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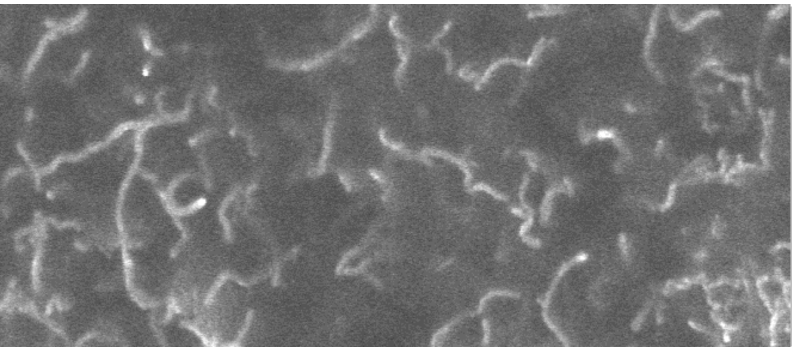

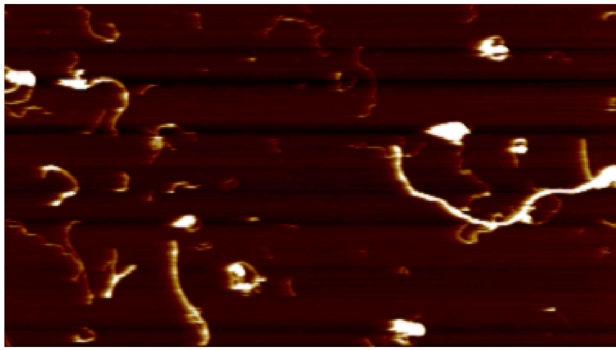

Characterization of the Electrical Properties of Individual Multi-Walled Carbon Nanotubes

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U.S. DEPARTMENT OF ENERGY

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NNSA National Nuclear Security Administration

Usage of Fiber-Reinforced Composites

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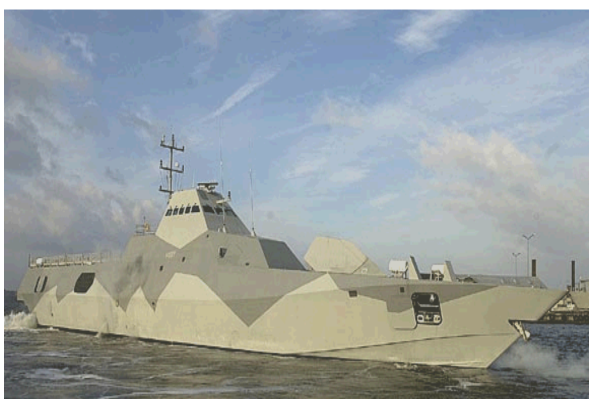
- Over the past 50 years, increased usage of composite materials

Commercial aircraft systems

Future and legacy spacecraft

Military aircraft



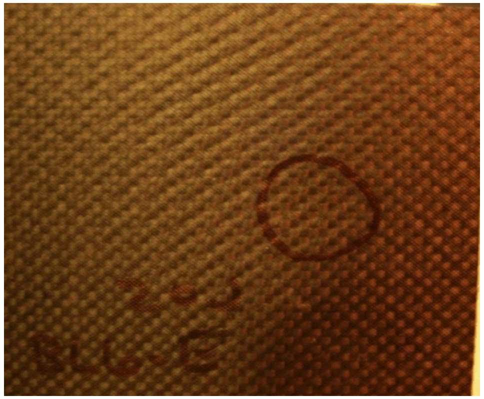
Naval structures

Wind turbine blades

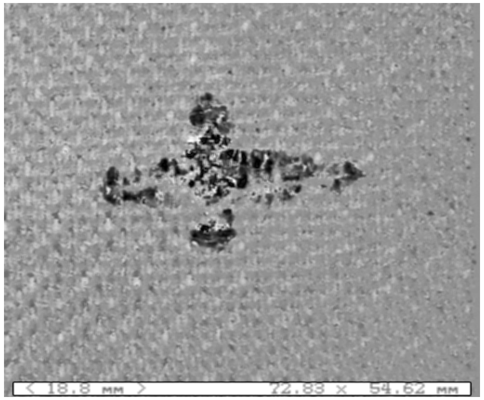
CFRP cable stay bridge

Composite Damage Modes

- Susceptible to damage due to:
  - Strain, impact, chemical penetrants, multi-axial fatigue
- Damage modes:
  - Matrix cracking
  - Fiber-breakage
  - Delamination
  - Transverse cracking
  - Fiber-matrix debonding
  - Matrix degradation
  - Blistering
- Difficult to detect
  - Internal to laminate structure
  - Nearly invisible to naked eye
  - Current methods are laborious



