confinement effects occur effectively at much large feature sizes (<30nm).**

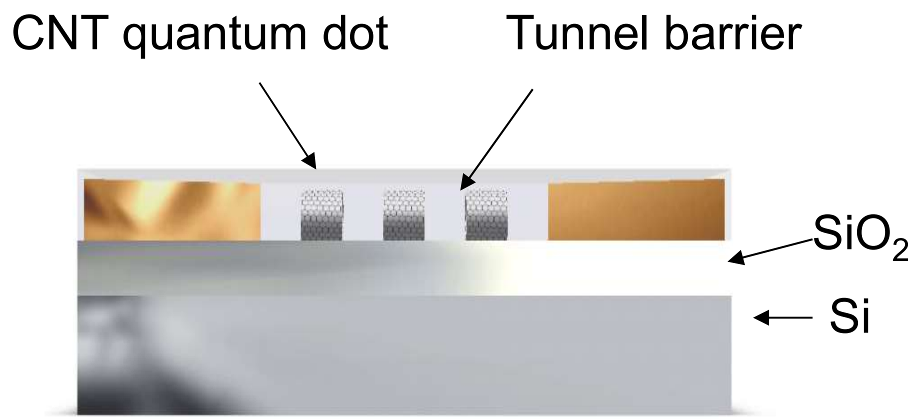



Henk W. Ch. Postma, *et al.* *Science* **293**, 76 (2001);

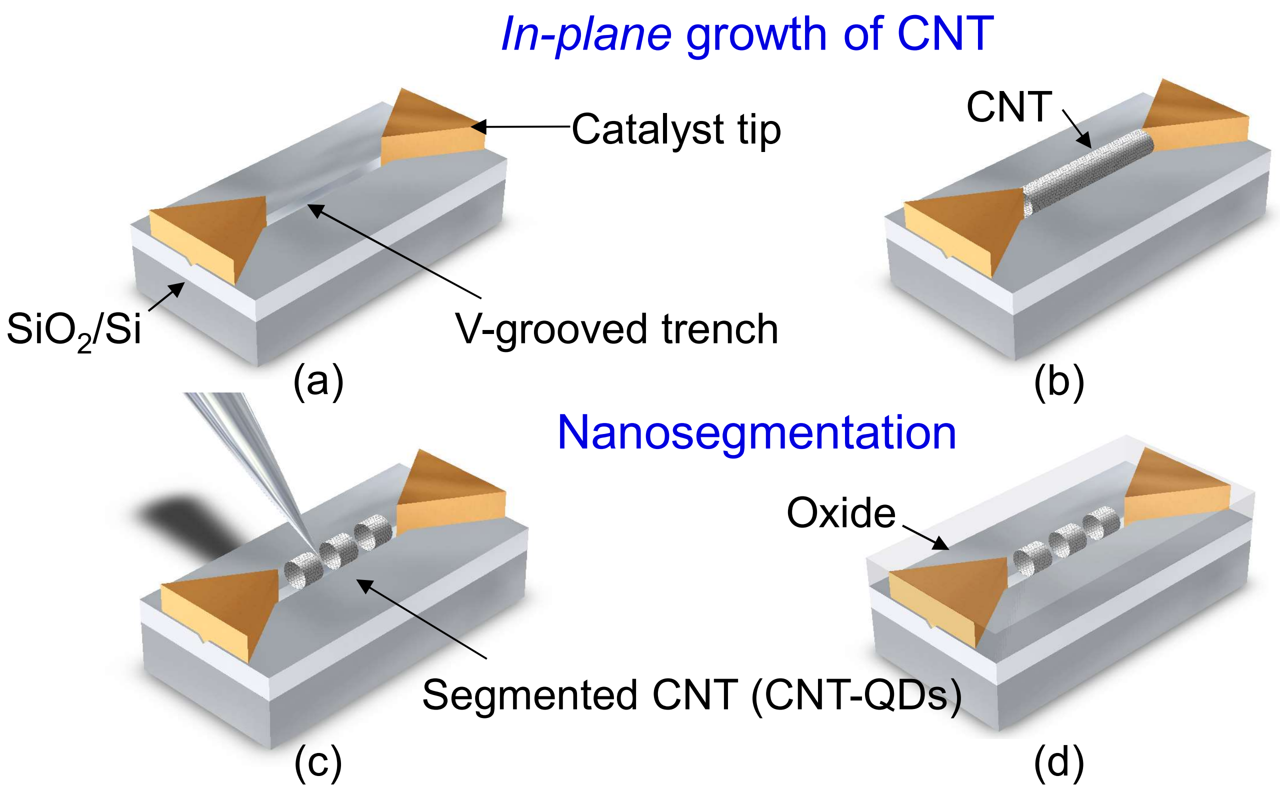
# CNT QDs



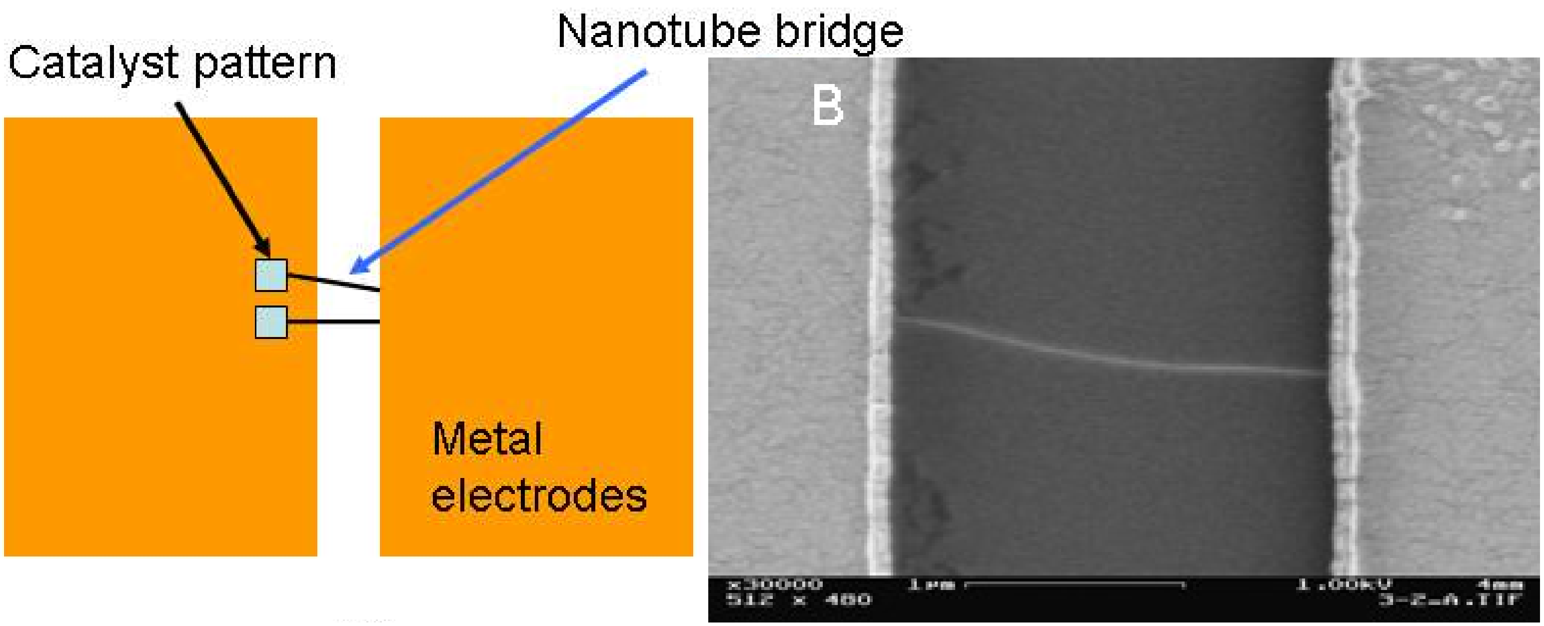

- Near-term goal: CNT-based single electron transistor, stable up to room temperature by creating CNT quantum dots via a controlled growth followed by nanosegmentation process.

# CNT-QD Fabrication Process



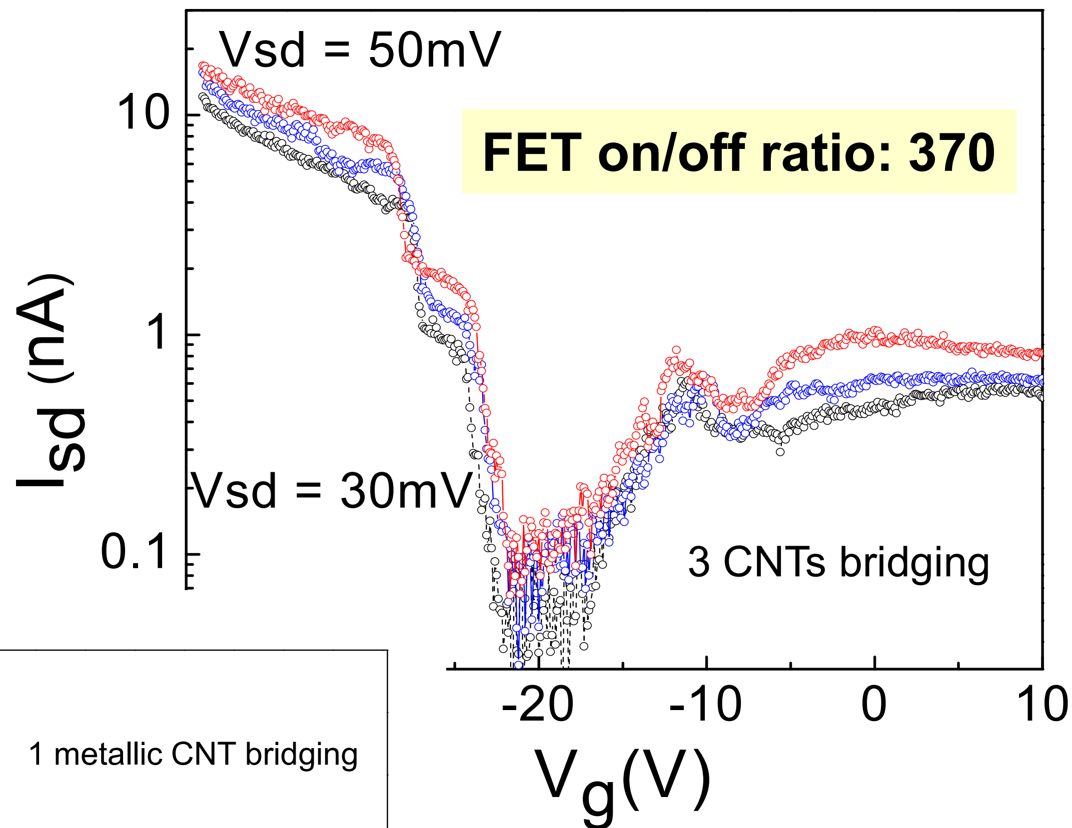

(a) The V-grooved nano-trench generated. Catalyst patterns defined
(b) CNT grown from the catalyst tip along the trench
(c) CNT segmented by VAFM

