

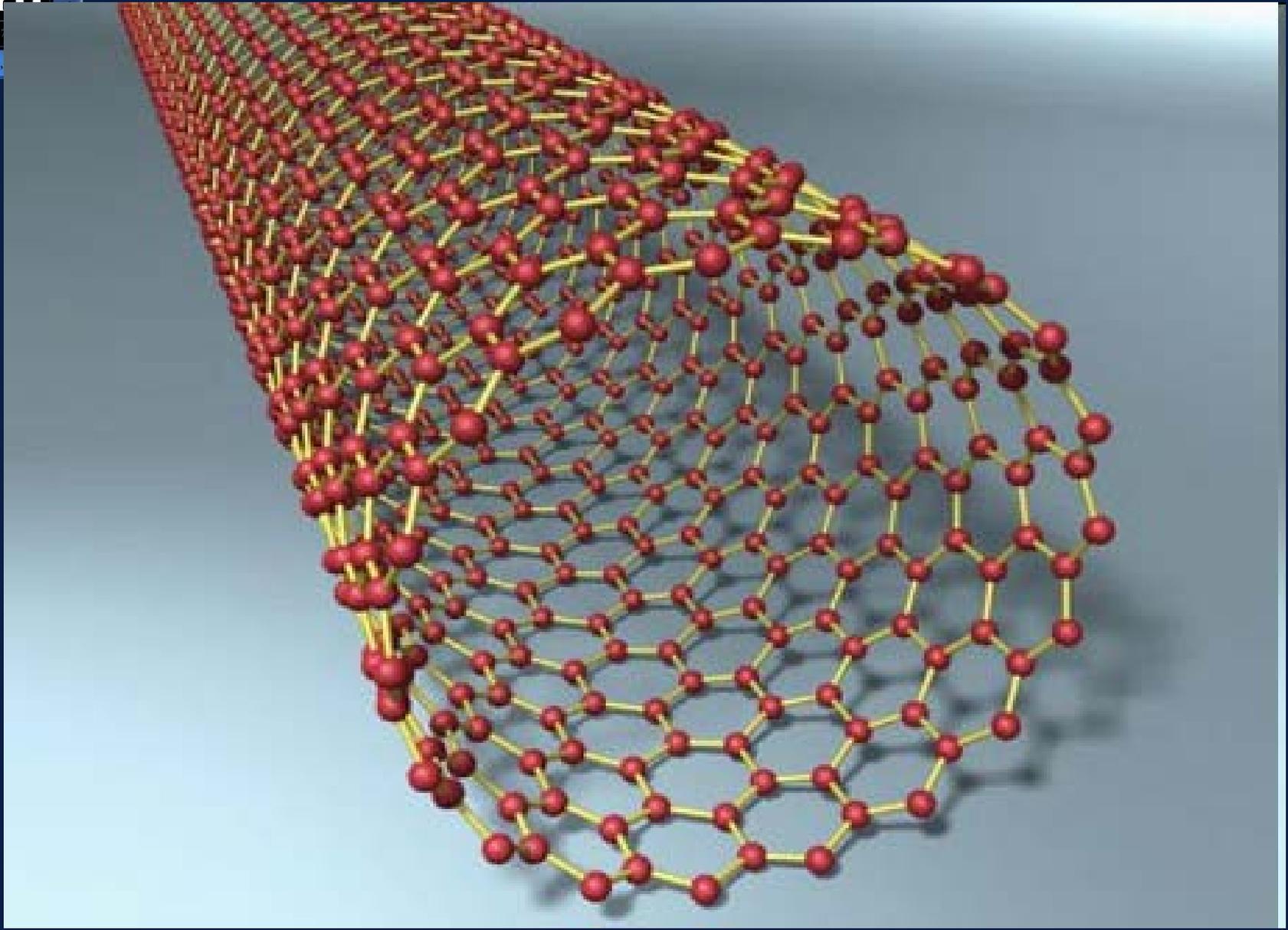


Applications of carbon nanotubes

David Allred

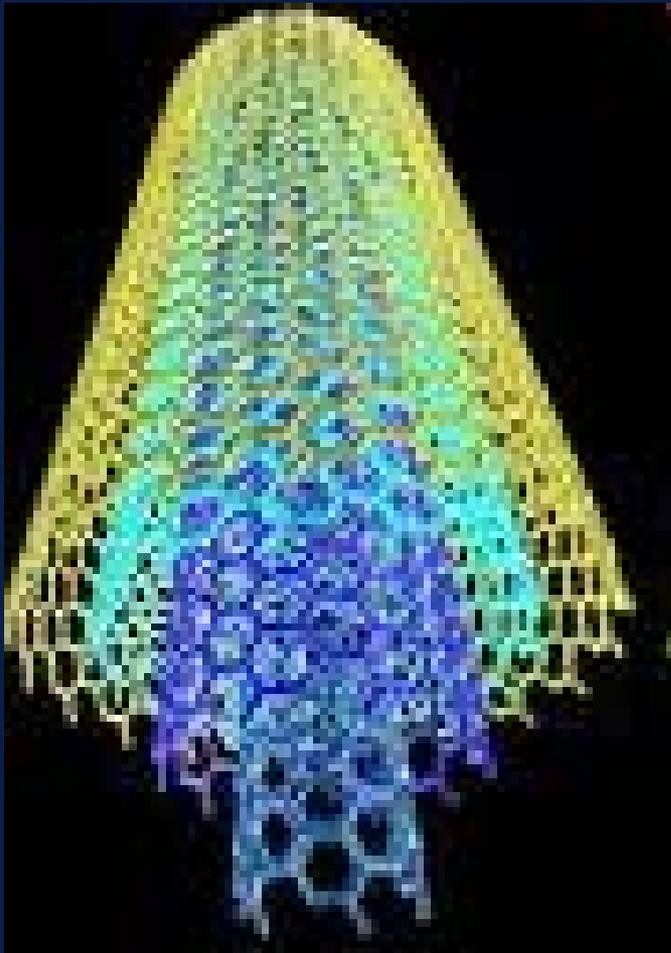
Dept of Physics & Astronomy, BYU

734-0418, 422-3489





Multiwall tubes







CVD system adaptation and qualification.

Differences at BYU

1. Furnace length and number of zones
2. Gas control via MFC- We calibrate.
3. Tube Diameter same



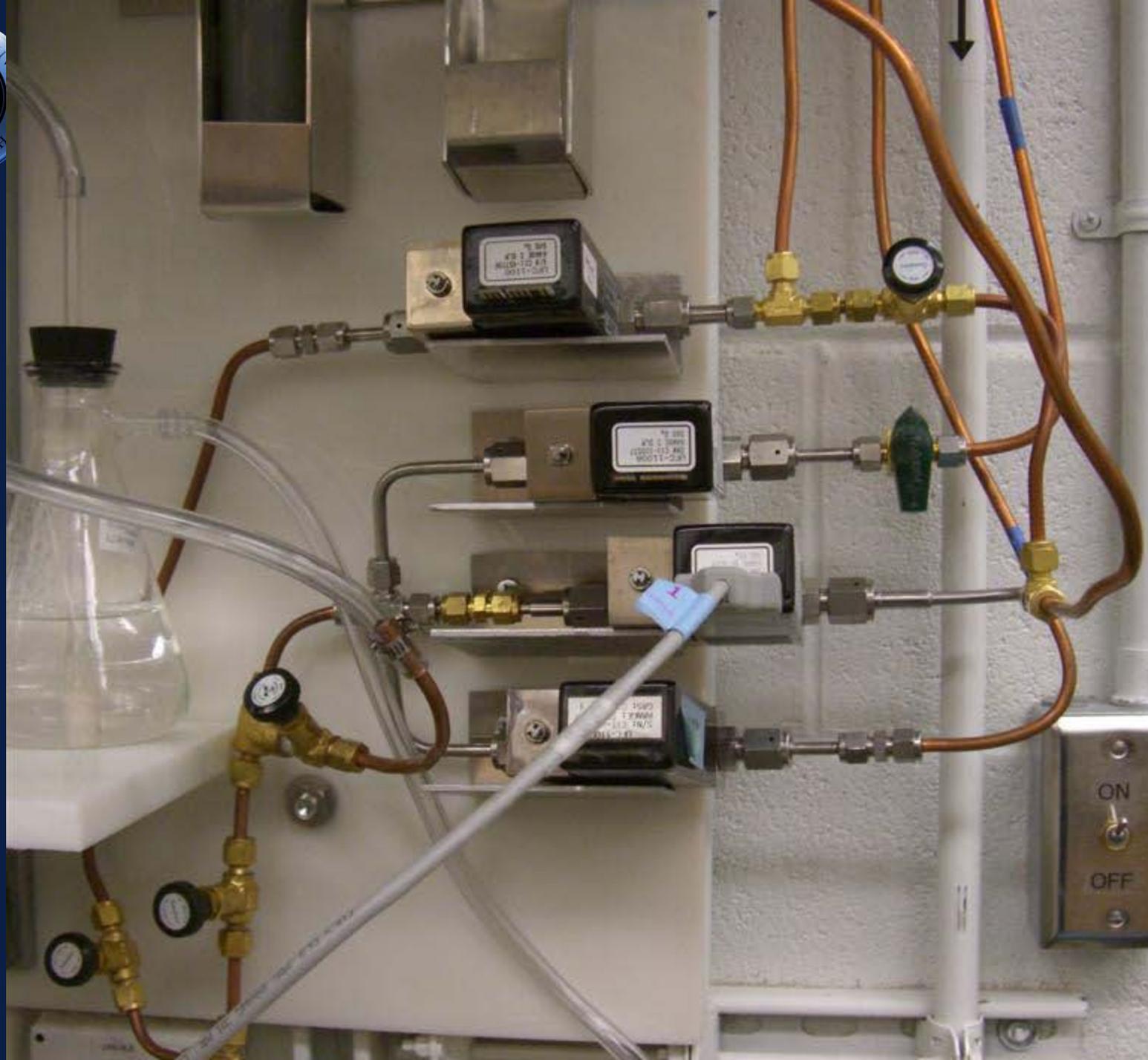




CVD system adaptation and qualification.

Differences at BYU

1. Furnace length and number of zones
2. Gas control via MFC- We calibrate.



Handwritten labels on the top panel of the instrument:

- Purge** (top row of switches)
- MFC output** (middle row of switches)
- Valve** (bottom row of switches)
- R output** (right side)
- 1, 2, 3** (position labels for various controls)
- OFF = CLOSED** (label for the middle valve switch)

A yellow sticky note is attached to the right side of the panel with handwritten text:

Purge
Ethylene
Hydrogen

UNIT INSTRUMENTS URS-100-5

Five digital displays with handwritten labels below them:

- 1: 02.4
- 2: 00.0
- 3: 00.1
- 4: 00.0
- 5: 00.0

Each display has a corresponding control knob and a SET/OFF/MANUAL switch below it.





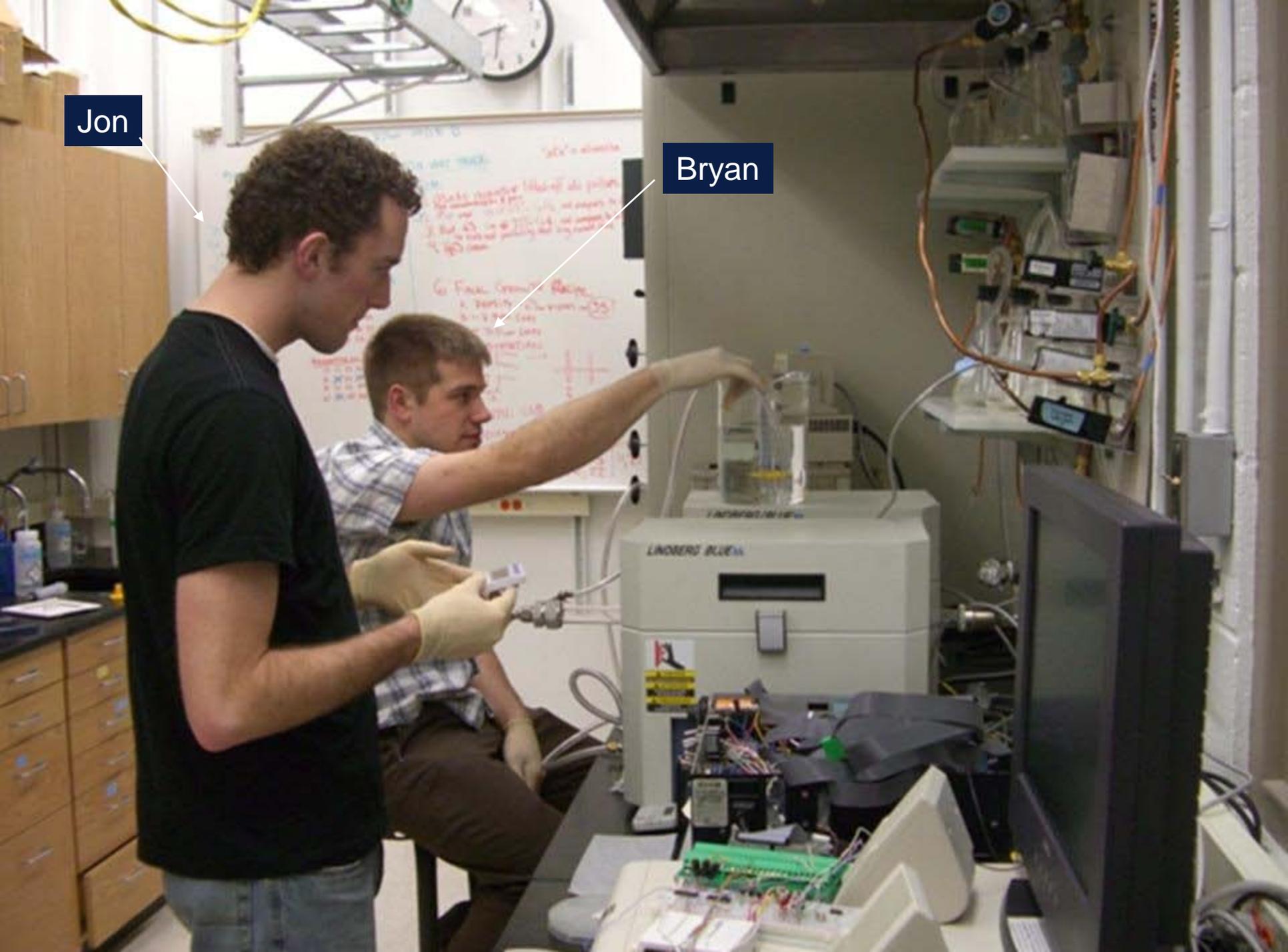
CVD system adaptation and qualification.