Visual inspection



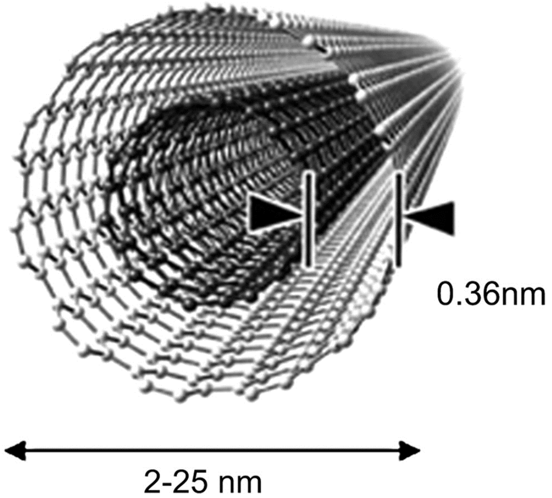
C-SCAN ultrasound image

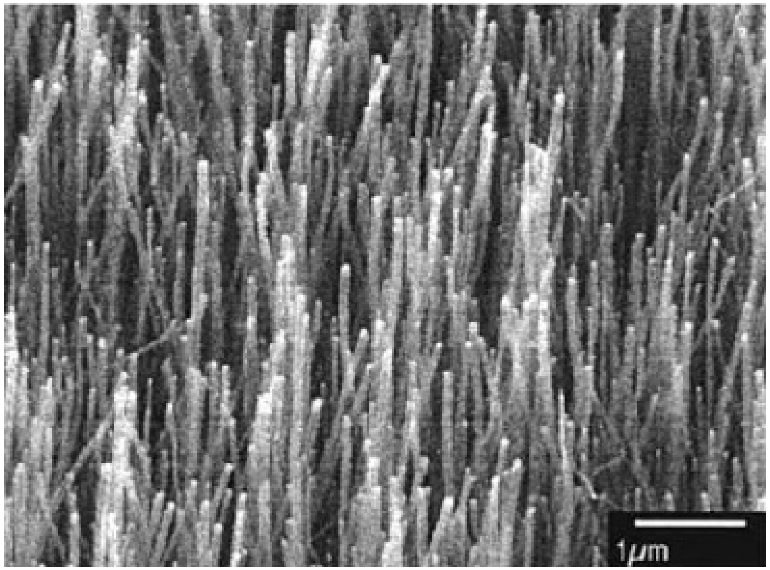
CFRP panel after 20 Joule impact

Aircraft ultrasonic inspection (Composites World)

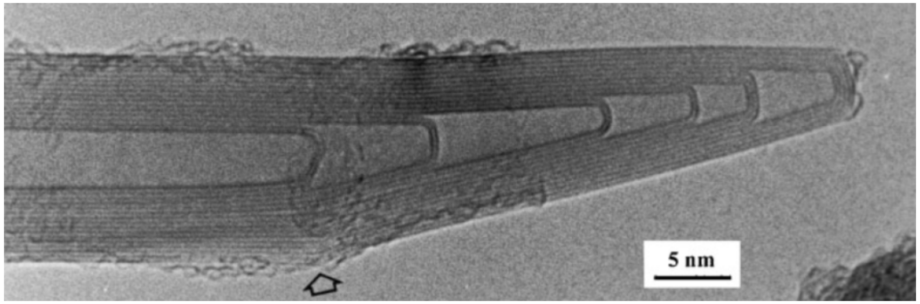
Carbon Nanotubes

- Multi-walled carbon nanotubes (MWNT):
  - Rolled concentric cylindrical structures constructed of graphene sheets
  - Diameter: 6 ~ 100 nm
  - High-aspect ratios: ~10^3 to 10^7
  - Metallic conductivity
  - Five times stiffer and ten times stronger than steel





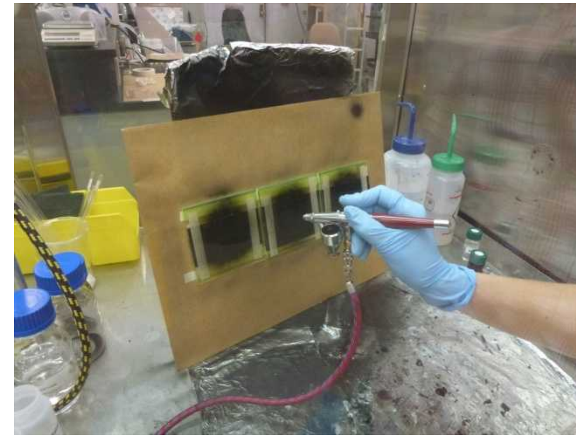
Aligned carbon nanotube forest
Thostenson, et al. (2001)