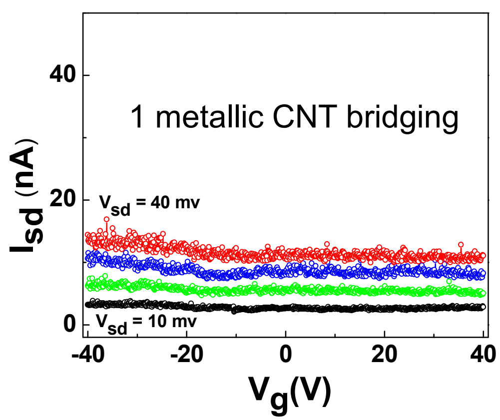
# Growth of In-Plane CNTs




- Substrate: 200 nm $SIO_2$/Si
- Growth conditions:
  - Pressure: 10 mTorr, Gas mixture: $CH_4/Ar_2$ (1:4)
  - Temperature: 850$^o$C, Growth time: 10 min

# Semiconducting SWNTs



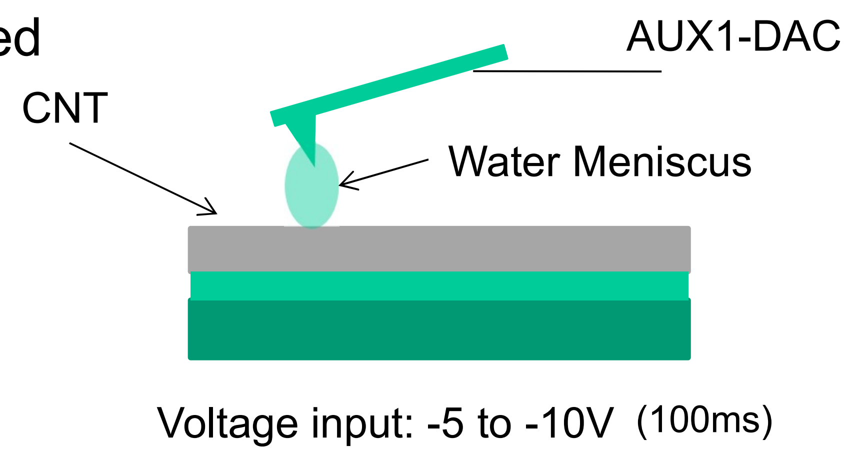




CNTs with pronounced semiconducting behavior

➔ 1st step towards exploiting CNT quantum dots with the goal to realize single electron device elements at room temperature.

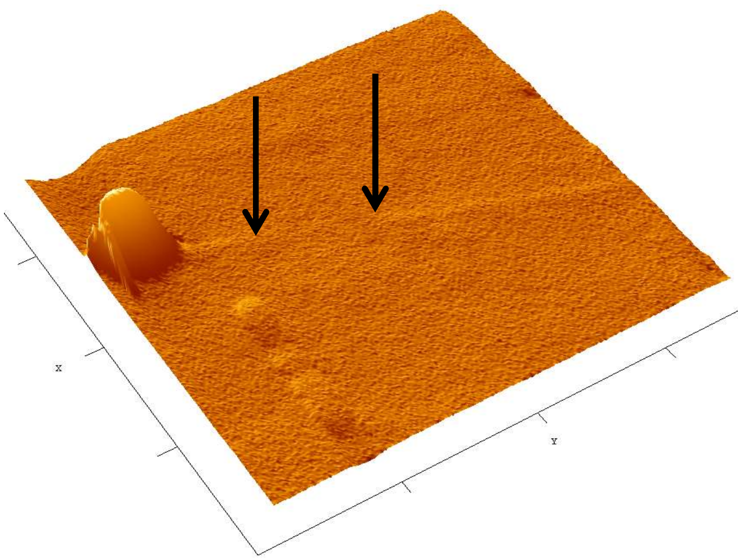
# CNT Segmentation

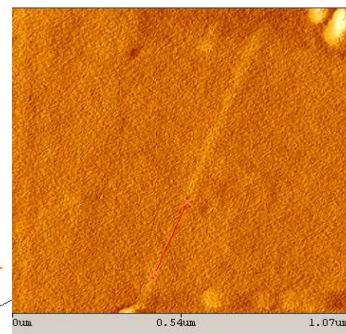
- ***Voltage-applied AFM (VAFM) process***
  - Application of an electric field causes dissociation of the $H_2O$ molecules into H+ and OH-.
  - Material can be either removed (volatile carbon oxide) or deposited (trapped oxygen in the carbon lattice) depending on process parameters: voltage, tip height, humidity, scan speed

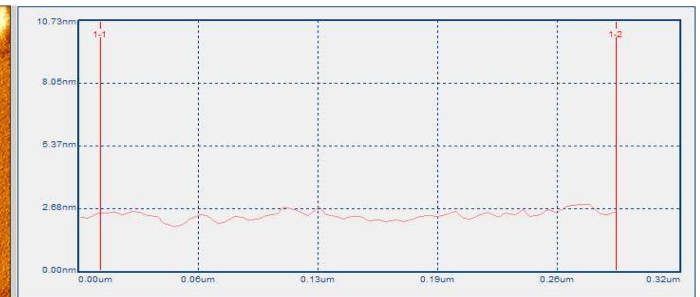


# Segmented CNT (>100nm)

Applied Voltage: -10 V
Scan Speed: 0.05 µm/s



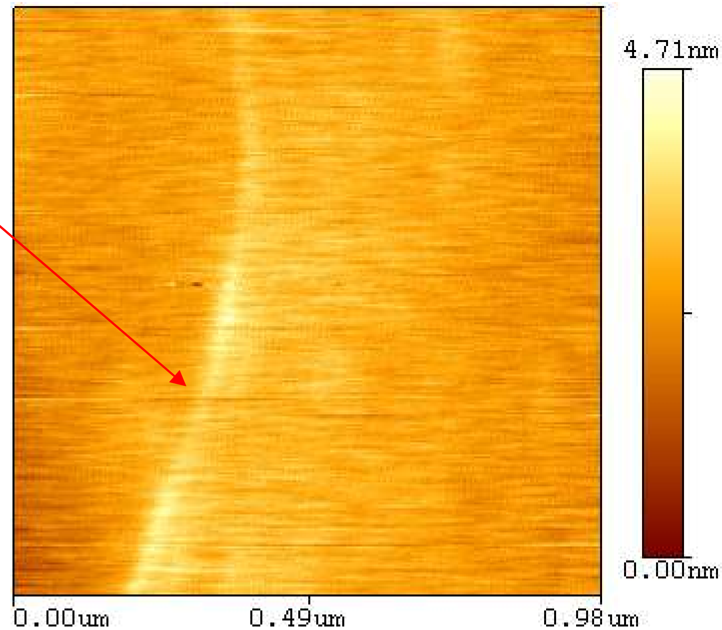




Line Angle:

| | | Pair 1 | | Pair 2 | |
|---|---|---|---|---|---|
| | | D (um) | Z (nm) | D (um) | Z (nm) |
| Line1 | 1 | 0.011 | 2.485 | | |
| | 2 | 0.288 | 2.519 | | |
| | distance | 0.277 | 0.034 | | |
| | pt.angle | 0.01 ° | | | |

# A few nanometers Cut?

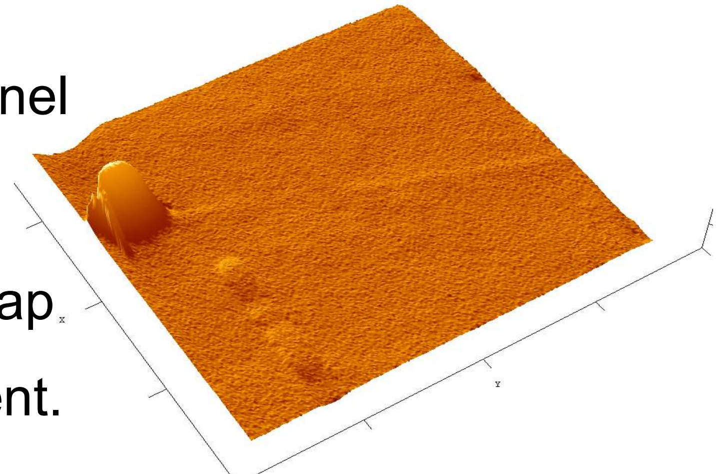
Cut

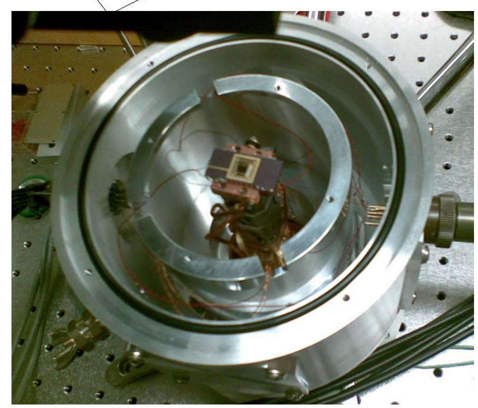



Applied Voltage: -10 V
Scan Speed: 0.01 μm/s

# Now and Future: Exploring Strong Confinement Region

- Create ~10nm CNT-QDs with <5nm tunnel barriers
- (For semiconducting CNTs), the band gap plays a strong role in the transport current.
- Perform a systematic study to understand the single electron transport and storage properties ➔ Differential conductance measurements and scanning probe spectroscopy under optical excitation.