Differences at BYU

1. Furnace length and number of zones
2. Gas control via MFC- **We calibrate.**

Jon

Bryan





High Aspect Ratio Microfabrication by Chemical Infiltration of Carbon Nanotube Frameworks

MRS 2009 Talk by Prof. Robert C. Davis
Department of Physics and Astronomy
Brigham Young University

allred@byu.edu



CNT MEMS Researchers

BYU Physics

- David Hutchison
- Brendan Turner
- Katherine Hurd
- Matthew Carter
- Nick Morrill
- **Jun Song**
- Adam Konniker
- Ricky Wymant
- Taylor Wood
- **Dr. Richard Vanfleet**
- **Dr. Robert Davis**
- **Dr. David Allred**

BYU Mechanical Engineering

- Quentin Aten
- Dr. Brian Jensen
- Dr. Larry Howell

National Science Foundation

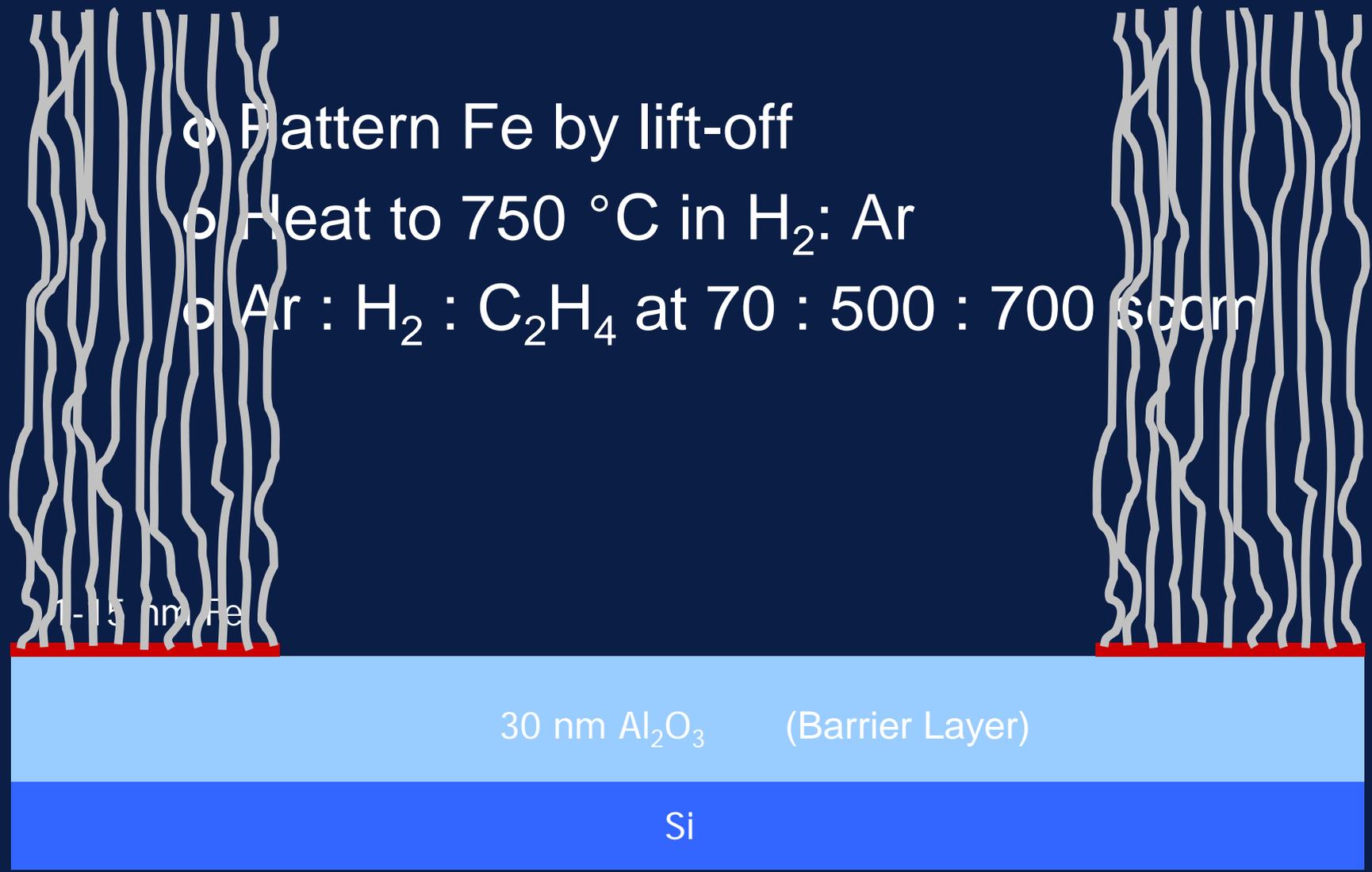
Brigham Young University
Environment for Undergraduate
Mentoring Grant

Partial funding provided by Moxtek
Inc.



VACNT growth details

- o Pattern Fe by lift-off
- o Heat to 750 °C in H₂: Ar
- o Ar : H₂ : C₂H₄ at 70 : 500 : 700 sccm



1-15 nm Fe

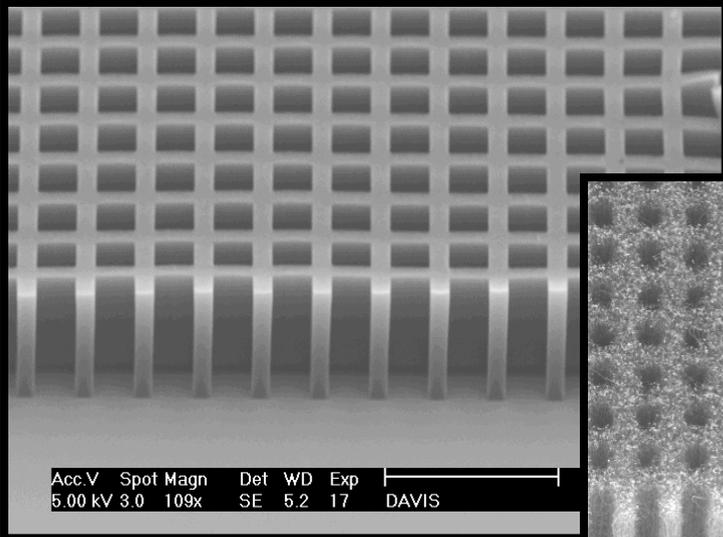
30 nm Al₂O₃ (Barrier Layer)

Si

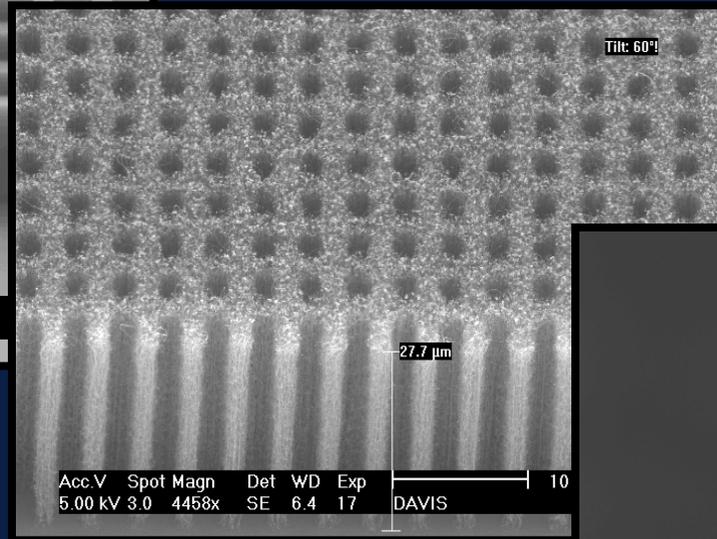


SEM - VACNT forest

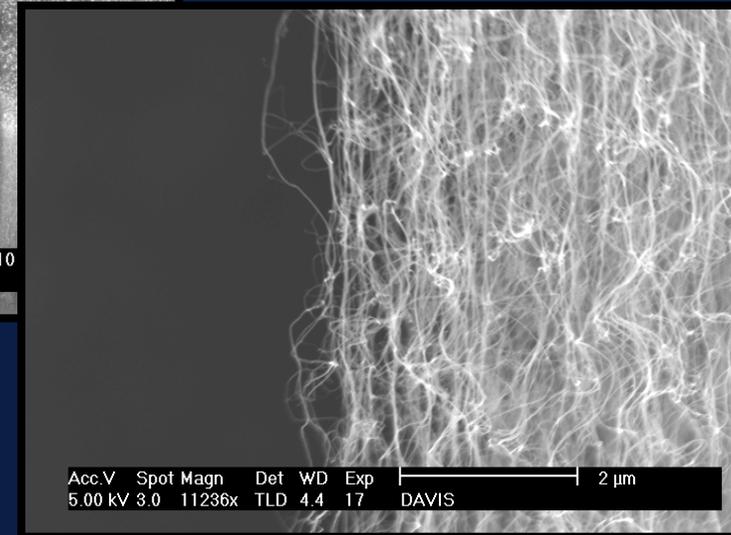
Can top surface and edge roughness be improved?