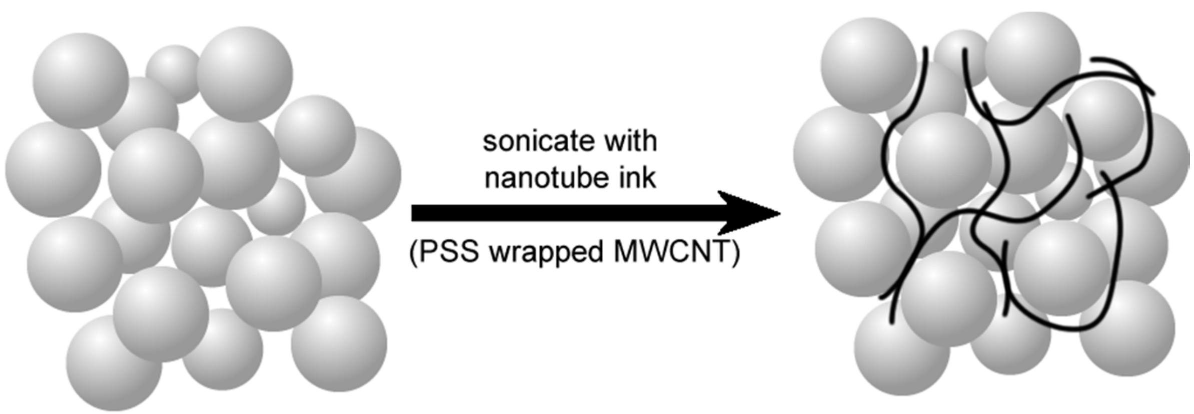


TEM imagery of an end cap of a MWNT
Harris (2004)

Sprayable MWNT-Latex Thin Film

- Rapid large-scale deposition
  - Required for mass deployment of methodology
- MWNT-PSS/Latex paint formulation
  - Collaborated to improve initial Sandia formulation
  - Sub-micron PVDF creates mold for MWNT organization
  - Off-the-shelf deposition method



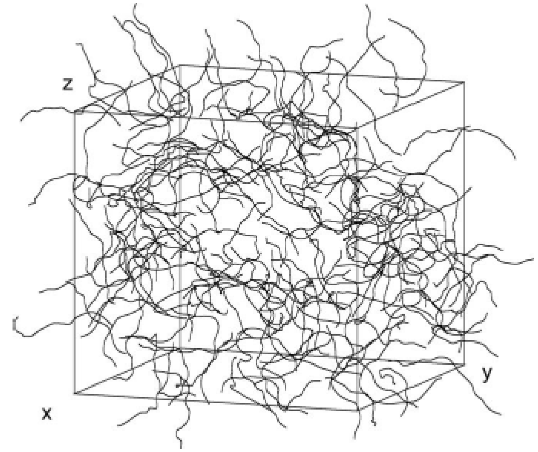


Kynar Aquatec™ latex solution (avg. particle size 150nm)

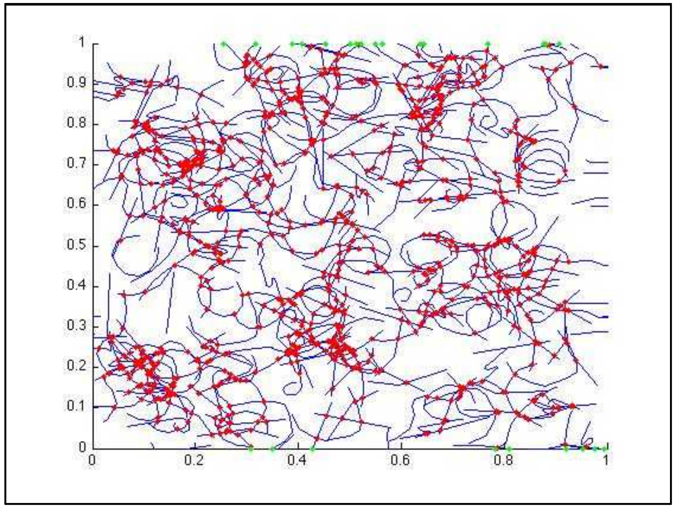
Forms segregated MWCNT network

CNT Network Modeling

- Network response over variety of environments
  - Cost prohibitive to test thin films over a wide variety of environments
  - Better to capture thin film response in a model
- Network creation
  - Statistical or imaged network
- Inputs
  - Lengths
  - Diameters
  - Geometry / Curvature
  - Conductivity of MWCNTs
  - Junction resistances