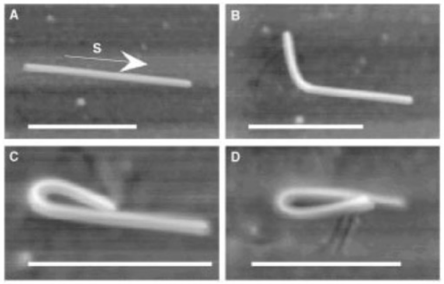


Low-temperature (4-300K) vacuum setup
Strauf, Stevens (Physics)

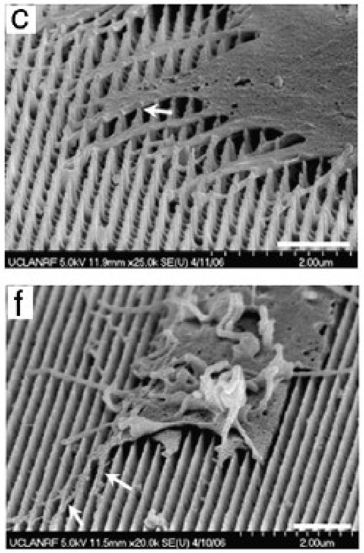
# I-2: CNT Nanoactuator

- Bimorph: piezoelectric, magnetic, thermal (optical, RF….)
- Low thermal expansion compared to metals:
  - $\alpha_{CNT}$ = < $3x10^{-6}$ /K
  - $\alpha_{Al}$ = $23x10^{-6}$ /K
- Vertical nanoactuator arrays

➔ Individual nanoactuator

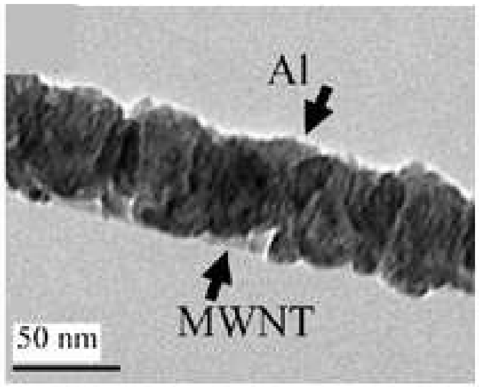



Falvo M. et al, "Mechanics and friction at nanometer scale", Journal of nanoparticle research, Vol. 2, p237

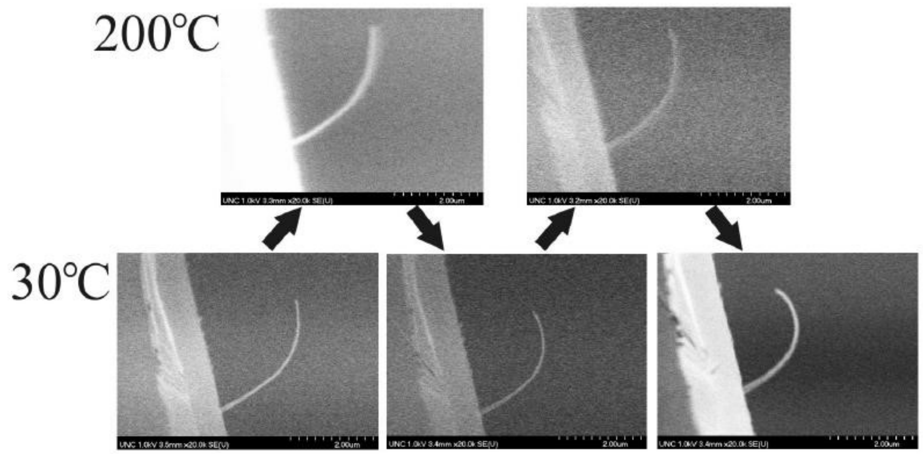



Choi C. H. et al, Biomaterials, Vol. 28, p1672 (2006)

# MWNT-Al Bimorph?




Thermally evaporated Al films

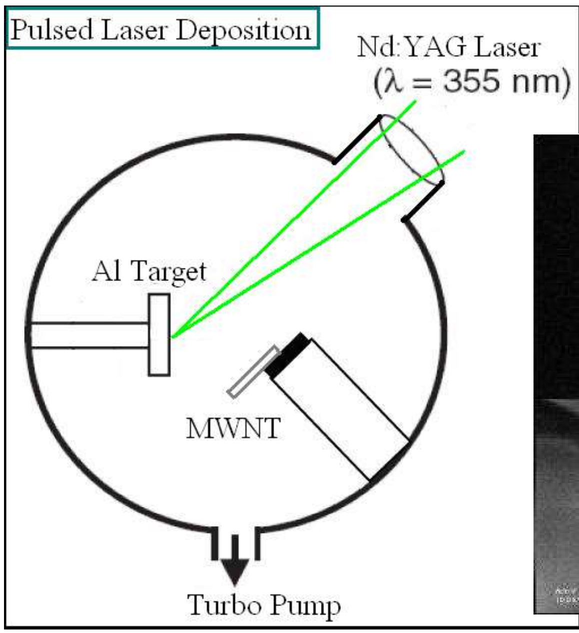


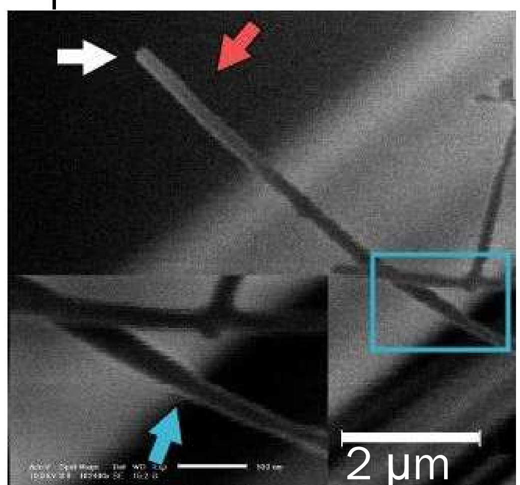

Actuation inside an SEM chamber

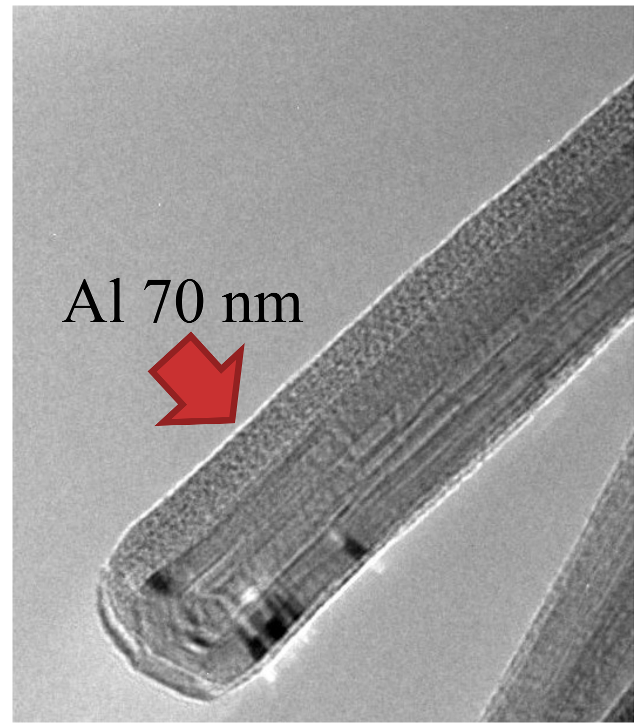
- Thermal evaporation on MWNT at RT and 150 K.
- Metal grains in various morphology
- Inconsistent deflection behaviors: do not follow the prediction well.

# MWNT-Al Bimorph PLD

## Pulsed Laser Deposition (PLD)



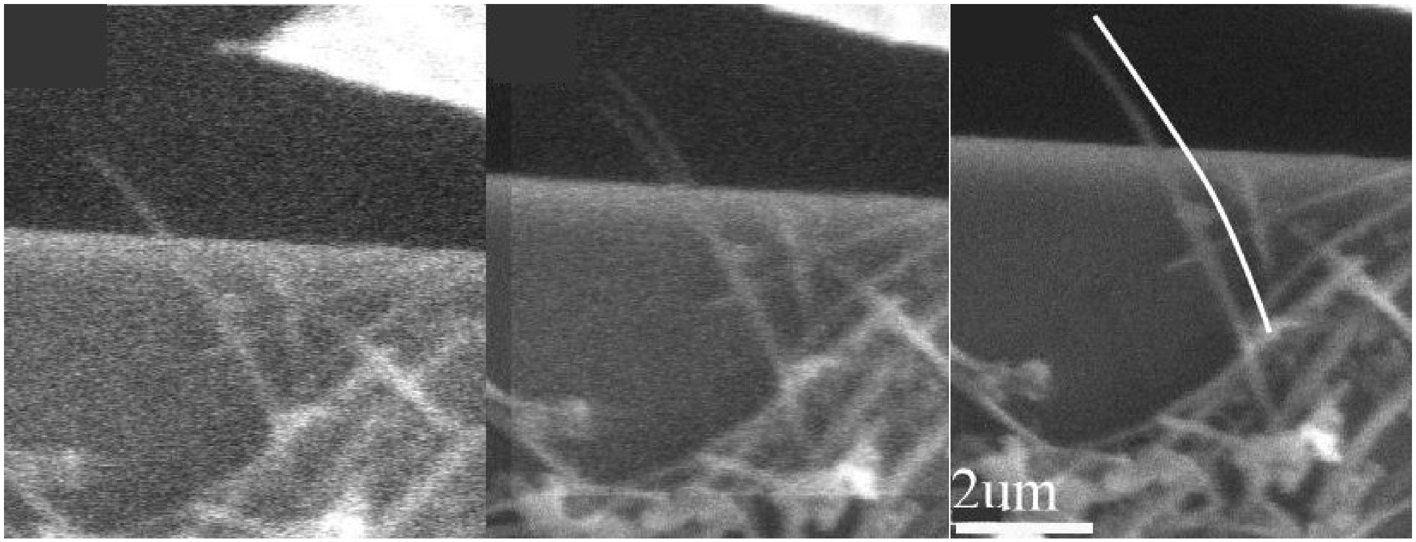




- Deposition Rate: 0.6 Å per min @ 160 mJ/pulse in 10 Hz
- Precisely formed metal film on only one side of a MWNT