100X – 50 μm holes



4500X – 3 μm holes

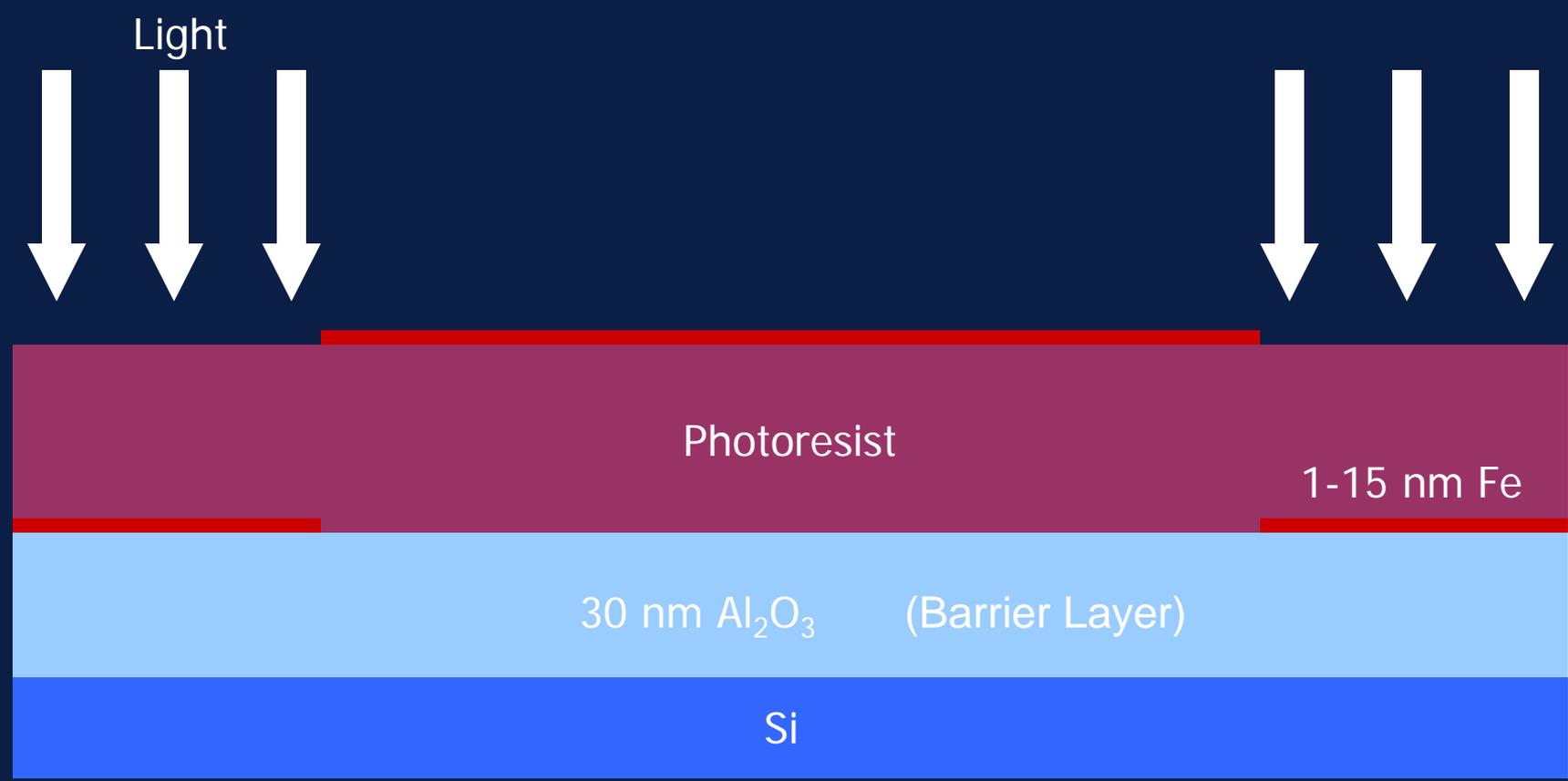


30000X – edge



VACNT growth process

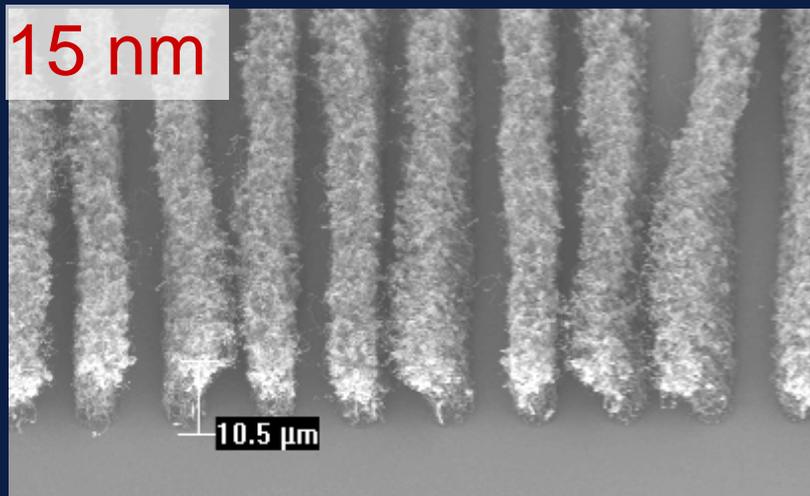
- o Photolithography & lift-off



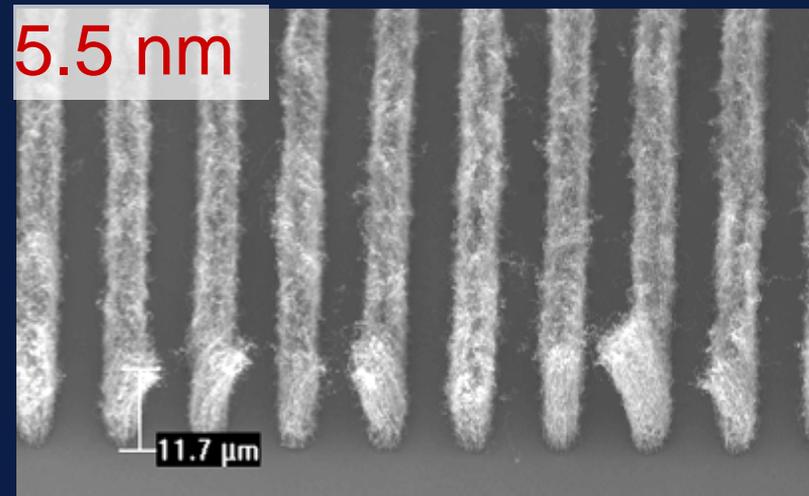


Dependence on Fe Thickness

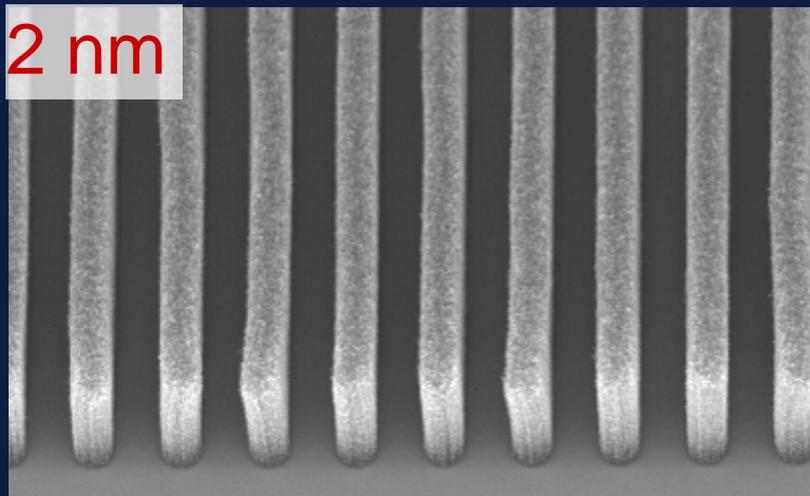
15 nm



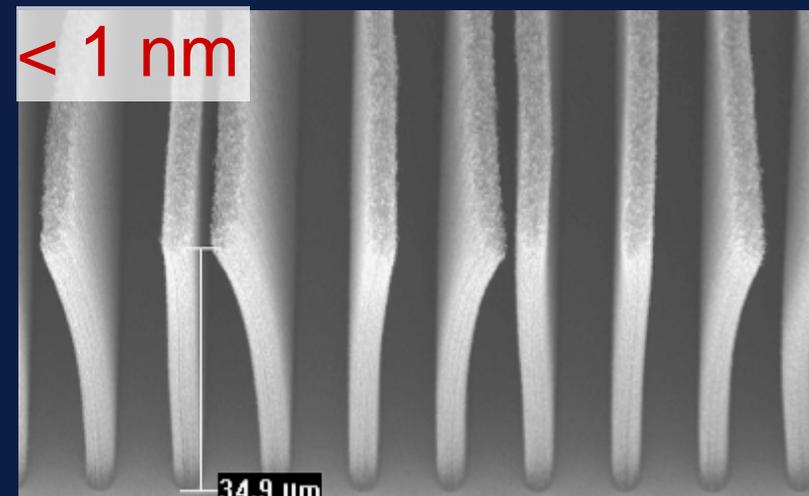
5.5 nm



2 nm



< 1 nm



Good repeatability

Rate strongly dependant on thickness

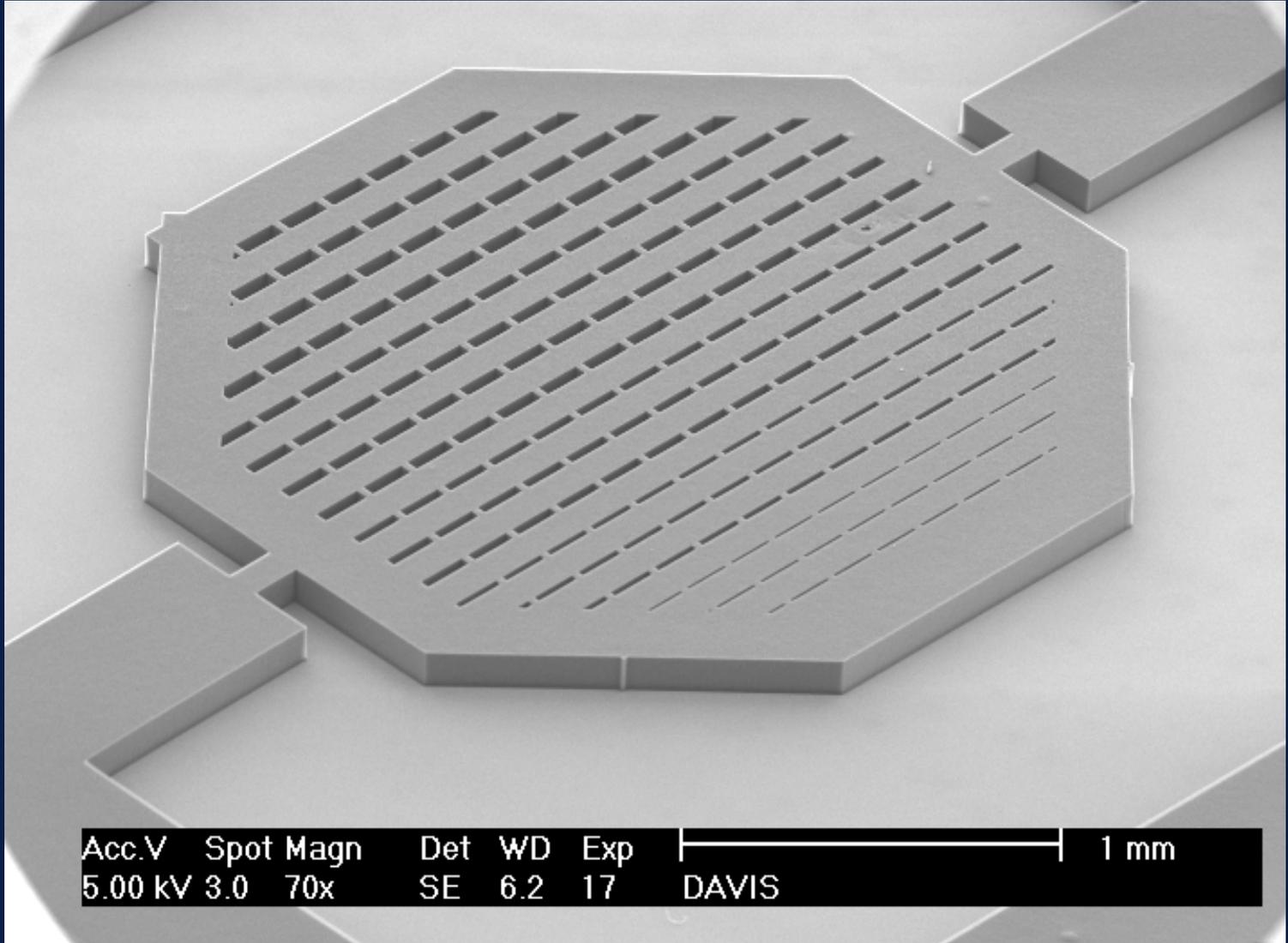


2 nm Fe on Al₂O₃

- Small voids (< 200 nm across)
- Sharp features (few stray tubes);
Sidewall roughness < 200 nm
- High growth rate ~50 μm/min



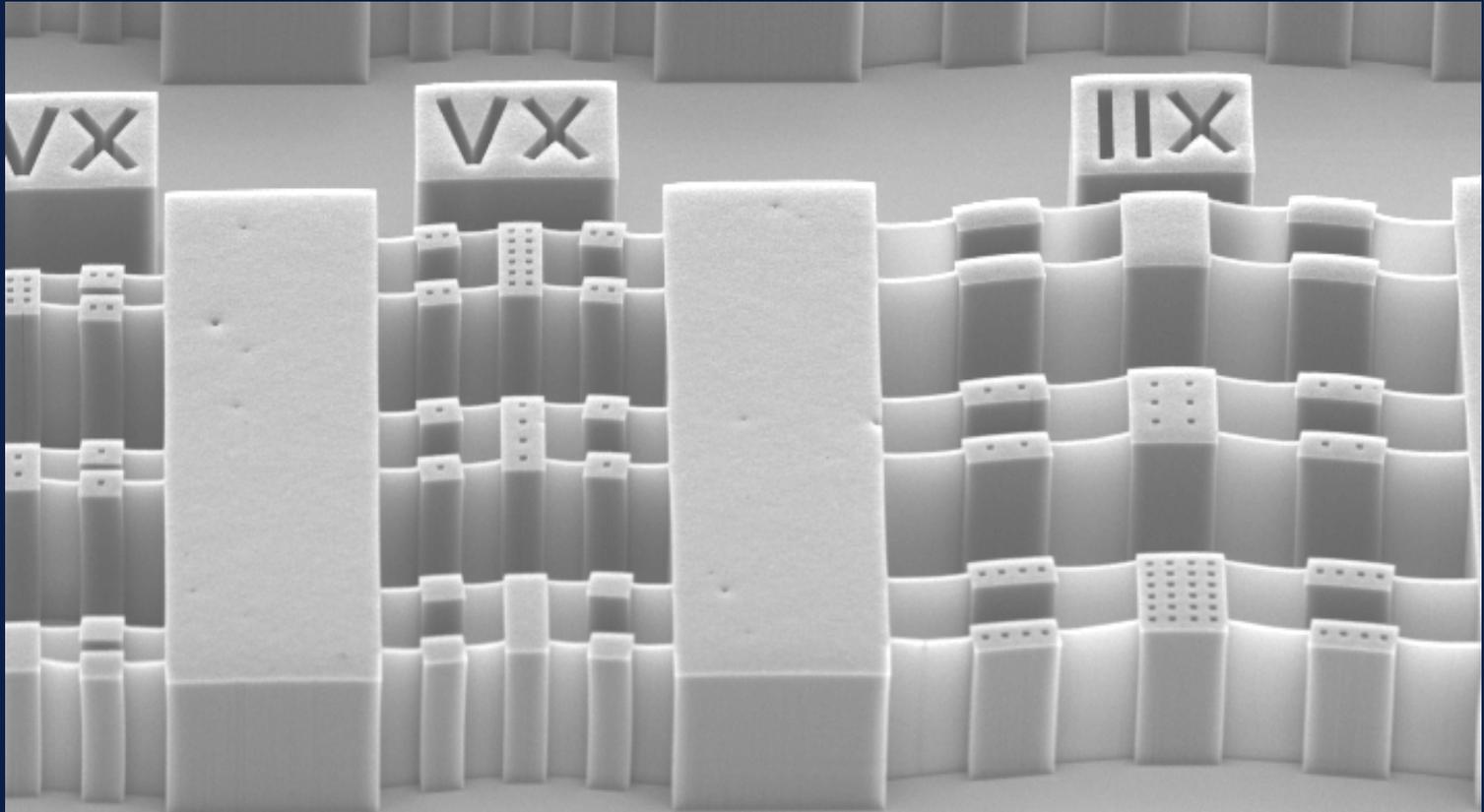
TEM Grid



Acc.V	Spot	Magn	Det	WD	Exp	-----		1 mm
5.00 kV	3.0	70x	SE	6.2	17	DAVIS		



Bistable Mechanisms



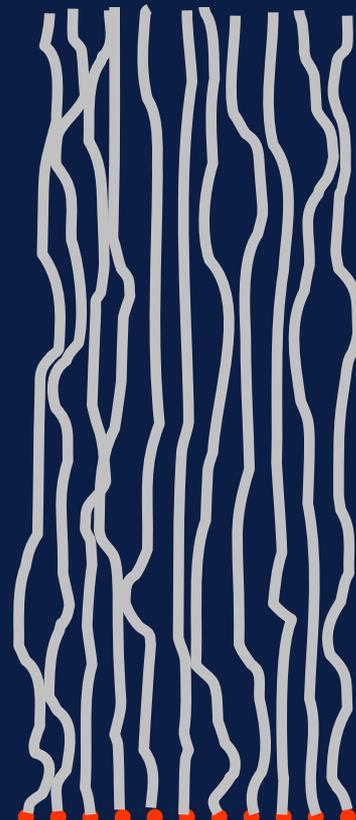
Acc.V	Spot	Magn	Det	WD	Exp		500 μ m
5.00 kV	3.0	134x	SE	7.8	17	DAVIS	



Nanotube “forest” growth



- Height: up to 1mm+
- Feature size: a few microns
- Speed: 10-100 $\mu\text{m}/\text{min}$
- Density:



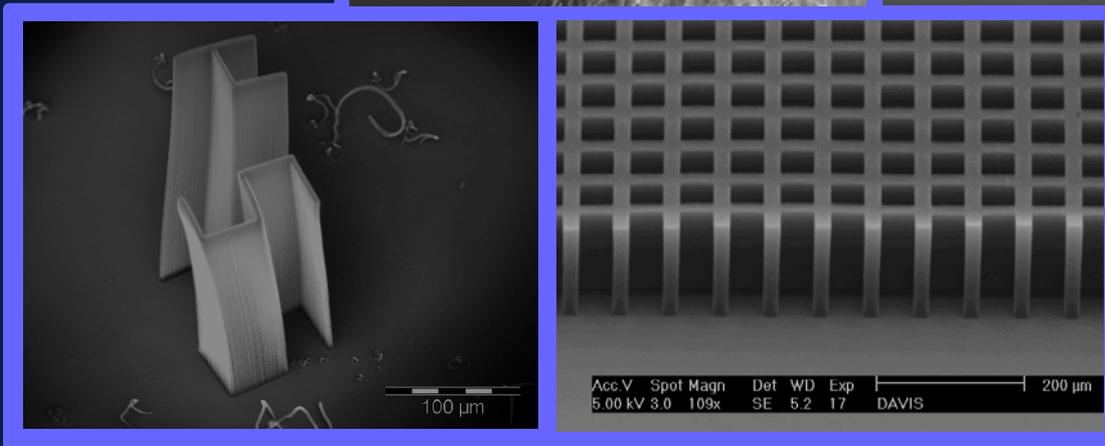
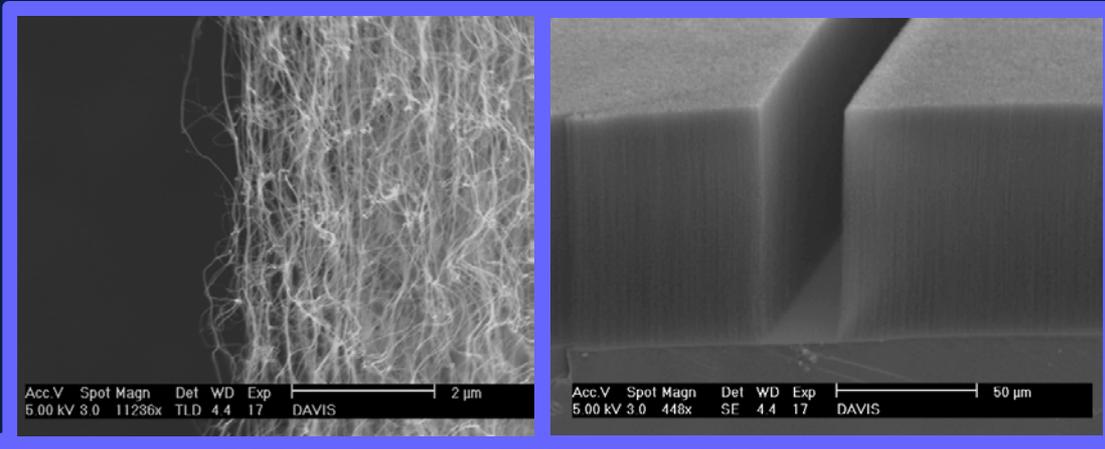
Material	Density (kg/m ³)
Air	1.2
Silica aerogel: lowest density	1.9
Measured density	9.0
Silica aerogel: usual density range	5 – 200
Expanded polystyrene	25 – 200





Vertically Aligned Carbon Nanotube (VACNT) Growth

Individual nanotubes wander but...



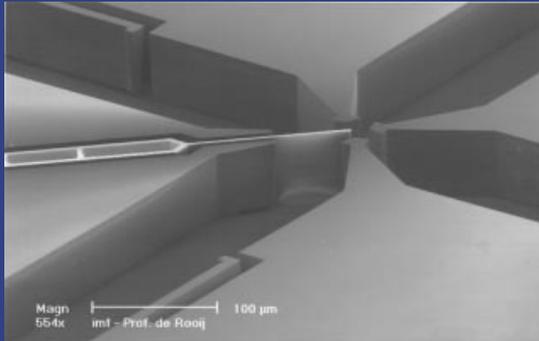
...forest grows perpendicular to growth substrate

Extraordinary growth among materials growth systems



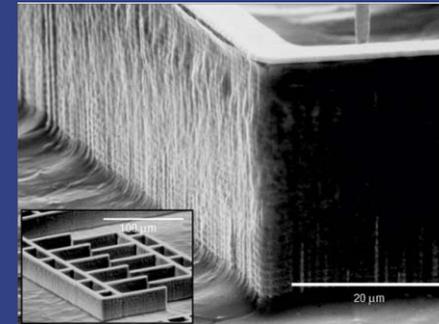
High Aspect Ratio Micromachining

Deep Reactive Ion Etching (Si)



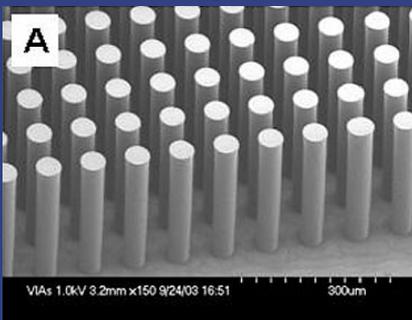
“Vertical Mirrors Fabricated by DRIE for Fiber-Optic Switching Applications,” C. Marx et al., *J. MEMS* **6**, 277, (1997)

MARIO Process (Titanium)



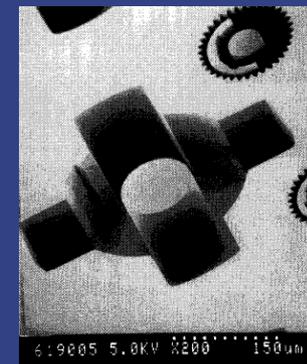
“High-aspect-ratio bulk micromachining of titanium,” Aimi et al., *Nature Mat.* **3**, 103-5 (2004)

SU-8 / C-MEMS (photoresist / carbon)



“C-MEMS for the Manufacture of 3D Microbatteries,” Wang et al., *Electrochem. Solid-State Lett.* **7** (11) A435-8 (2004)

LIGA process (photoresist)

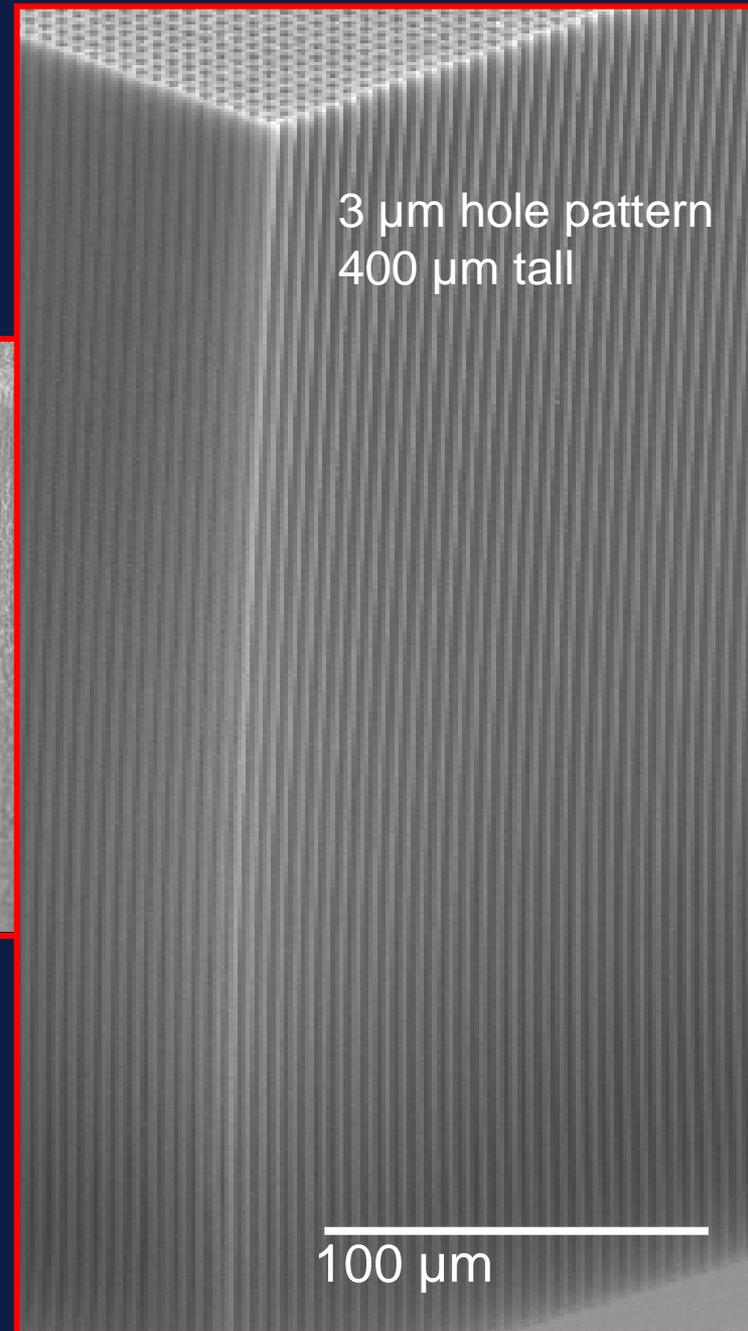
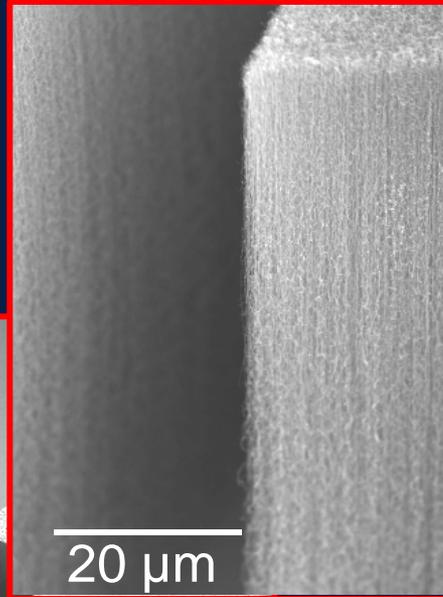
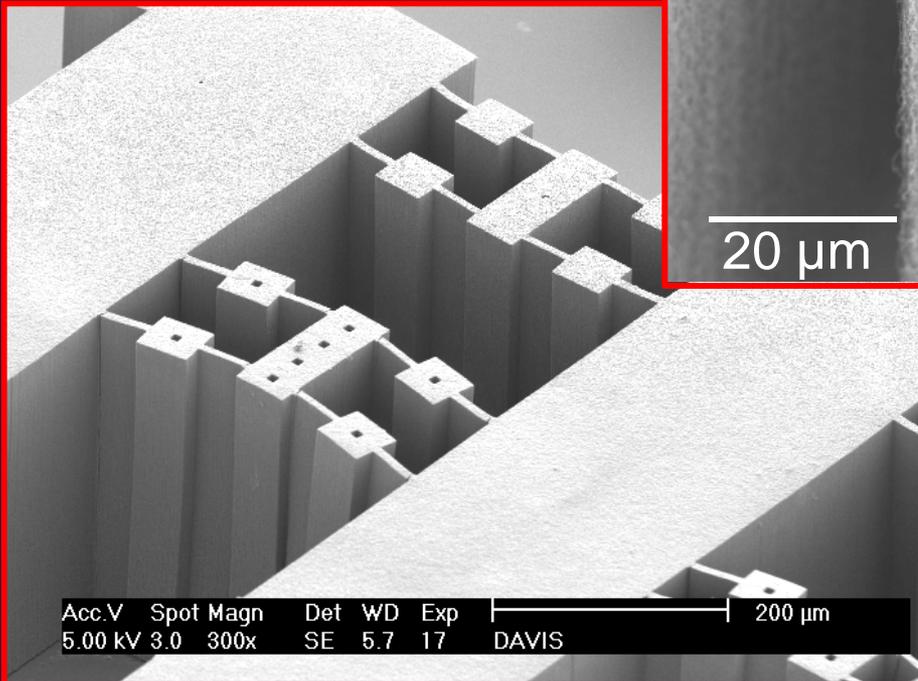


“Micromechanisms,” H. Guckel, *Phil. Trans. R. Soc. Lond.* **353**, 355-66 (1995)



VACNT: Extreme Aspect- Ratio Microstructures

- Smooth sidewalls ...
- <1 micron roughness
- Tall... up to several millimeters





Patterned forest structure



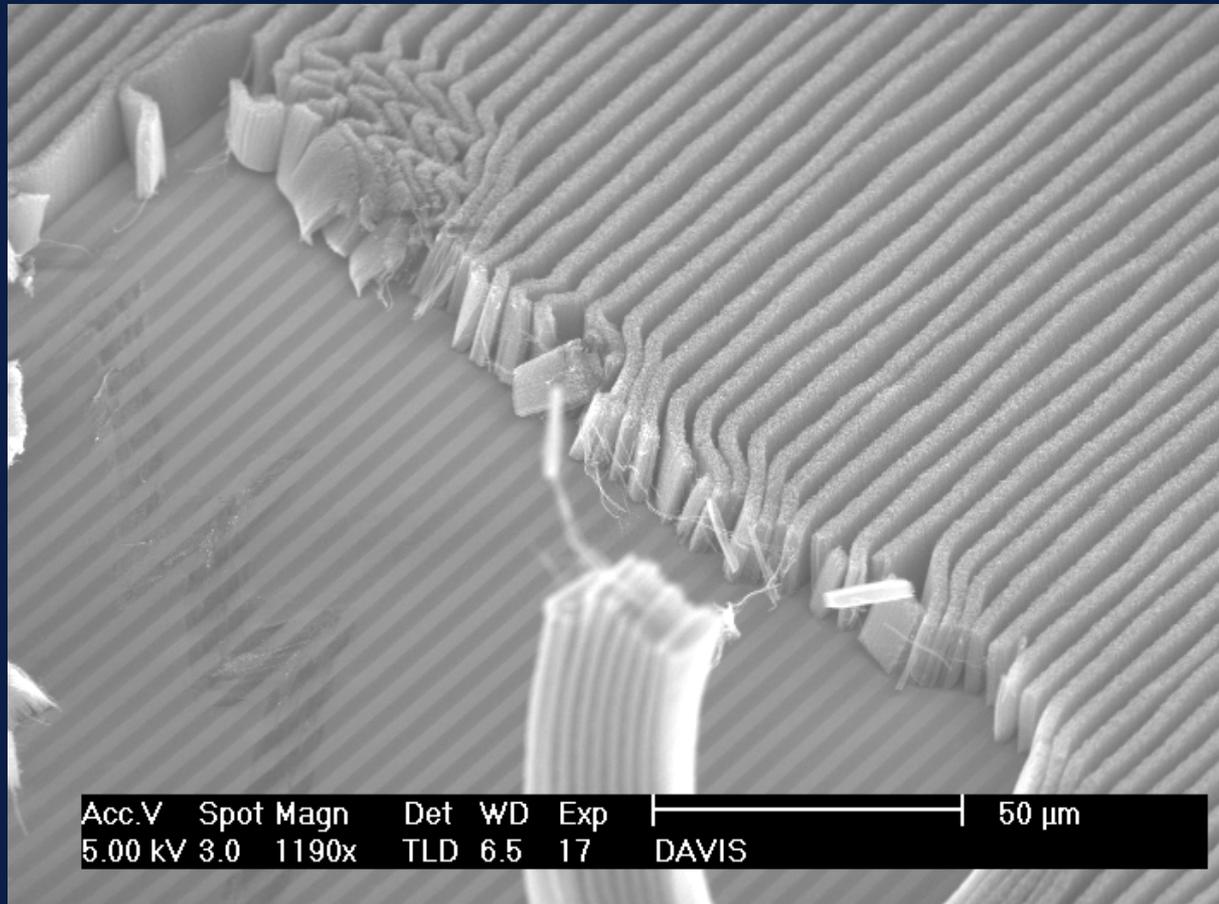
- Height: up to several millimeters
- Lateral feature size: down to 1 micron
- Speed: 10-100 $\mu\text{m}/\text{min}$
- Density:



Material	Density (kg/m ³)
Air	1.2
Silica aerogel: lowest density	1.9
Measured density	9.0
Silica aerogel: usual density range	5 – 200
Expanded polystyrene	25 – 200



Low density, weakly bound material



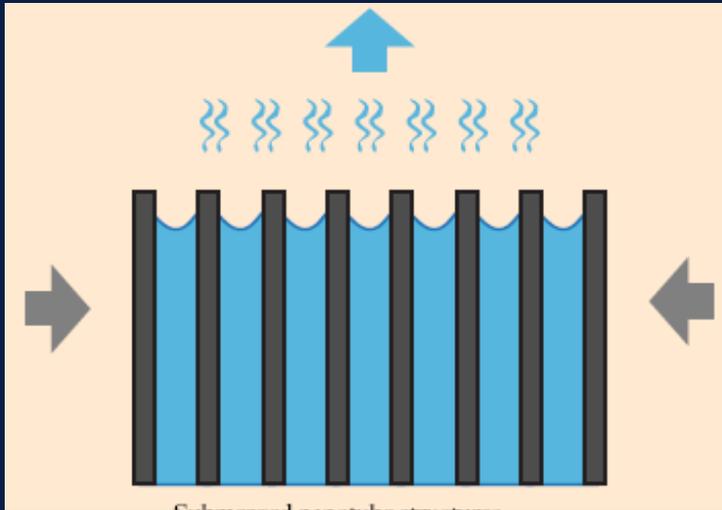
As-grown forests are flimsy and tear off the surface at the slightest touch



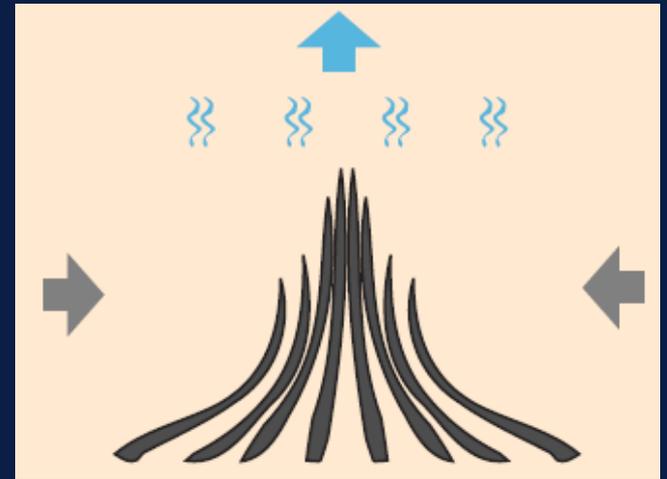
Dense Nanotube Structures



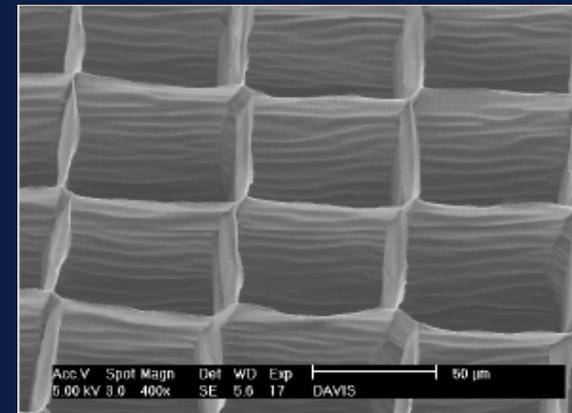
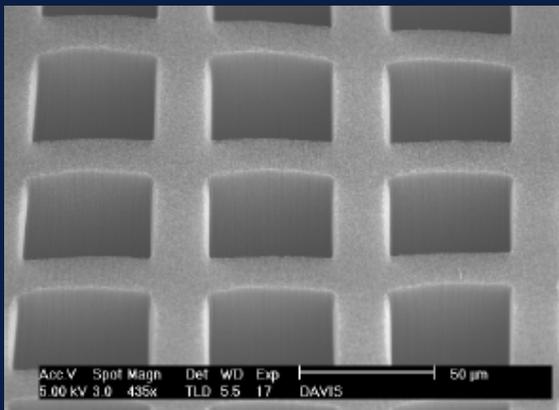
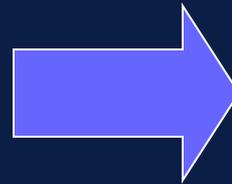
Liquid Induced Densification



Submerged nanotube structures

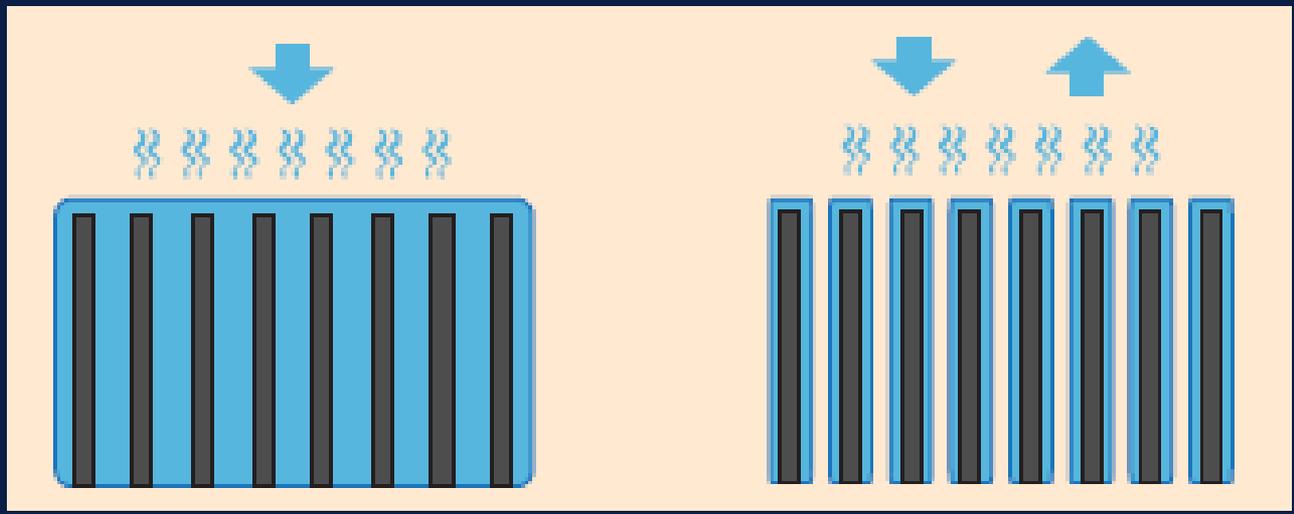


Dried structure



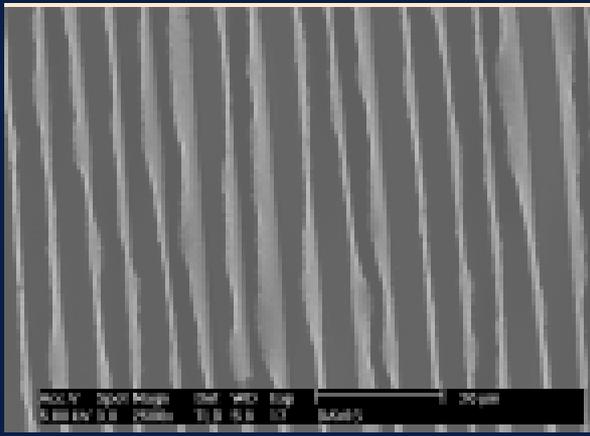
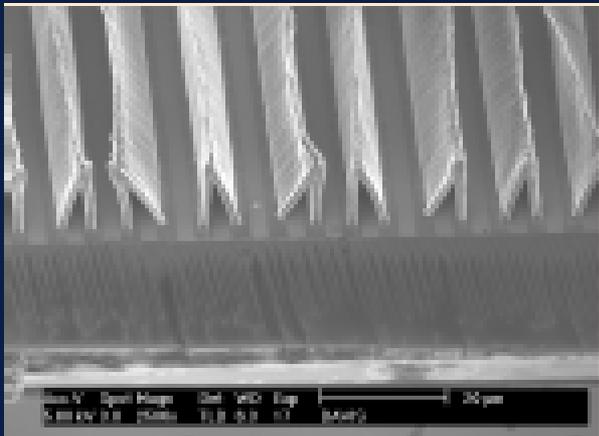


Vapor Condensation Induced Densification -- Lines



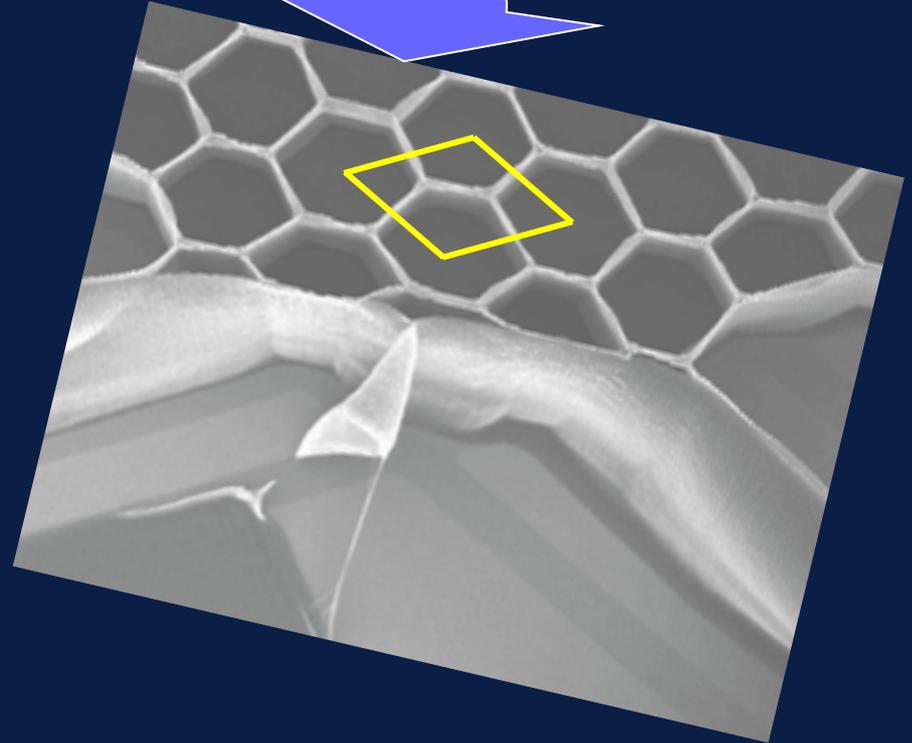
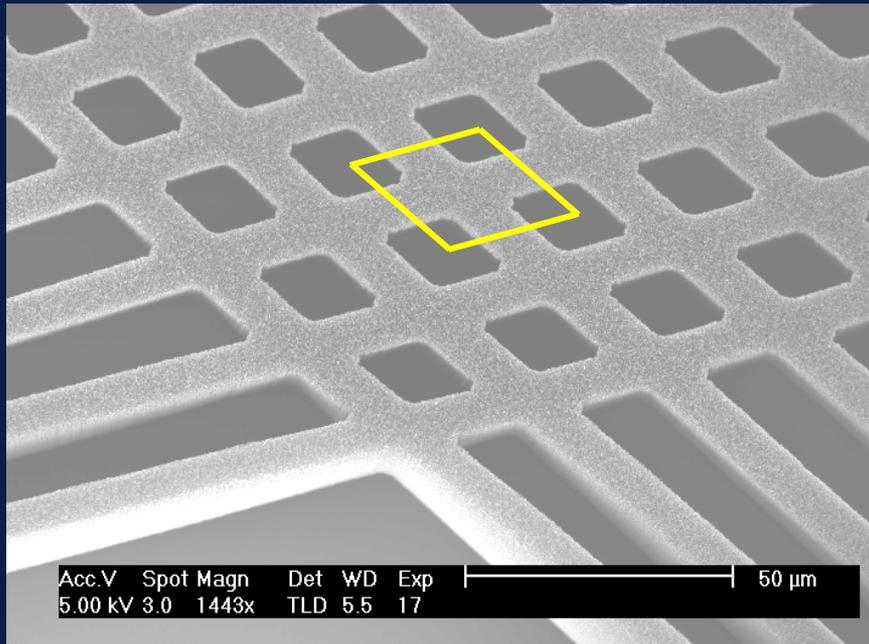
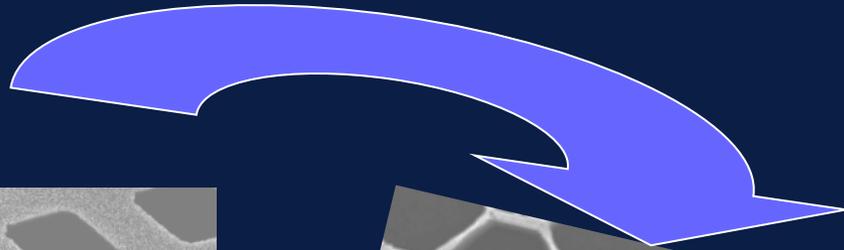
Longer exposure to vapor