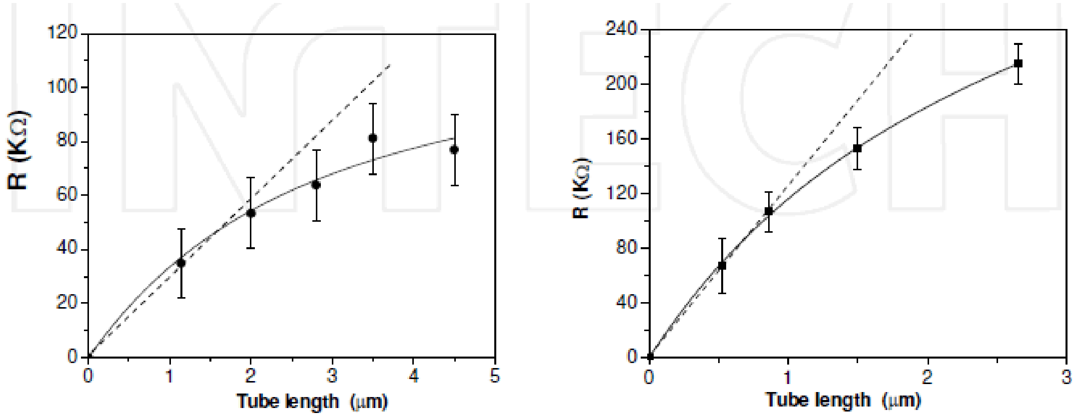
3D network of CNTs
(Dalmas et. al., Acta Materialia, 2006)



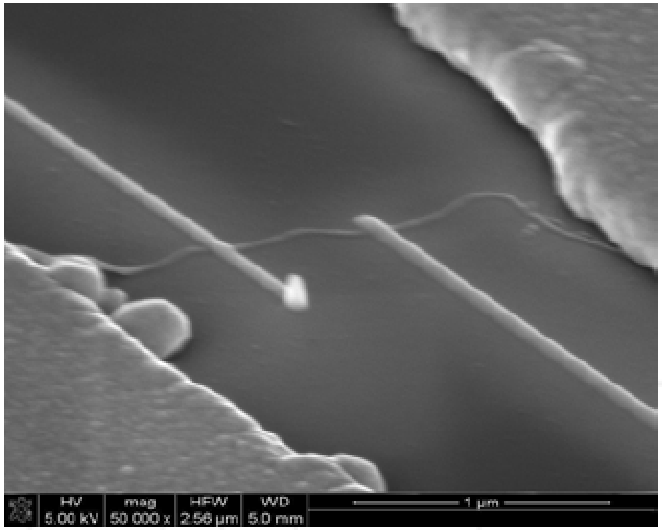
300 CNT model using realistic CNT geometries

CNT Electrical Properties

- MWCNT conductivities
  - Most important of attribute of parameters in network
  - Difficult to obtain
    - Nano-scale characterization
    - Tedious specimen fabrication
  - Different for each MWCNT type
- MWCNT junctions
  - Dominate network circuit response
  - Harder specimen fab
    - Need to get 2 MWCNTs to cross and interact with electrodes



Two- (Left) and Four-point (right) probe measurements of 15 nm diameter CVD-grown MWCNTs (Moshkalev et. al., Solid State Circuits Technologies, 2010.)



(Moshkalev et. al., Solid State Circuits Technologies, 2010.)

Conductive-Atomic Force Microscopy

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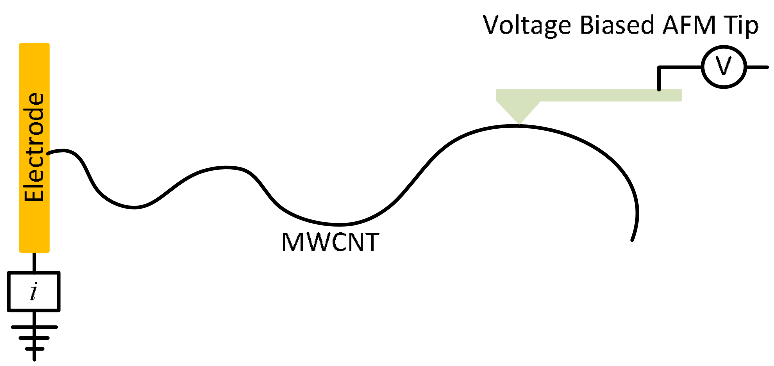
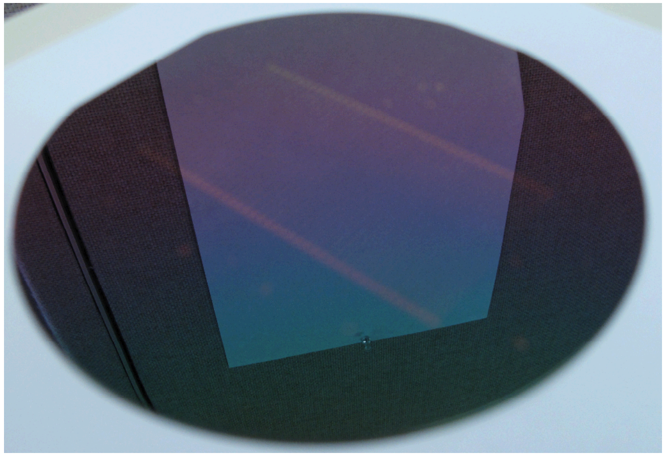
- Conductive - AFM
  - Biased tip will complete circuit when in contact with MWCNT
  - Can scan large [100 μm]^2 areas
  - Can use topographic image to land tip for I-V curves
    - Determine if metallic/semi-conducting (SWCNTs)
  - Allows for nanoscale measurements without needing sample exchange like SEM
- Process
  - Electrode deposition/patterning on wafer
  - Dielectrophoretic CNT depostiion
  - AFM measurement