# CNT Bimorph Actuation

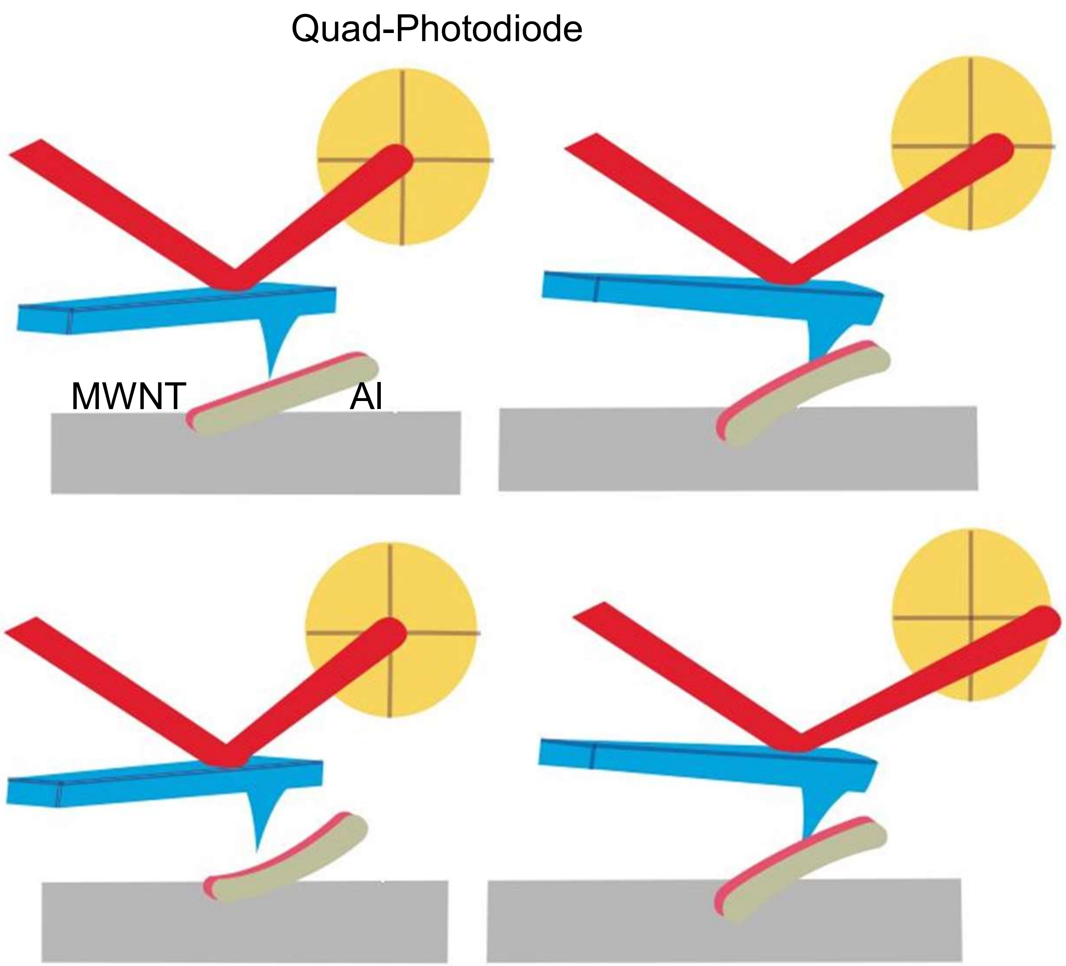





$$\Delta = \frac{3L^2(\alpha_{Al} - \alpha_{MWNT})\Delta T(d_{Al} - d_{MWNT})}{d_{Al}d_{MWNT}}\left[\left(\frac{d_{Al}}{d_{MWNT}}\right)^2 \frac{Y_{Al}}{Y_{MWNT}} + 4\frac{d_{Al}}{d_{MWNT}} + 6 + \left(\frac{d_{MWNT}}{d_{Al}}\right)^2 \frac{Y_{MWNT}}{Y_{Al}} + 4\frac{d_{MWNT}}{d_{Al}}\right]^{-1}$$

- From calculation, Δ = 430 nm.
- Measurement, Δ = 500 ± 200 nm at ΔT ~100K.

# LFM Measurement




No Actuation:

Reactive response only

at room temperature

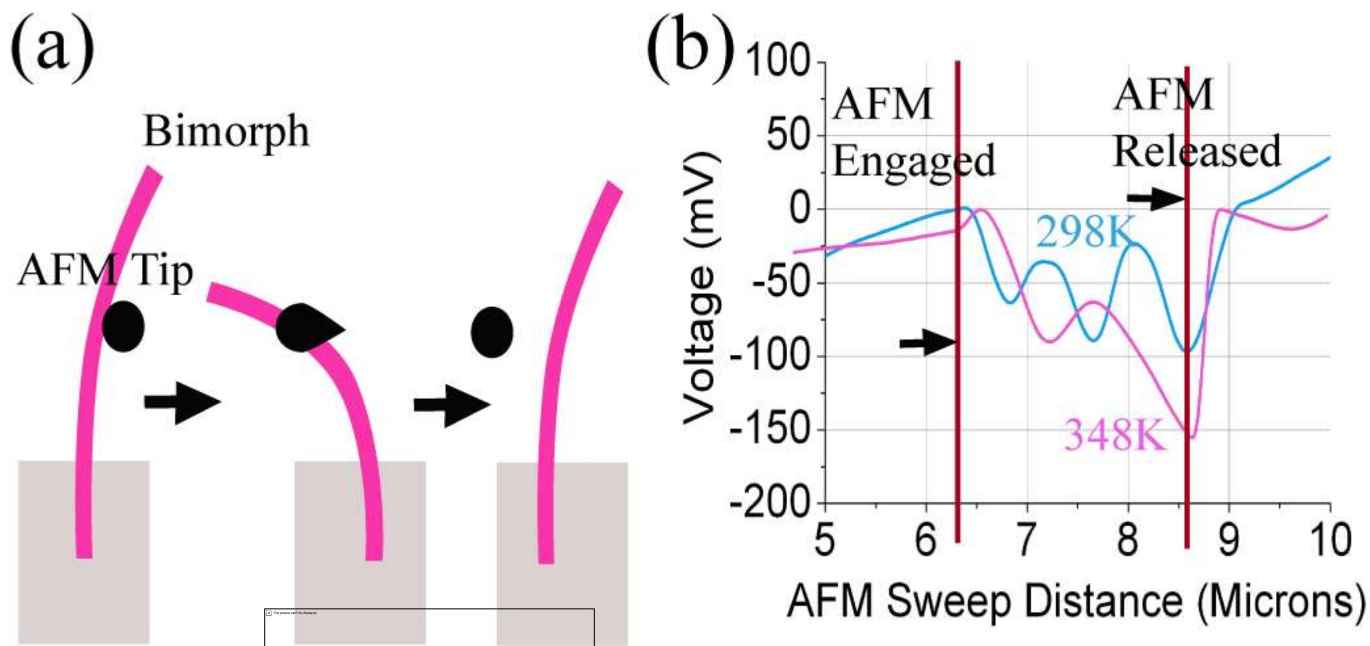
Actuation:

Reactive response + thermal response

at high temperature

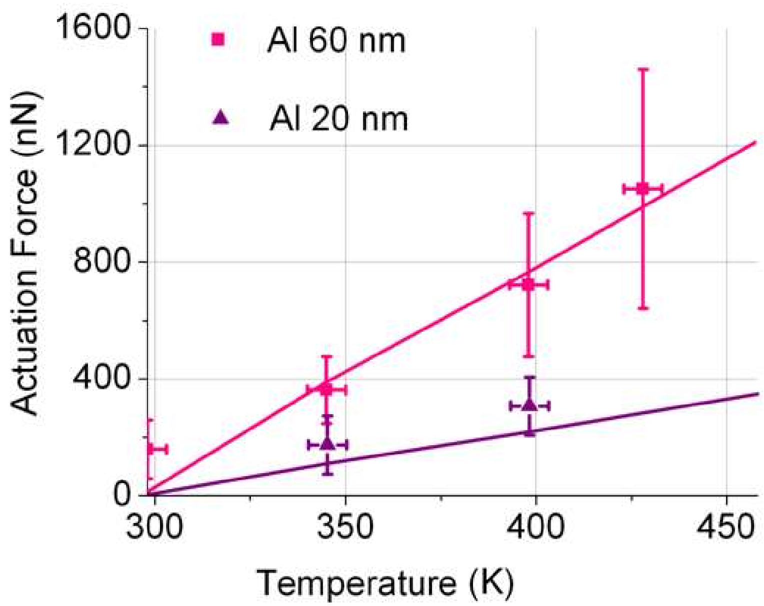
# Force Measurement






O. Sul ad **E. H. Yang,** “A Multi-Walled Carbon Nanotube-Aluminum Bimorph Nanoactuator,” *Nanotechnology*, vol. 20, 095502, 2009.