Shorter exposure to vapor





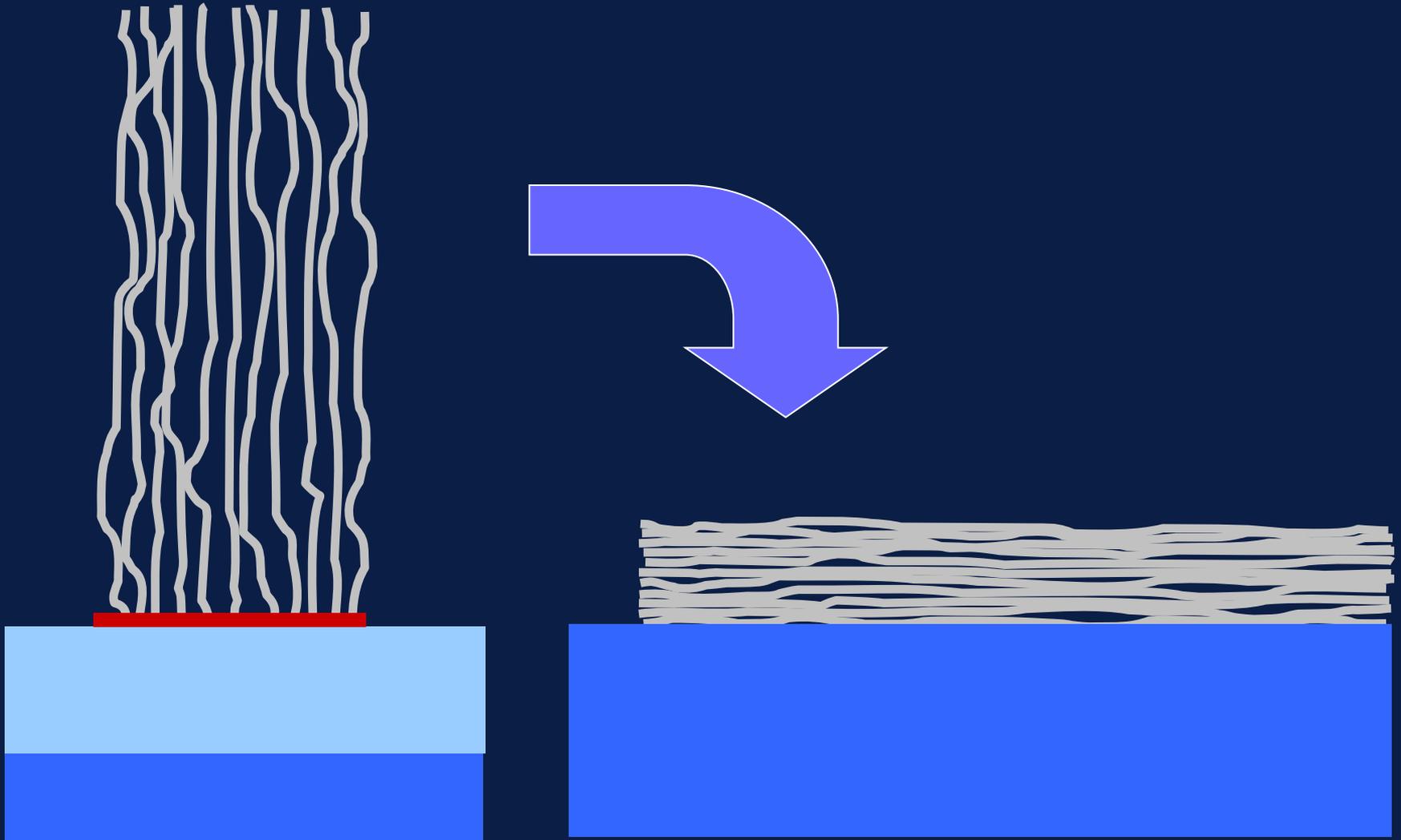
Surface forces dominates



Unequal angles become equal angles.
Final structure depends on initial structure surface forces
Difficult to control what results!

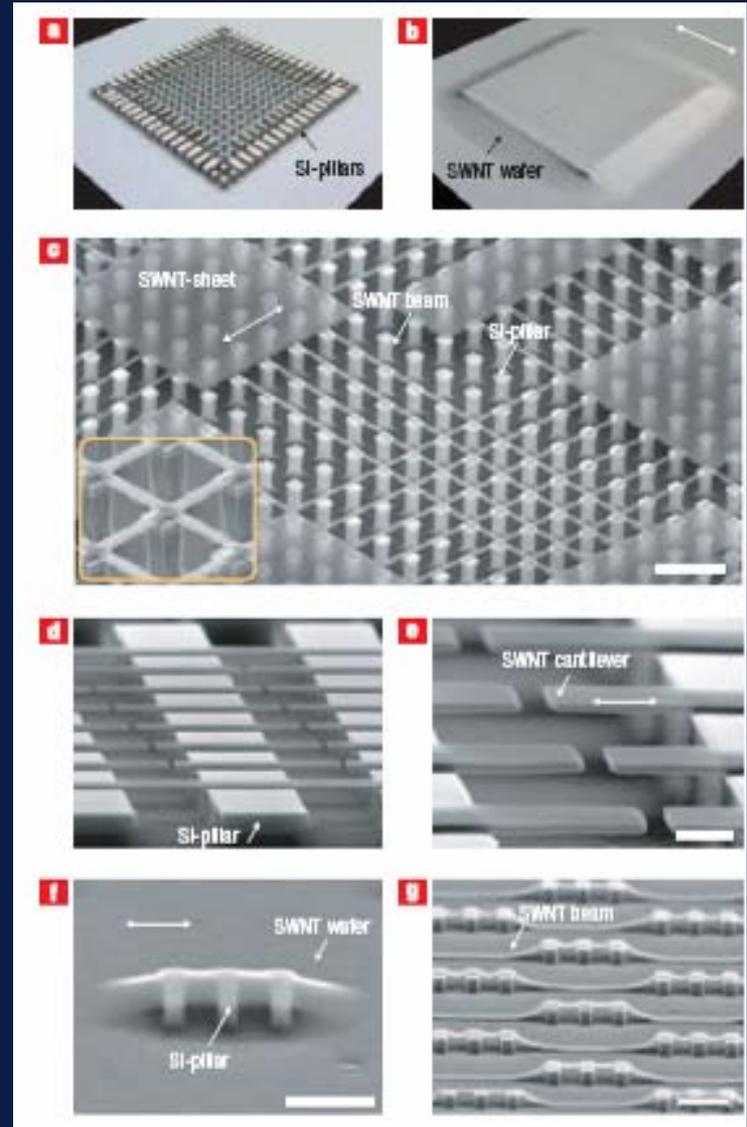
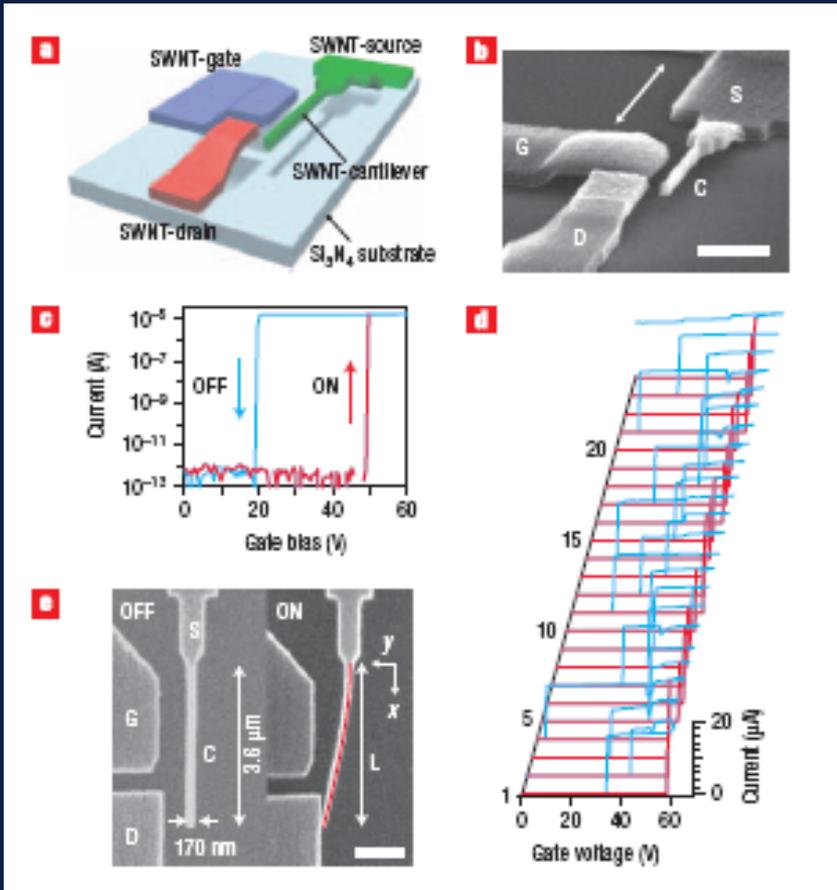


Horizontal Aligned CNT films





AIST Japan group working on in-plane aligned CNT MEMS



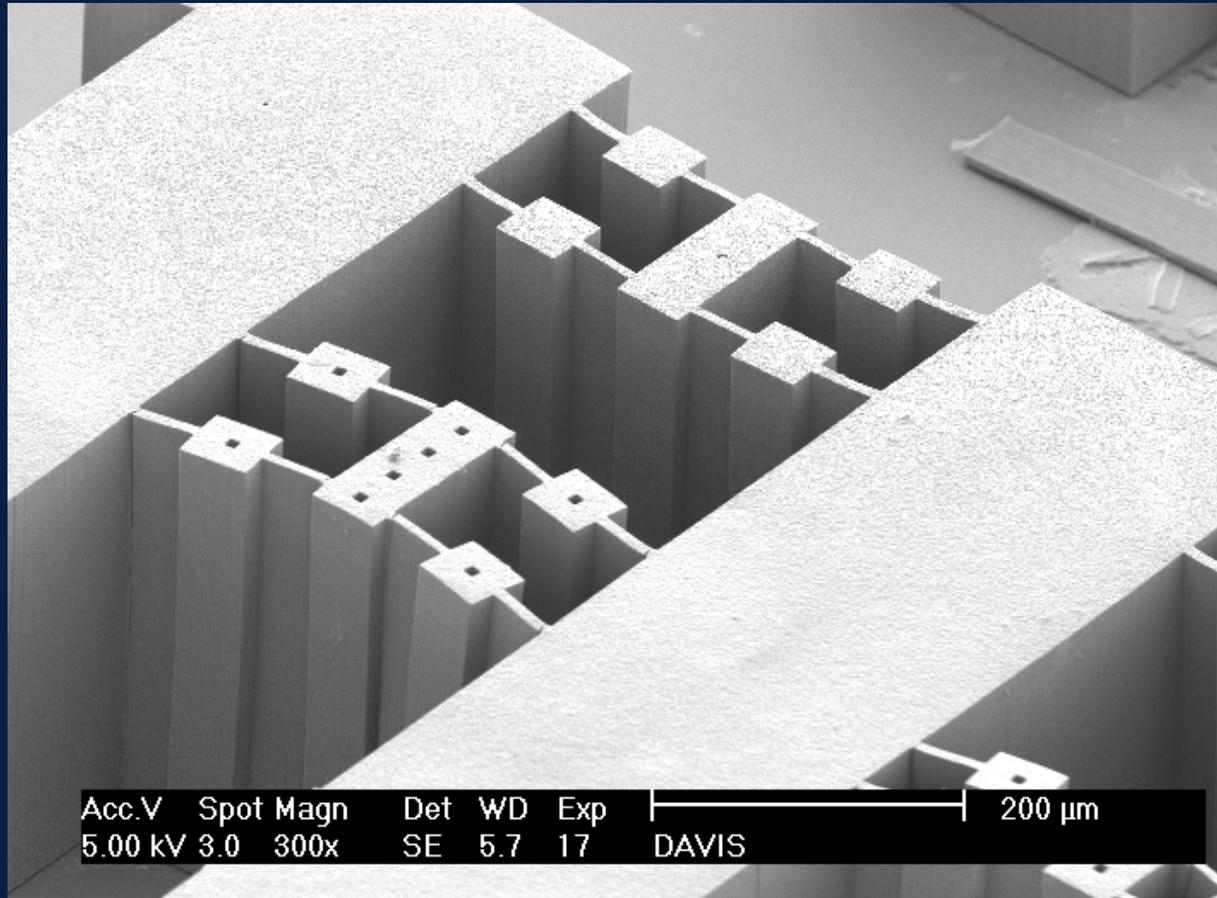
Yuhei Hayamizu, Takeo Yamada, Kohei Mizuno, Robert C. Davis, Don N. Futaba, Motoyuki Yumura, & Kenji Hata **Nature Nanotechnology** 3, 241 (2008).



Microstructured VACNT Composites



Leave the nanotubes vertical?