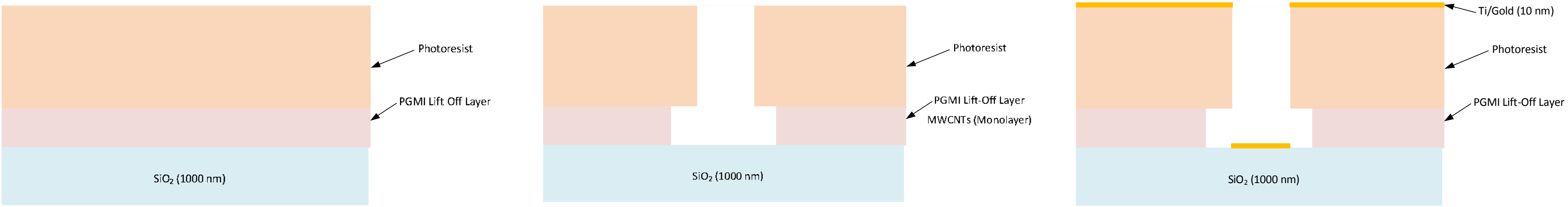
Contact Conductive AFM

Electrode Deposition

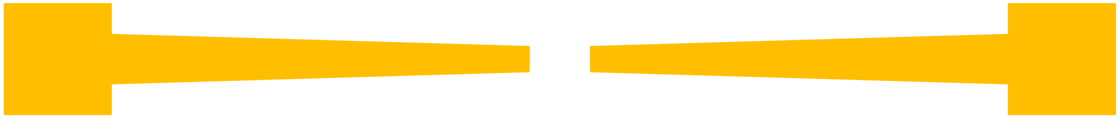
- Opposing electrode configuration
  - Allows for di electrophoresis
  - Doubles number of CNT-electrode connections
  - Au metal contacts
    - 5 µm wide electrodes
  - 5 mm sized contact pads
    - Micro- to macro-scale circuitry
  - Fabricated with traditional lithography practices



Wafer with Au electrodes



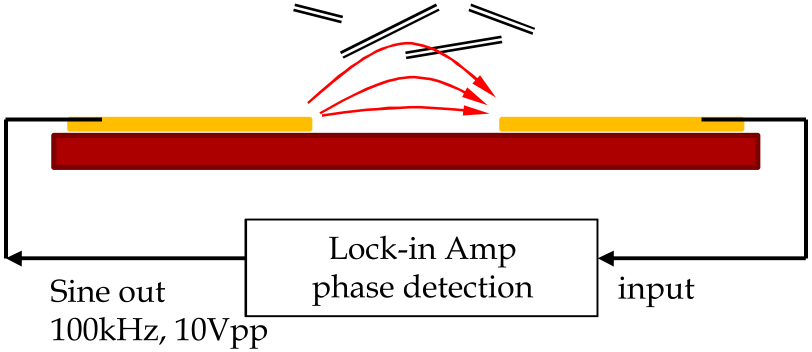
Au electrode deposition process

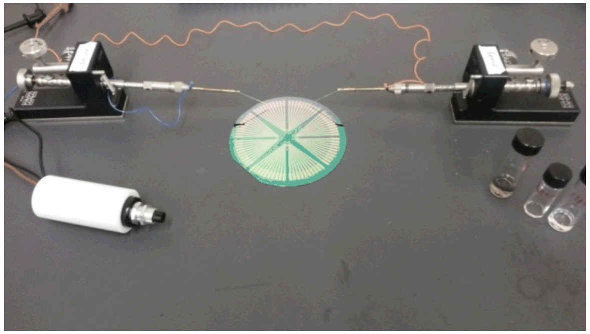


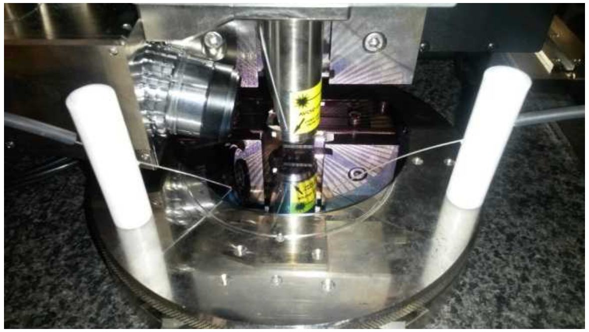
Opposing electrode pattern

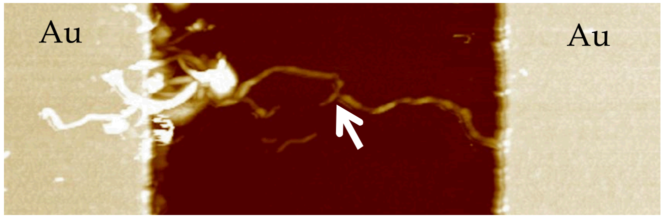
CNT Capture / Alignment

- Dielectrophoresis
  - Oscillating voltage through CNT solution
  - Captures CNTs between the electrodes
  - Electric forces orients CNTs normal to the electrode
  - Allow for out of cleanroom processing/capturing of CNTs