# Actuation Force



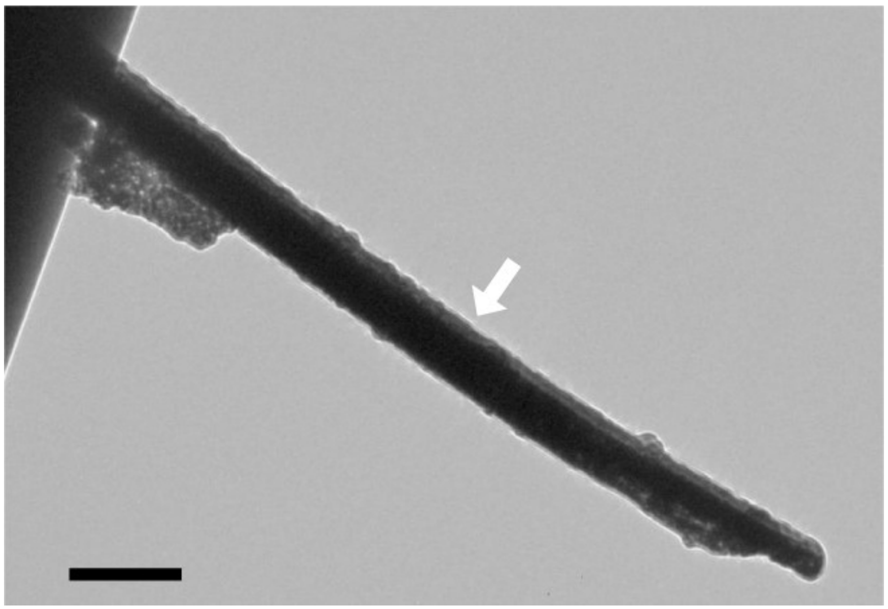

MWNT used

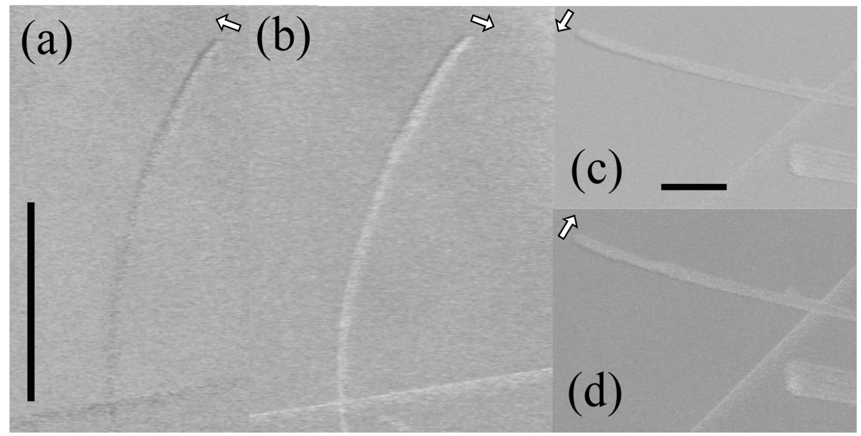
- L = 2.0 ± 0.2 μm long
- φ = 200 ± 20 nm

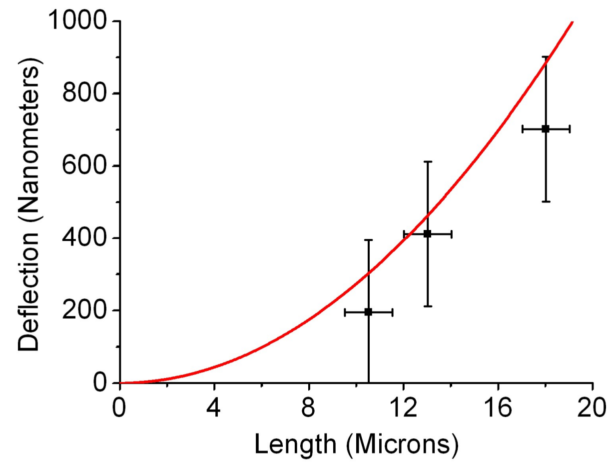
**Measured force up to 1 μN.**

$$F = \frac{w(\alpha_{Al} - \alpha_{MWNT})\Delta T}{L} \frac{Y_{Al} Y_{MWNT} d_{Al} d_{MWNT} (d_{Al} + d_{MWNT})}{d_{Al} Y_{Al} + d_{MWNT} Y_{MWNT}}$$

# $Ni\text{-}Al_2O_3$ Bimorph Actuation



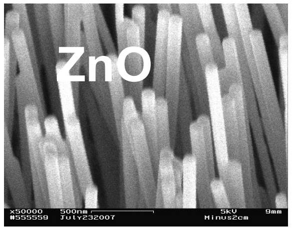


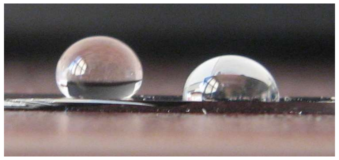



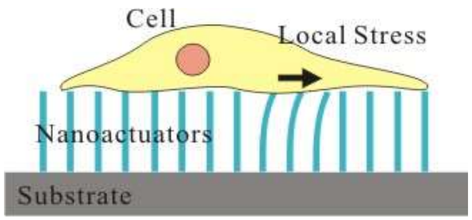

O. Sul, S. Jang and **E. H. Yang,** "Fabrication and Characterization of a $Ni\text{-}Al_2O_3$ Bimorph Nanoactuator", *Journal of Vacuum Science and Technology B; in review*

# Vertical Nanoactuator Arrays

On-demand control of surface features ➔ wetting behaviors
(Example: Change in water contact angle)



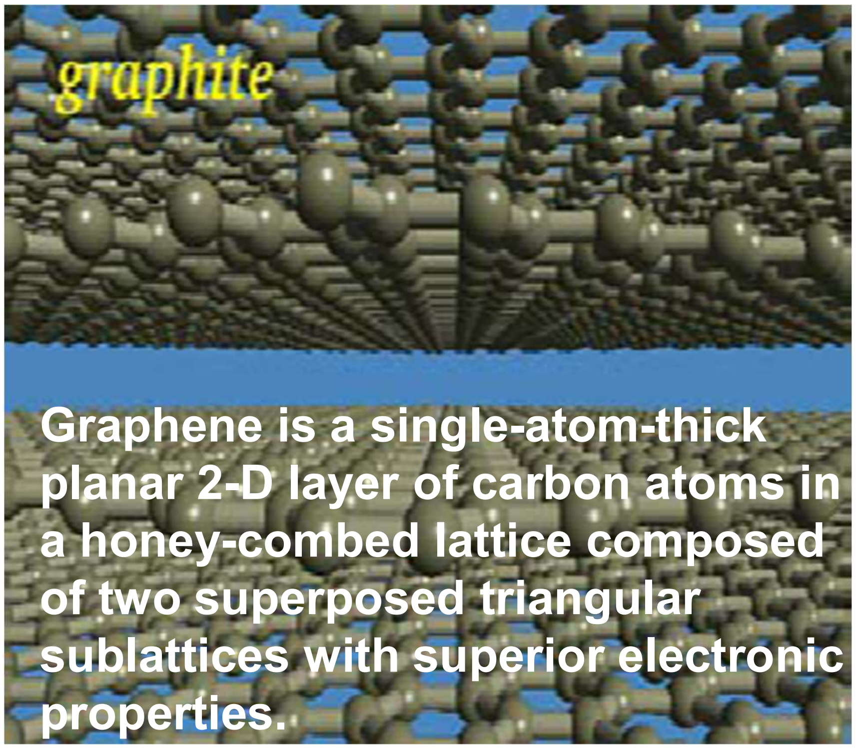






+ Nanoantenna?

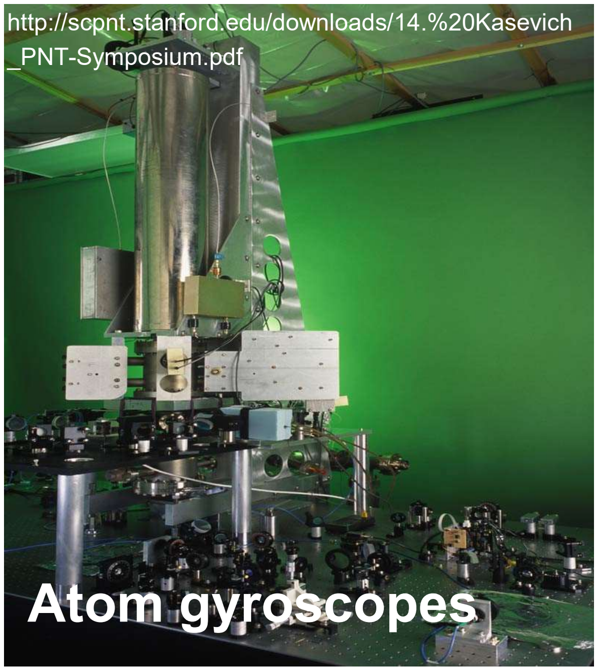
On-demand control of surface features ➔ cell behaviors
(Example: Adhesion, cell morphology, surface antigen display, gene expression…)

# II-1: Graphene Gyroscope?

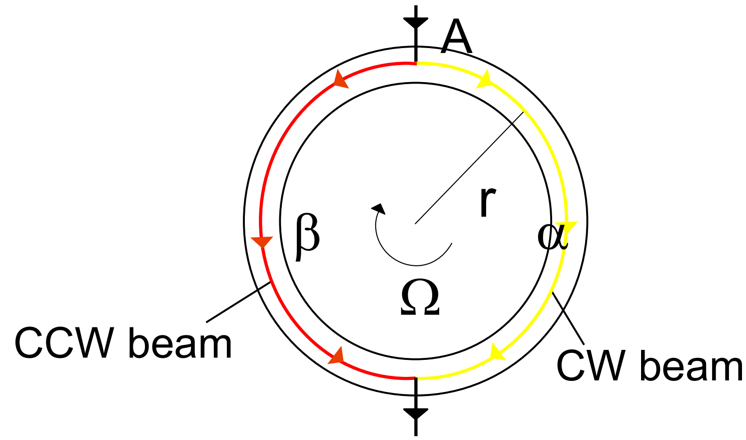



- Conduction and valence bands touch at two nodal zero-gap points in the first Brillouin zone.
- Electron mobility as high as 200,000 $cm^2/Vs$ at RT, with carrier density of $10^{13}/cm^2$ and mean free path ~ 1 $\mu$m.
- Stable up to 3,000 K and has a quantum hall effect at RT.

# Matter Wave Gyroscopes?




- Rotational motion can be detected via a phase shift between two arms of an interferometer.