VACNT Composites

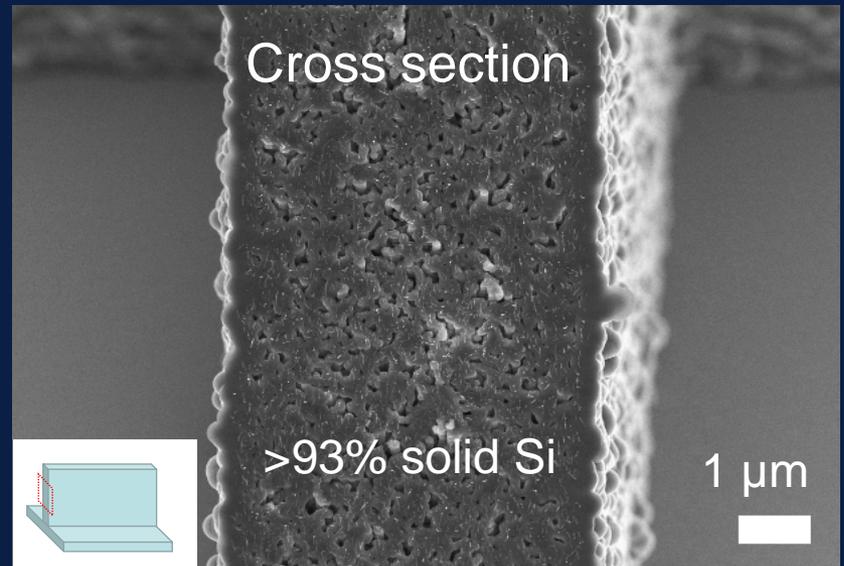
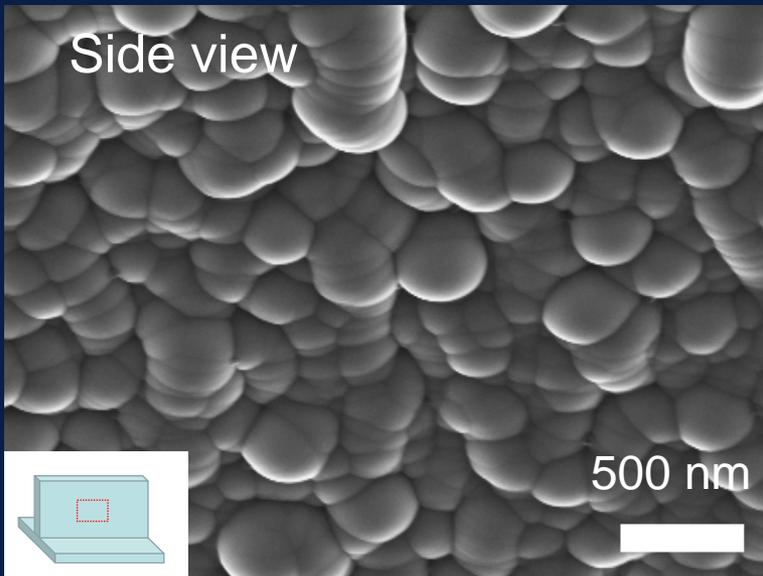
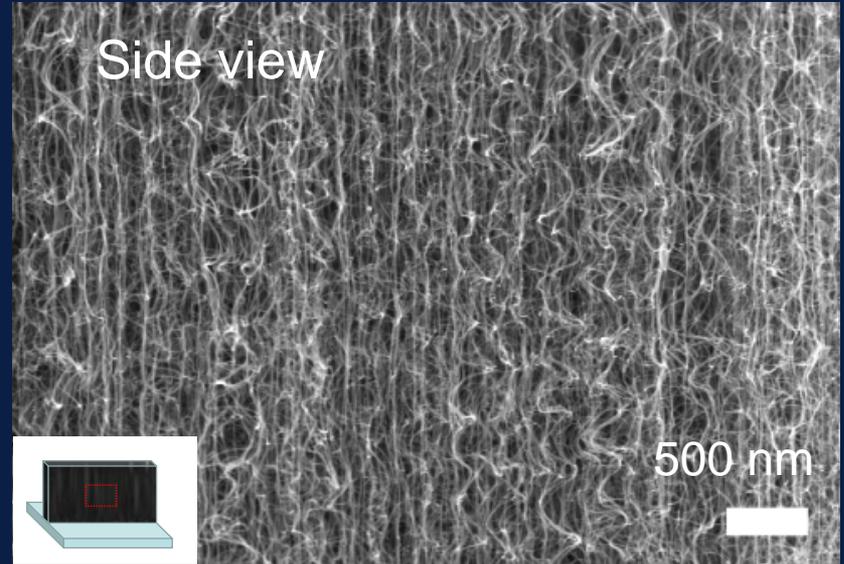
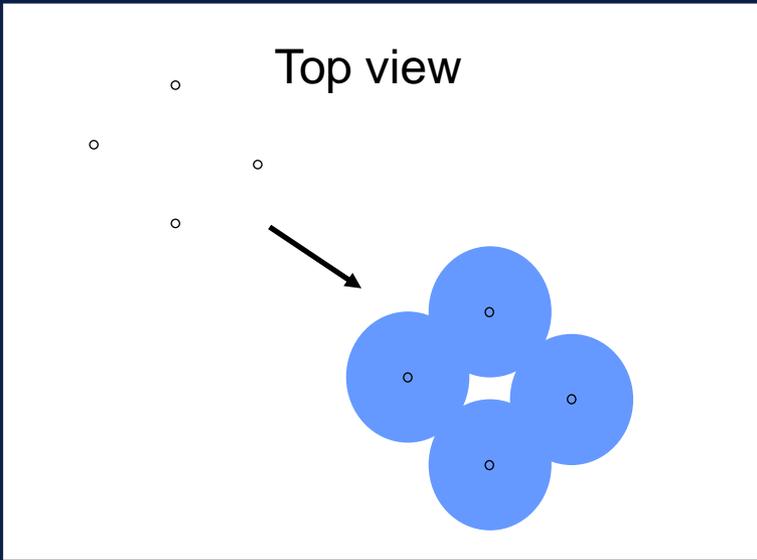


Filling in with Si by LPCVD



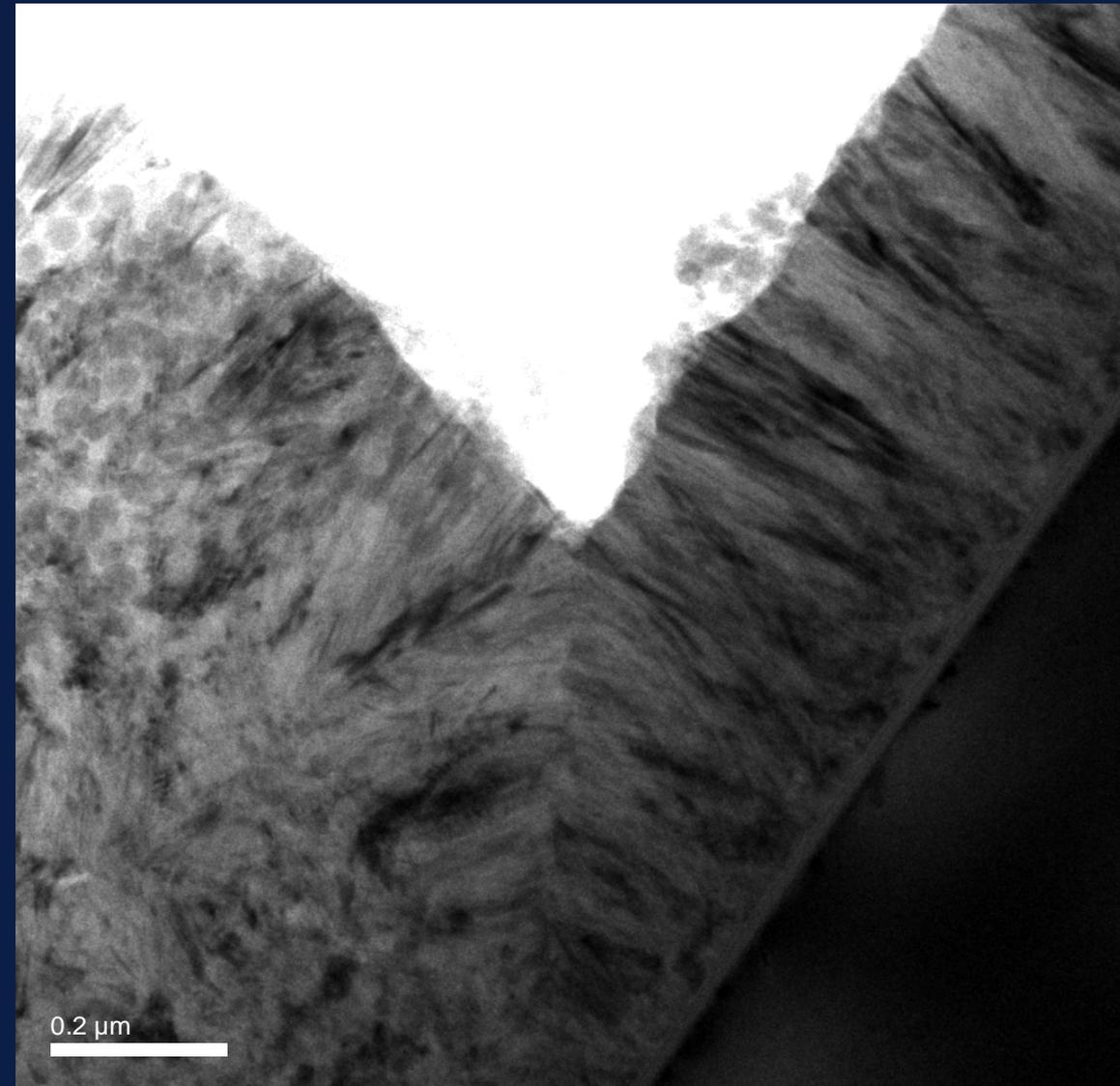


Nearly complete Si infiltration

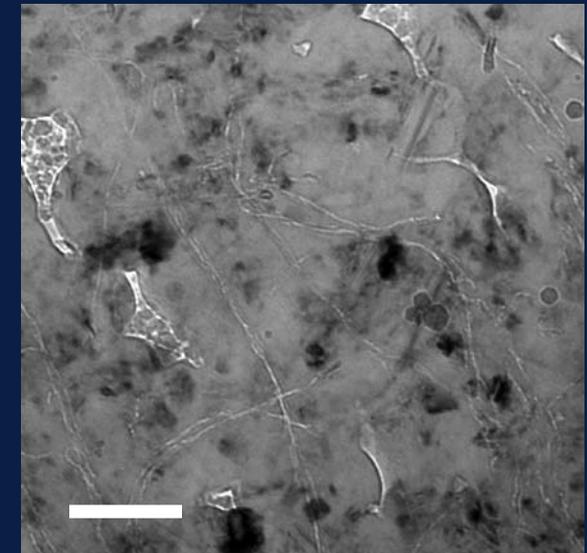
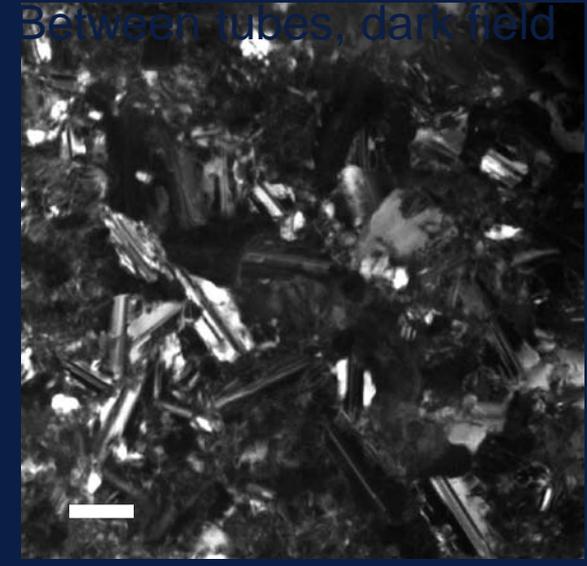




Microstructure: Poly-Si

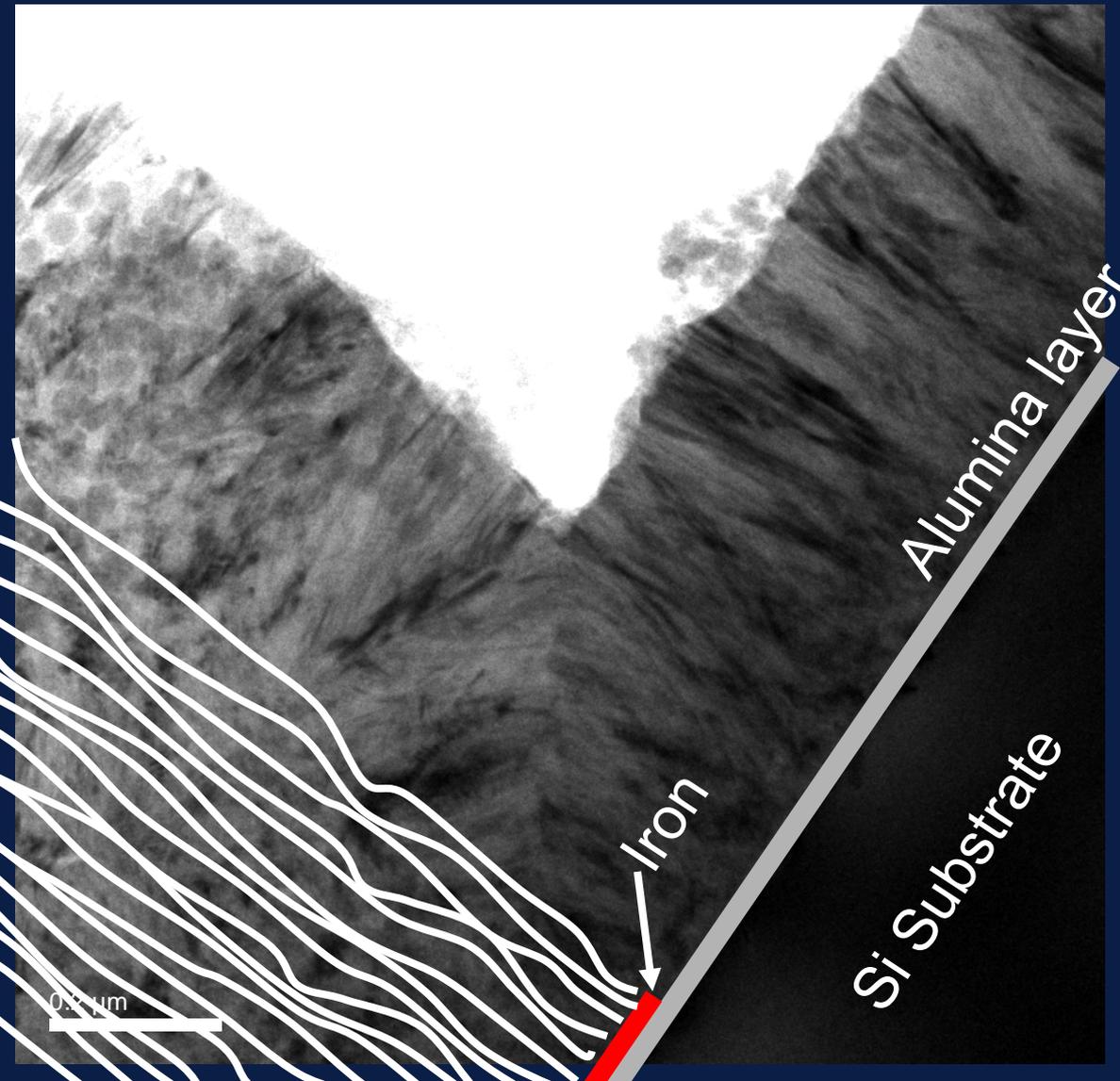


Between tubes, dark field

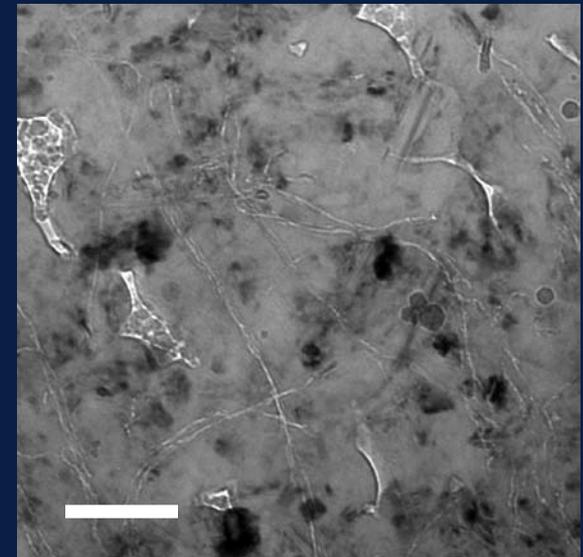
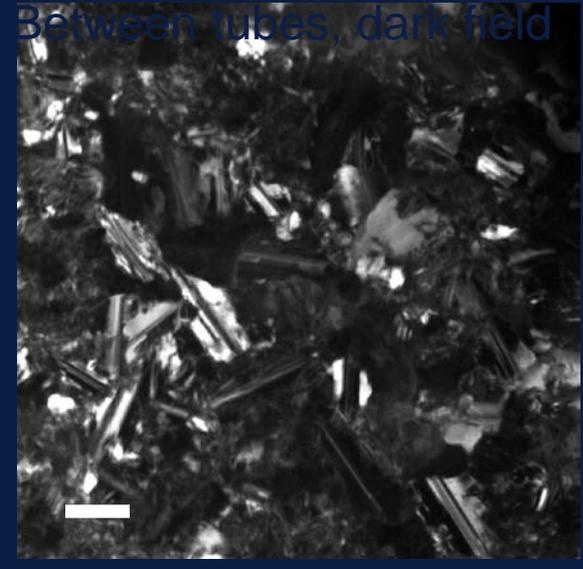