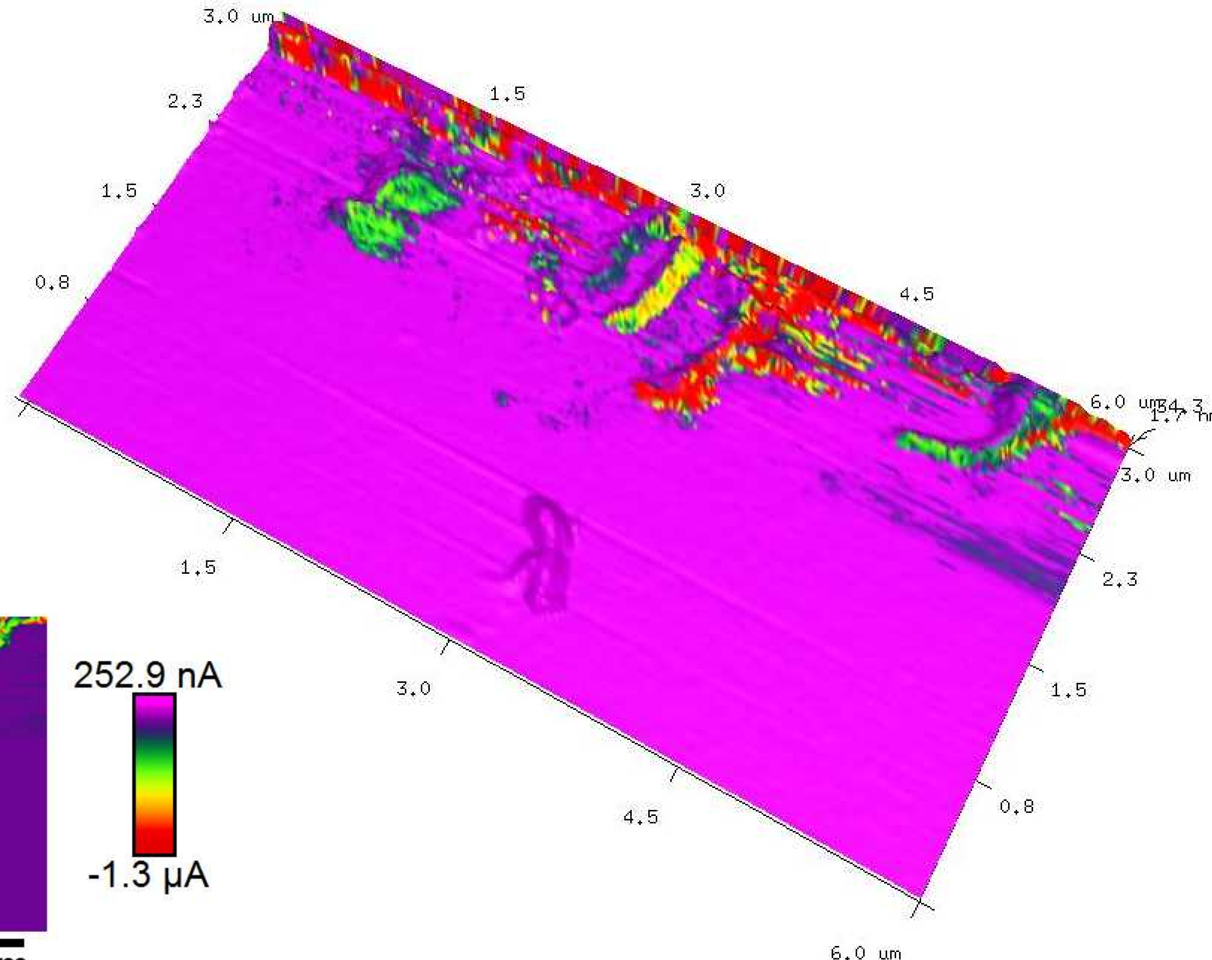


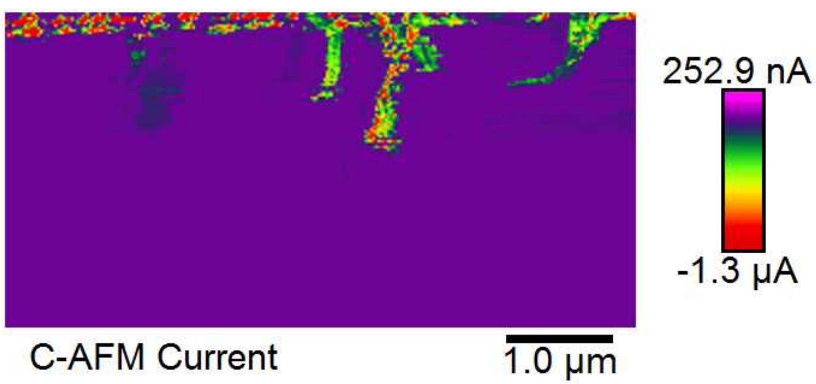




C-AFM Data

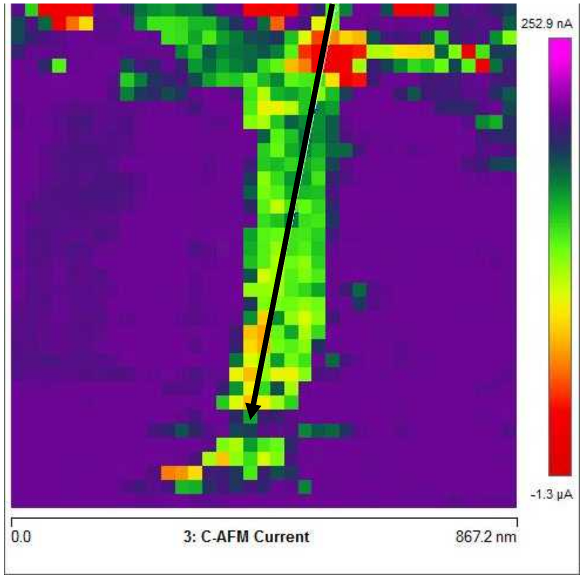
- Current mapping
  - Concurrent with topographic data
  - Easily characterize multiple MWCNTs as once



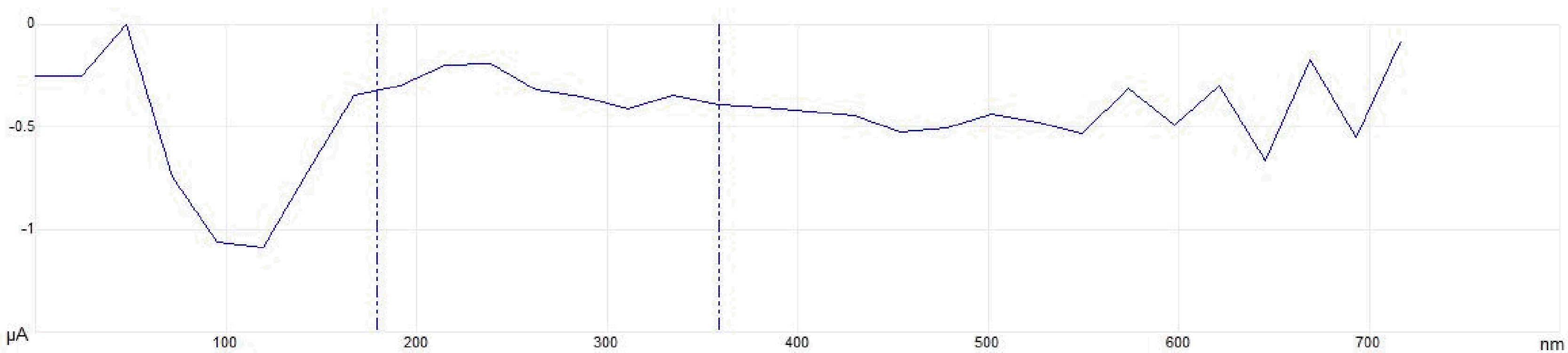


Mapping C-AFM Characterization

- Highly variable current measurements
  - Contact area of probe to MWCNT varies
    - Tip versus side contact
  - Difficult to determine where to place the trace to read out resistance per length
  - Additionally, the CNTs can move because they are not anchored to wafer