- Sensitivity: 60 μdegree/hr bias stability
- Operation at ~100nK, high vacuum, high magnetic fields
- Very heavy: several hundred kilograms

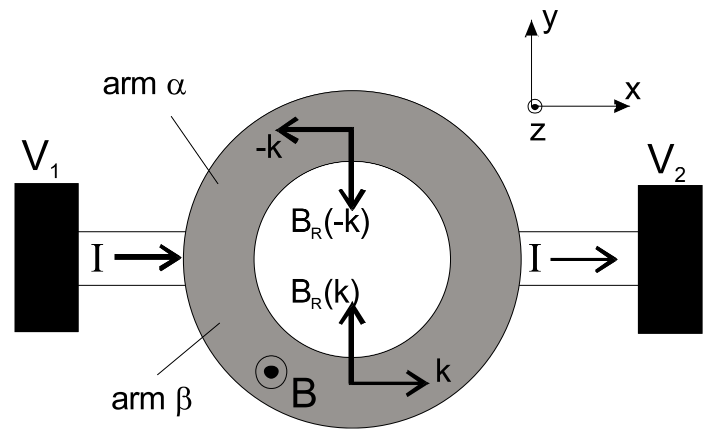
**Optical gyroscopes**

- No moving parts
- 0.01 degree/hr bias stability
- Costs > $ 250K
- 50 cm3, 19 lb, 30W

# Electron Interferometer

- **Electron interferometer:** Only experiments were done with electron beam in vacuum.

- Using electron Sagnac effect, the measured signal would be larger than an optical interferometer by, $Mc^2 / \hbar\omega \sim \mathbf{10^5}$



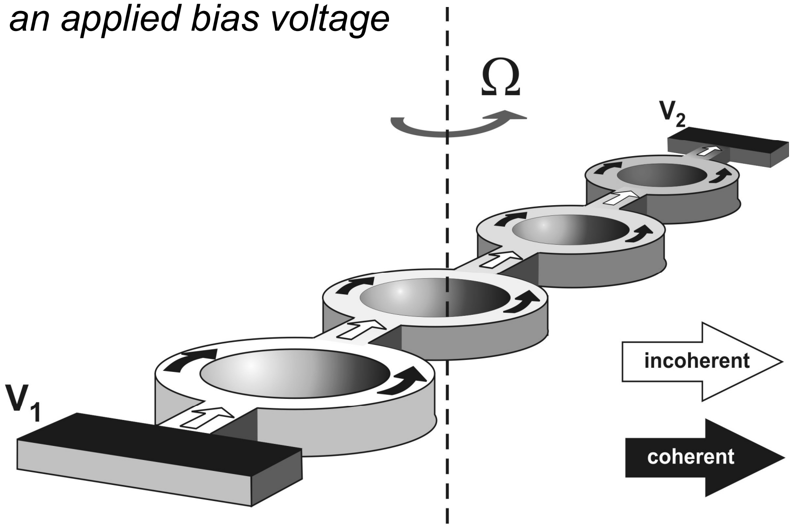

**Concept and calculation by Prof. Search, Stevens (Physics)**

M. Zivkovic, M. Jaaskelainen, C. P. Search, and I. Djuric, "Sagnac Rotational Phase Shifts in a Mesoscopic Electron Interferometer with Spin-Orbit Interactions," PACS, February 1, 2008

# Graphene Ring Design

- Solid-state electron interferometer
- Electron scattering lengths
- Sagnac phase shift proportional to both the enclosed area of the interferometer and the rotation rate.
- **Cascaded linear array enhances the sensitivity by $N^{1/2}$.**

*Consisting of a ballistic ring connected to two leads with an applied bias voltage*

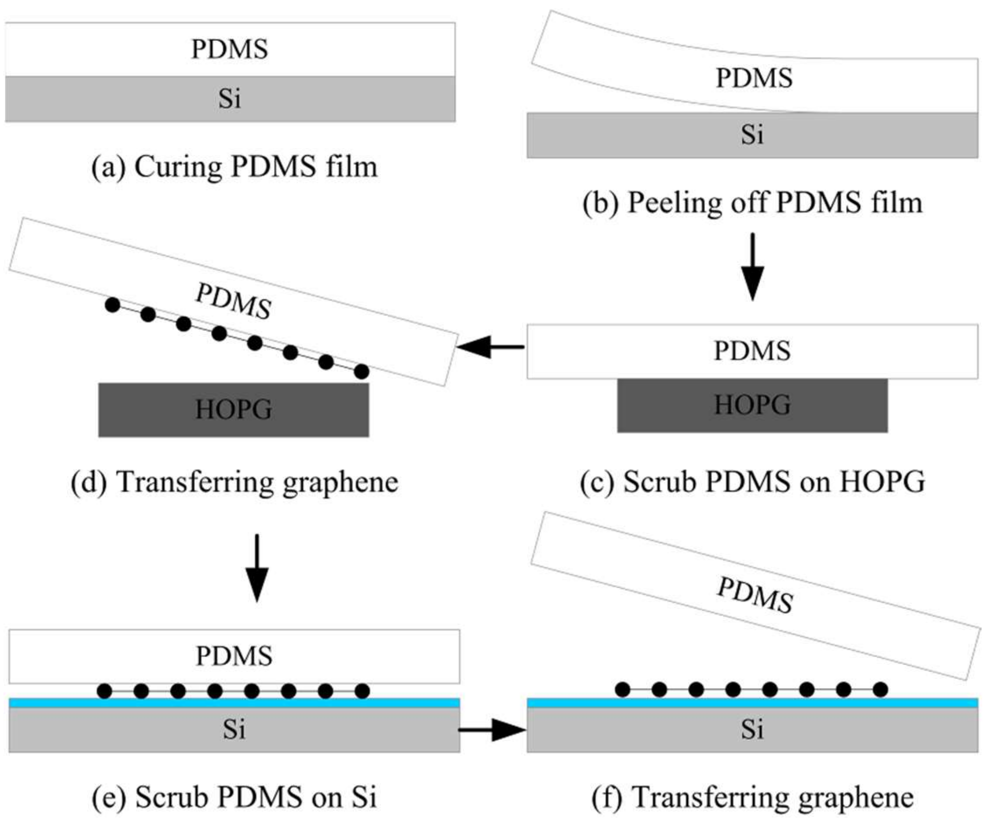


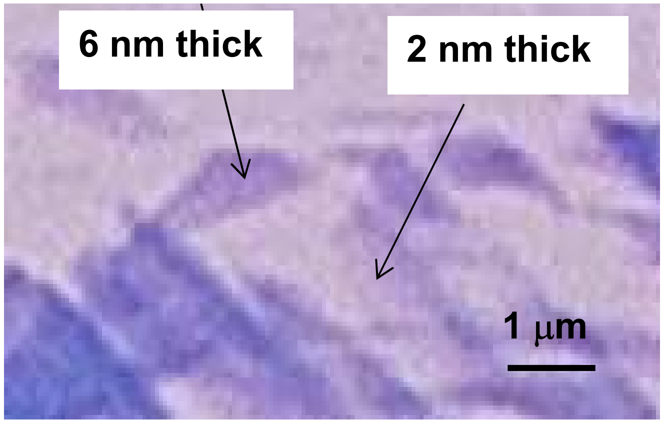

**Concept and calculation by Prof. Search, Stevens (Physics)**

M. Zivkovic, M. Jaaskelainen, C. P. Search, and I. Djuric., Phys. Rev. B **77**, 115306 (2008)

Provisional patent filed

# Graphene Synthesis



# Nanopatterning (VAFM)

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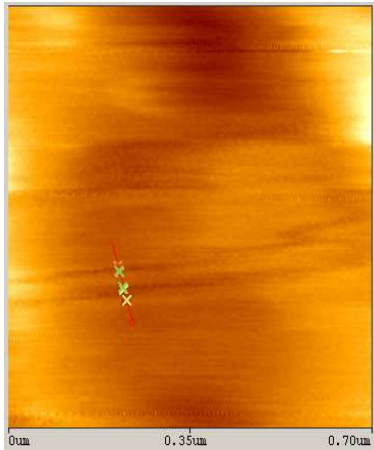
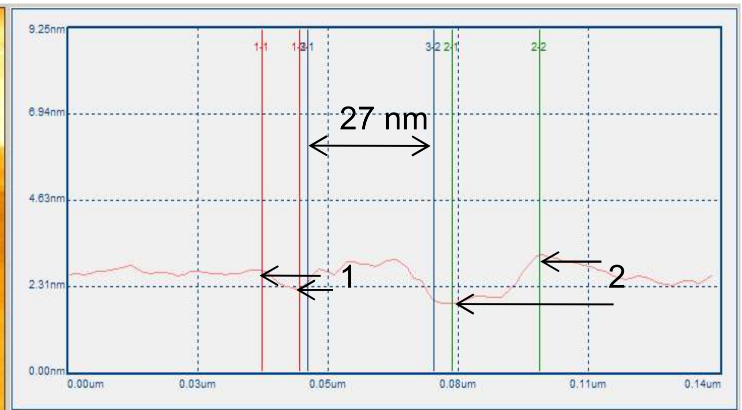
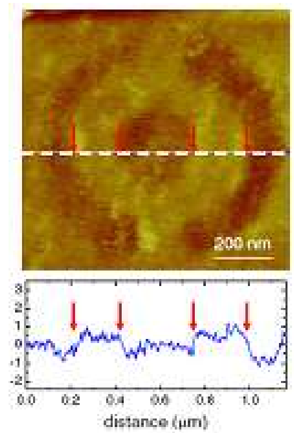


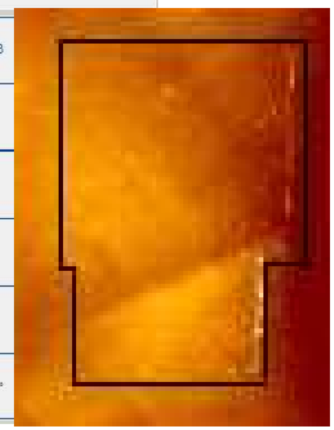




Line Angle:

| | | Pair 1 | | Pair 2 | | Pair 3 |
|---|---|---|---|---|---|---|
| | | D (um) | Z (nm) | D (um) | Z (nm) | D (um) |
| Line1 | 1 | 0.040 | 2.728 | 0.080 | 1.837 | 0.050 |
| | 2 | 0.048 | 2.203 | 0.099 | 3.142 | 0.077 |
| | distance | 0.008 | -0.525 | 0.018 | 1.305 | 0.027 |
| | pt.angle | -3.80 ° | | 4.06 ° | | -0.95 ° |




*Appl. Phys. Lett.* ***93****, 093107 2008*



Graphene oxide bumps on highly pyrolyzed ordered graphite.

K. Kumar, Y. T. Tsai, O. Sul and **E. H. Yang**, "Nanoscale Graphene and Carbon Nanotube Lithography using an Atomic Force Microscope," *ASME International Mechanical Engineering Congress and Exposition*, Lake Buena Vista, FL, Nov 2009

# Performance Testing

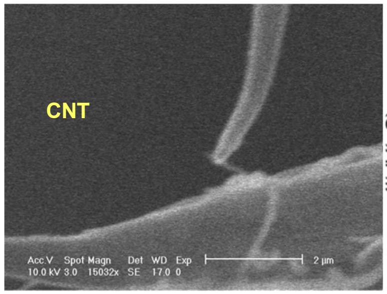
- The electron wave interference changes the conductance across the rings.

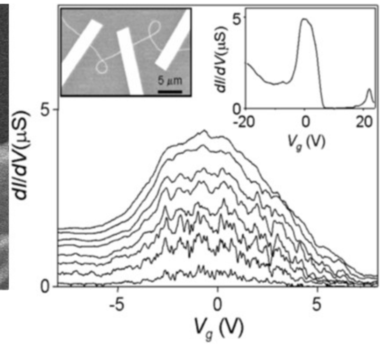
Phase shift depends on rotation rate

$$\Delta\phi = 2mA\Omega/\hbar$$

Conductance depends on phase shift

$$G = I/V = (e^2/h)\cos^2(\Delta\phi + \theta)$$




PRL 98, 246803 (2007)

- **Modulation of the path length using a gate electrode close to one arm to simulate the *rotation* (interferometer detuning)**

- **The fully developed device with *thousands times smaller* area of graphene would measure equivalent rotations as optical gyroscopes.**

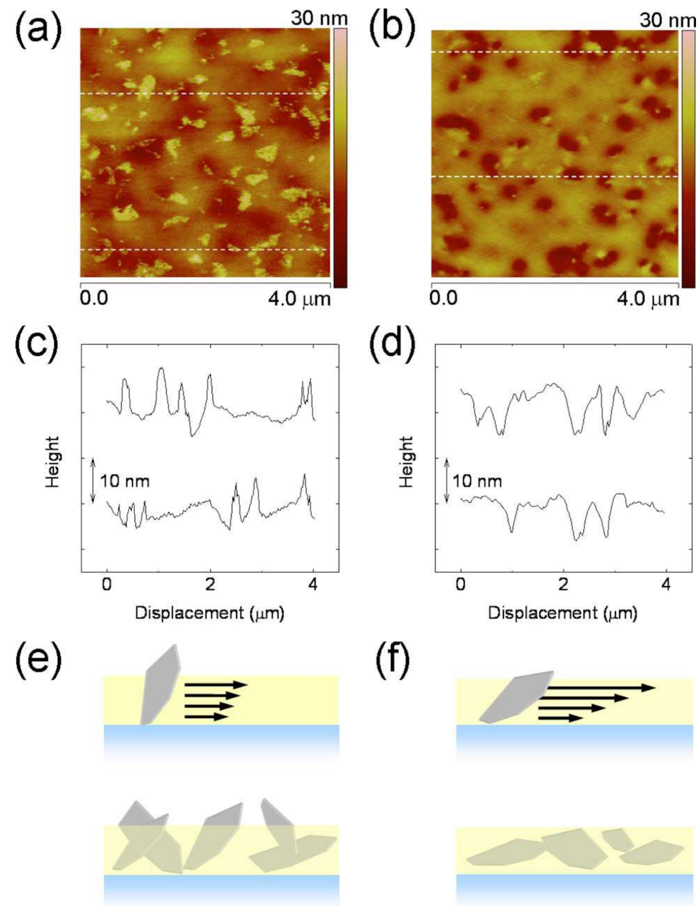
# II-2: Graphene Filed Emission

- CNT's field emission properties: turn-on voltage 1~3 V/$\mu$m and emission current as high as 100 $\mu$A from a single CNT
  - attractive as cold-cathode field emission sources and lightweight packages
- FE characteristics from graphene?
- Planar form of graphene ➔ CMOS compatible process, an advantage for potential industrial fabrication

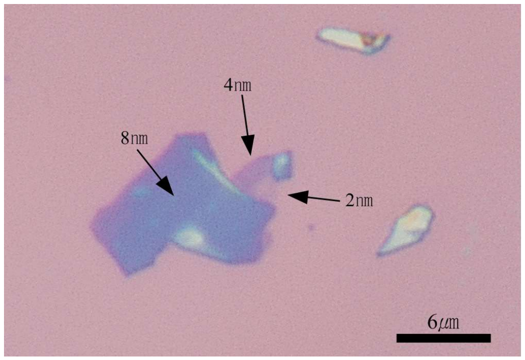
# Previous Study on Graphene Filed Emission

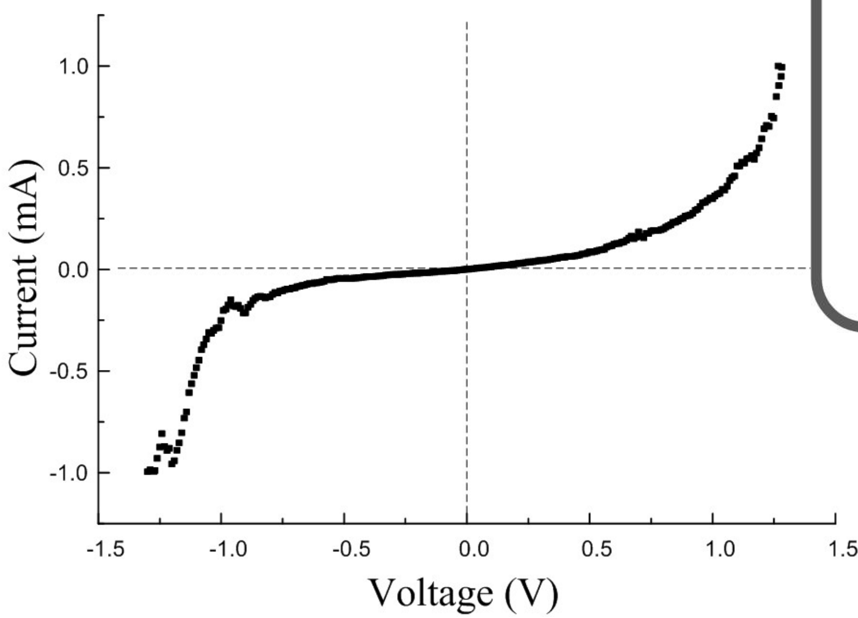
- Randomly oriented oxidized graphene sheets protruding from the film: *Applied Physics Letters **93**, 233502 2008*

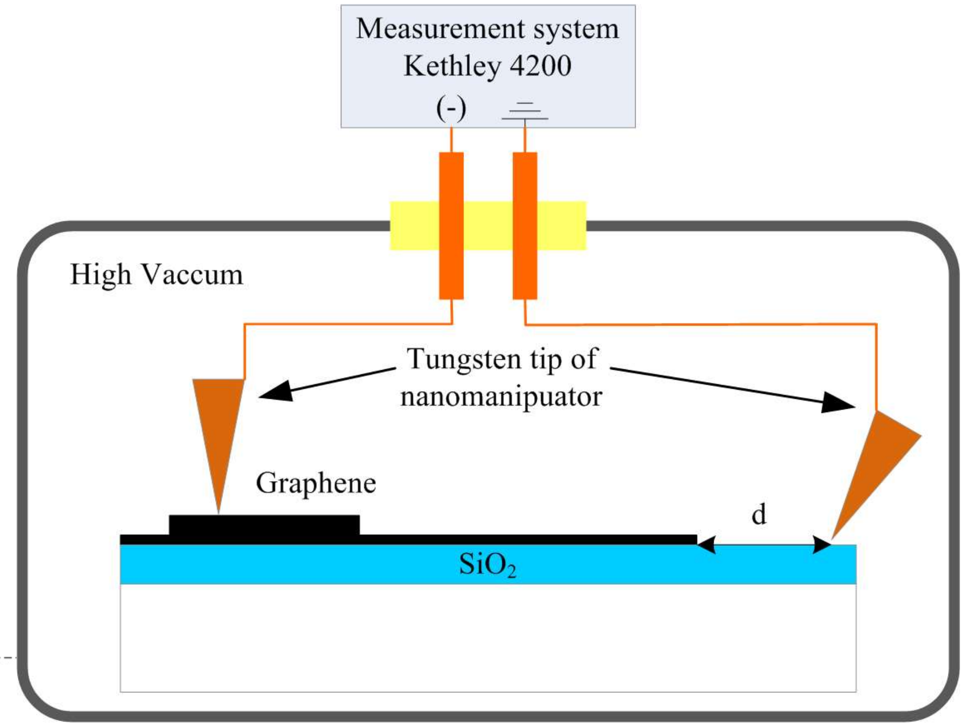
- ***There is no report on the field emission***
  - ***from high-quality (highly ordered pyrolyzed) graphene sheets ,***
  - ***in planar geometry, or***
  - ***in device applications.***



# Experimental Setup

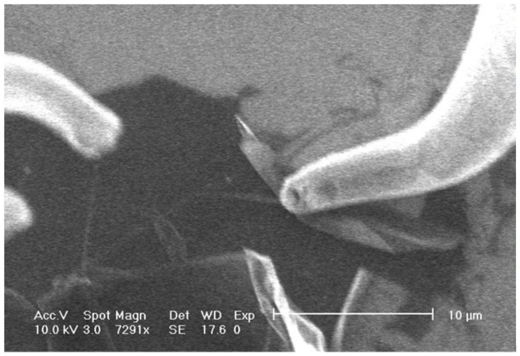


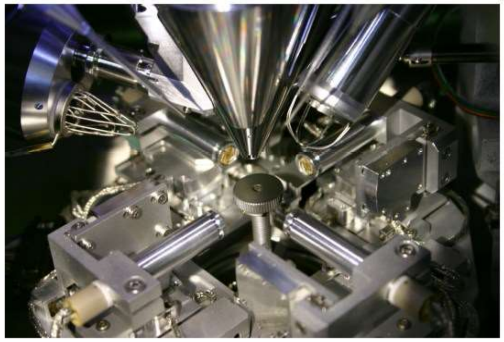



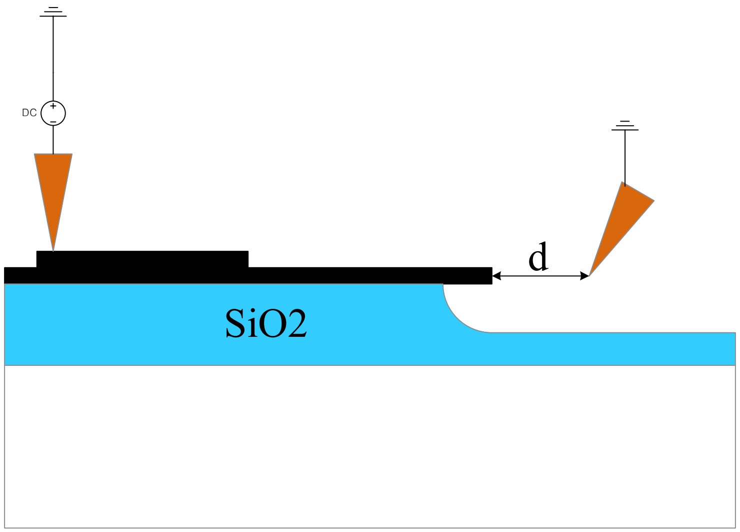

Keithley semiconductor measurement system through feed-troughs in the vacuum chamber