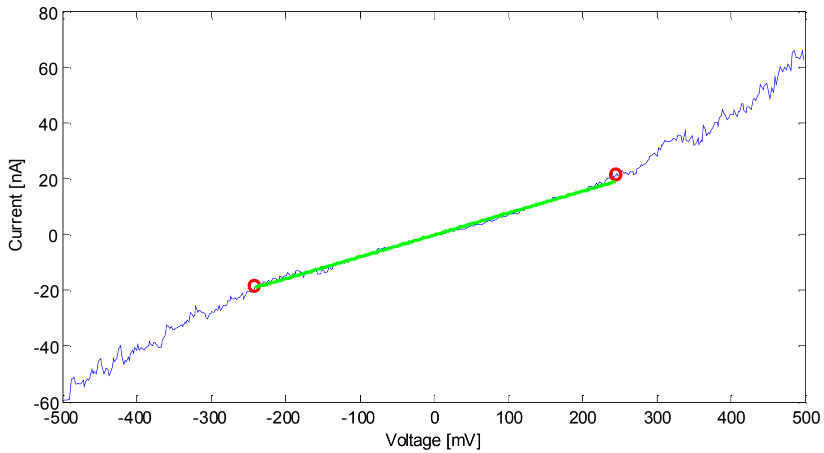


Current map indicating measurement trace

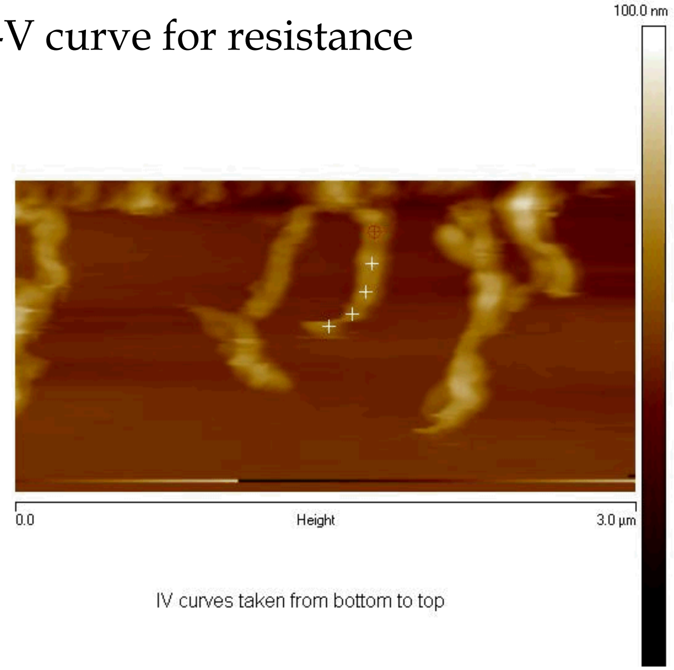


I-V Curve Characterization

- Strategy
  - Repeatable I-V measurements at 5 places along connected MWCNT
- 37 measurements taken among 5 pts
- Analysis
  - Linear fit conducted within linear region of I-V curve for resistance determination



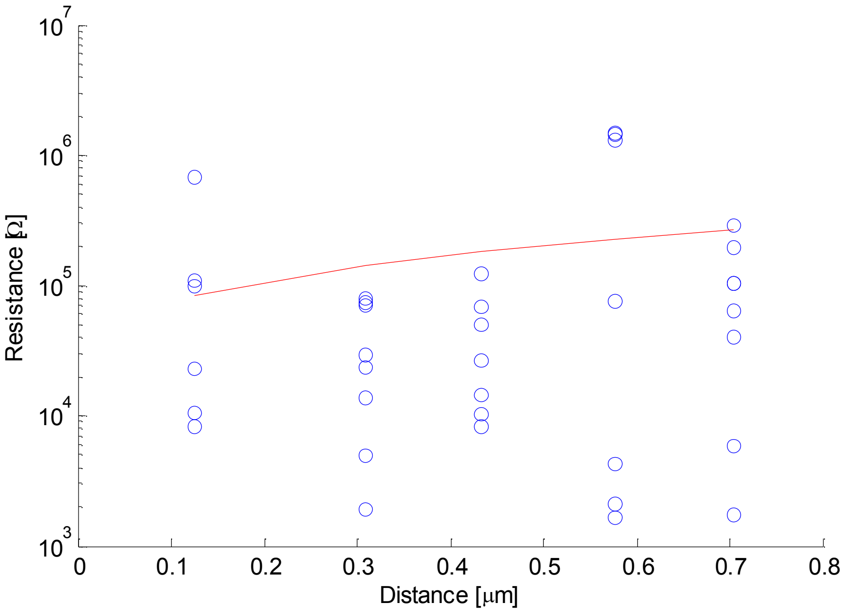
Sample I-V curve with linear fit region indicated



Topographic image of a connect MWCNT with I-V measurement locations indicated

Results

- Collected Data
  - High degree of scatter
    - Contact area/resistance
- Linear fit to data
  - Resistance per length
    - 315 kΩ/µm
  - Junction Resistance
    - 44.16 k Ω
- Consistency
  - Resistance per length consistent with Moshkalev et. al., 2006
  - Junction resistance consistent minimum values obtained by Buldum and Lu, 2000



Conclusions / Next Steps

- Demonstrated ability to take resistance measurements of MWCNTs using C-AFM approach
- Determined that C-AFM approach is fraught with difficulties resulting from tip contact resistance
- I-V approach was successful in getting values consistent with others in literature
- Next Steps
  - We are finding success in using a non-contact kelvin probe approach
    - Solves contact variation problem
  - We are nearing finalization of data and will be publishing soon

Thank You!

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Questions?

Acknowledgements:

Center for Critical Application Sensing