# Graphene Prepared for Field Emission Testing

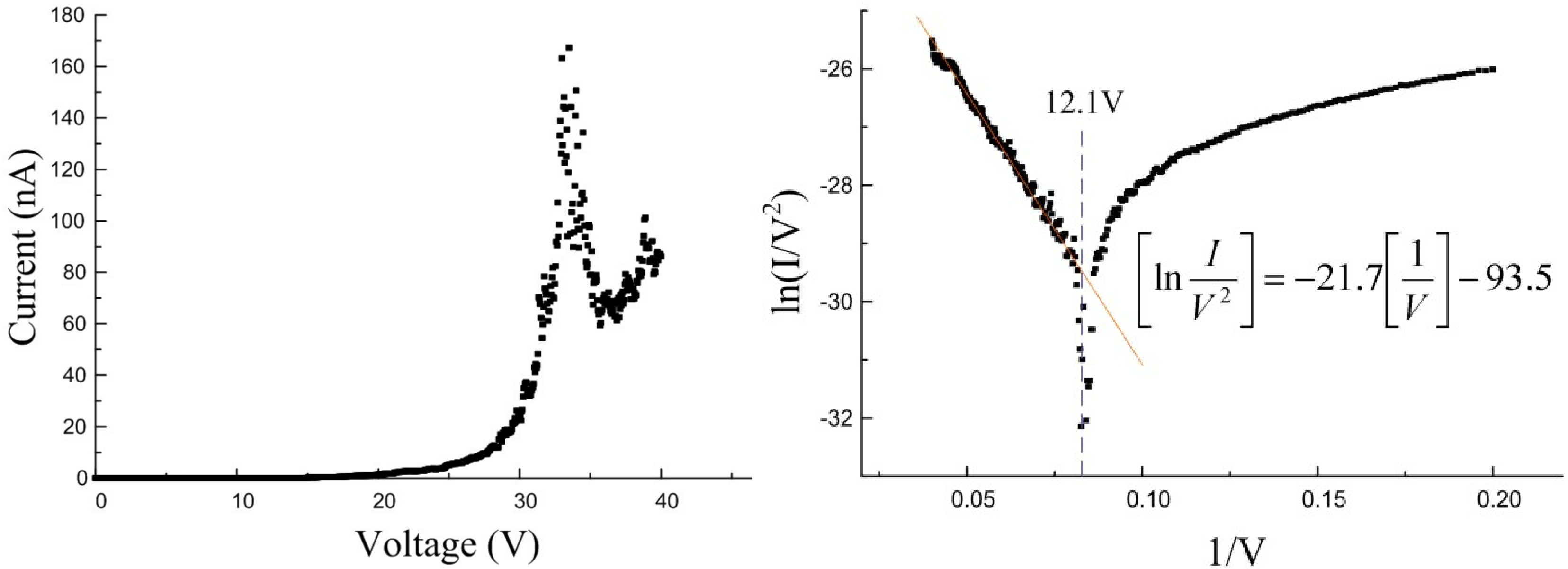


One tip placed on the sample directly as a cathode and the other placed apart from the edge of graphene sheet as an anode.




# Field Emission Characteristics

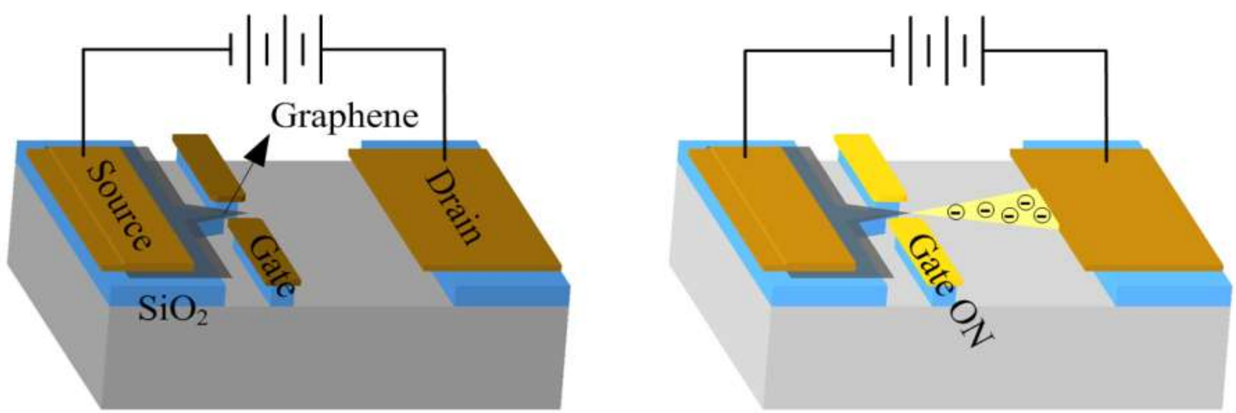



- Electron emission current exponentially increased up to 170 nA following the behavior of the Fowler-Nordheim relationship.
- For the exponentially increasing region (of current), the F-N curve shows linear relationship (confirming the field emission characteristic).


S. W. Lee, S. S. Lee and **E. H. Yang**, “Field Emission from Graphene Structures for Vacuum Transistor Applications,” ASME International Mechanical Engineering Congress and Exposition, Lake Buena Vista, FL, Nov 2009

# Near term goal: Graphene Triode

- Crystal orientation, number of layers, suspended structures…
- A graphene electrodes (source, drain, gate….) can be fabricated in-plane along with other components (e.g. precisely patterned graphene triode based on high-quality graphene sheets)

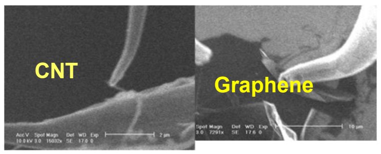



(a) Gate off (b) Gate on

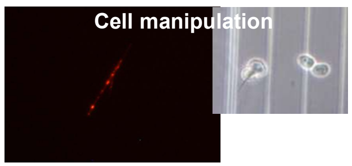
# Conclusions

- We are exploiting carbon nanotube and graphene nanostructures.
- Overcoming the technical challenges will enable one to leverage the outstanding properties of CNT and graphene in the development of next-generation devices with unrivaled functionality.
- Such capabilities show potential widespread application in areas such as sensors, actuators and nanoelectronc systems.

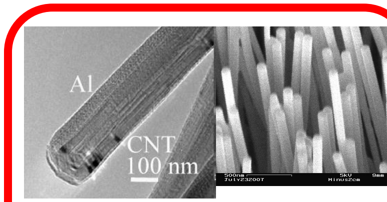
# Project Overview



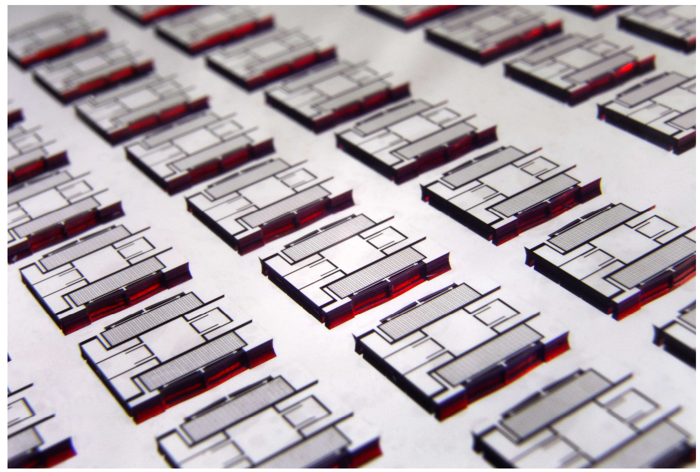
**CNT and Graphene nanoelectronics**



**Multi-functionalized nanowires**

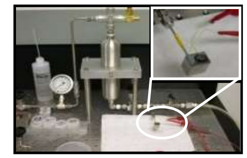


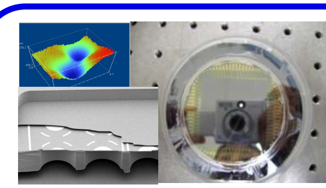
**Nanoactuators and nano-antenna arrays**

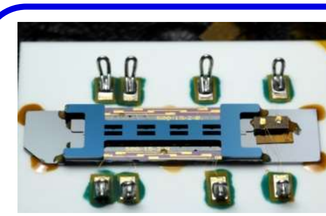


## Collaborators

**Strauf, Wang, Search, Fisher, Choi, Shi, Liang, Besser, Cui, Manoochehri (Stevens); Choi (U. Idaho); Myung (UC Riverside); Bar-Cohen, Fink, Yee (NASA/JPL); Su (NASA/LaRC); Getty, Li (NASA/GSFC); Wick (Sandia National Lab); Lu (RIT), Khodadadi (Auburn); Trolier-McKinstry (PSU); Jiang (TRS); Chary (Caltech); Huang (U. Arkansas); Lee (KAIST); Jeong (Inje); Yang (Ajou)**


**Leak-tight piezoelectric microvalve**


**Large-stroke deformable mirror**


**Linear microactuator**