Properties: Poly-Si

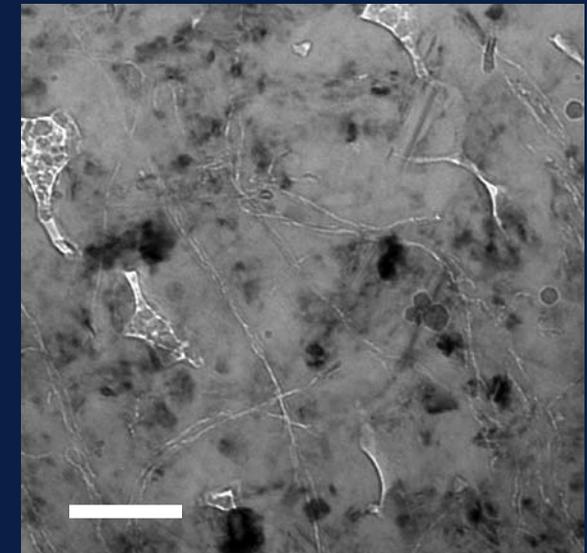
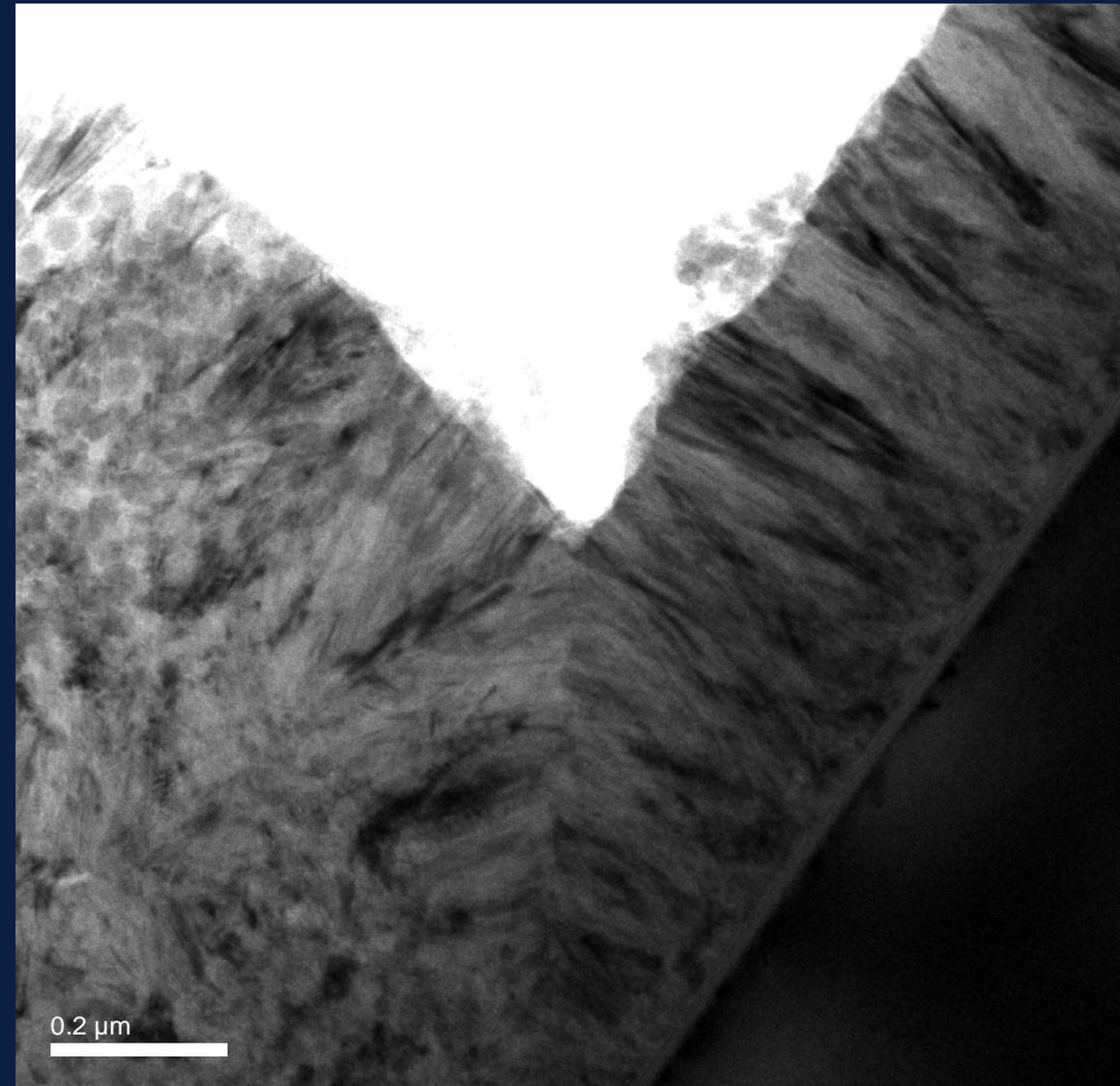


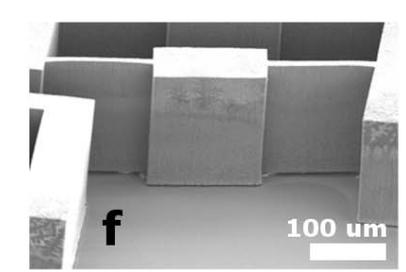
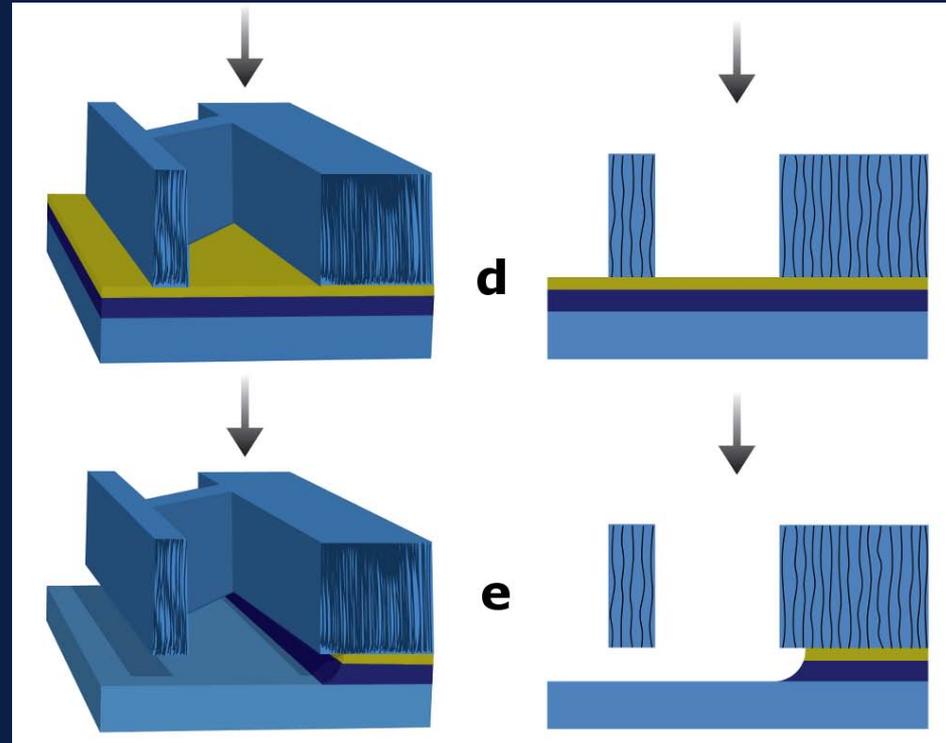
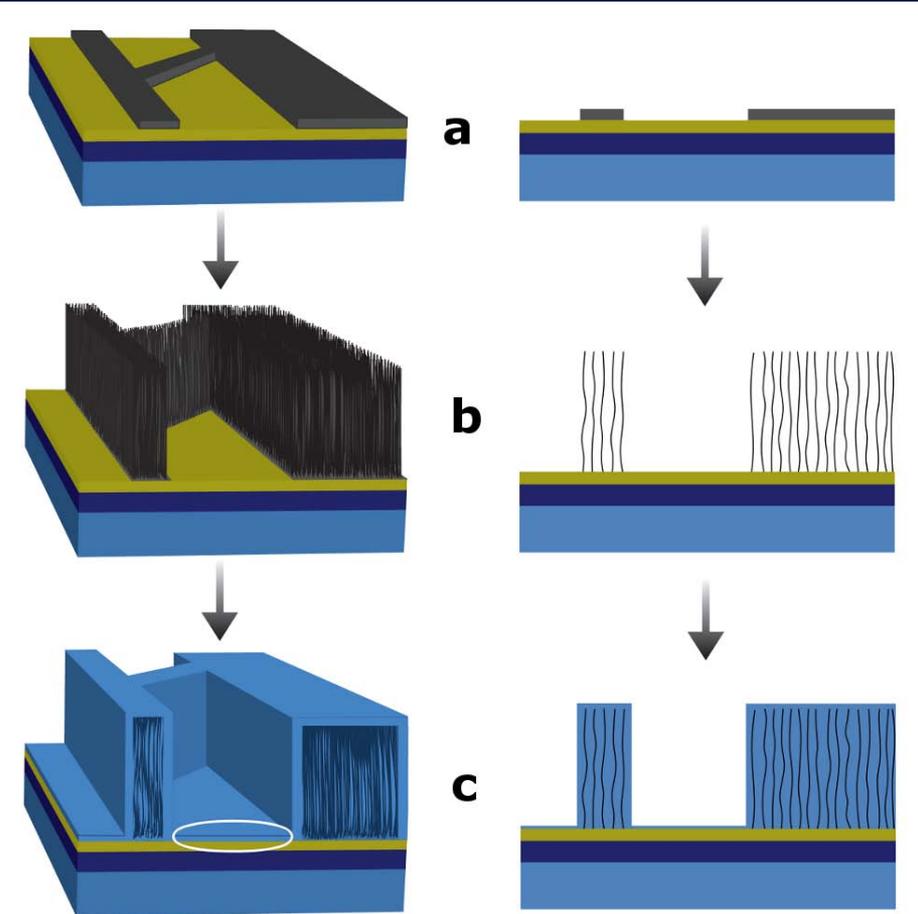
Between tubes, dark field





Properties: Poly-Si

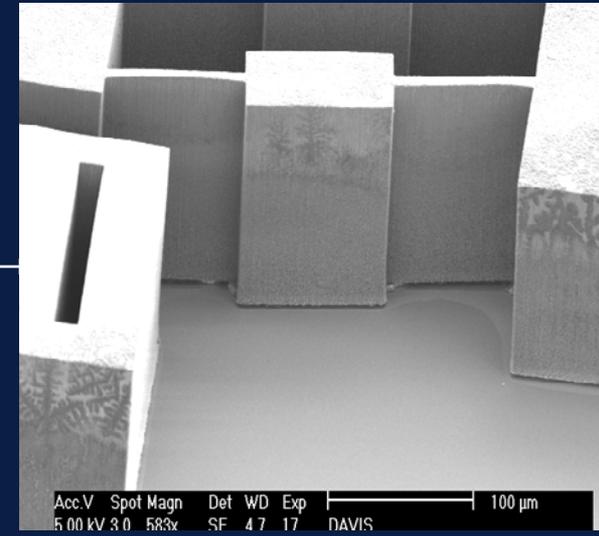
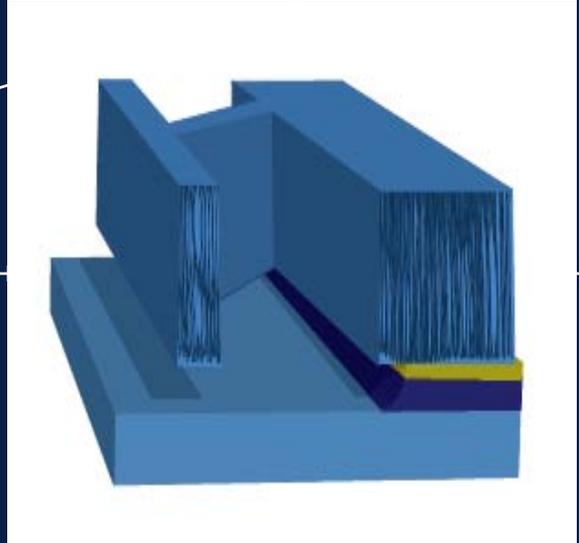
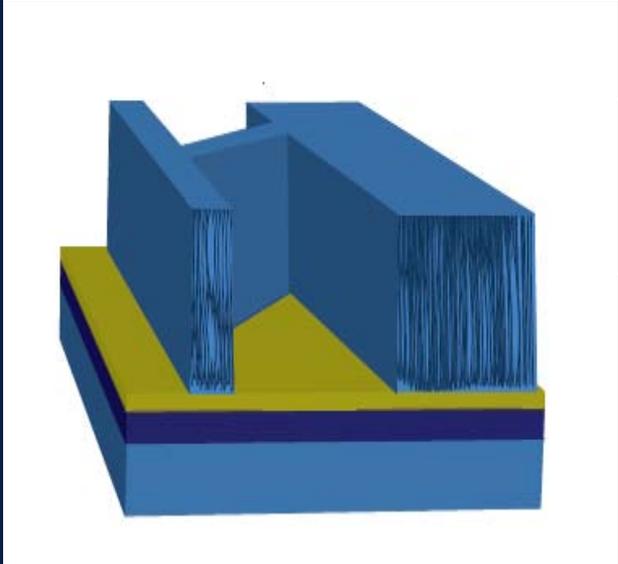
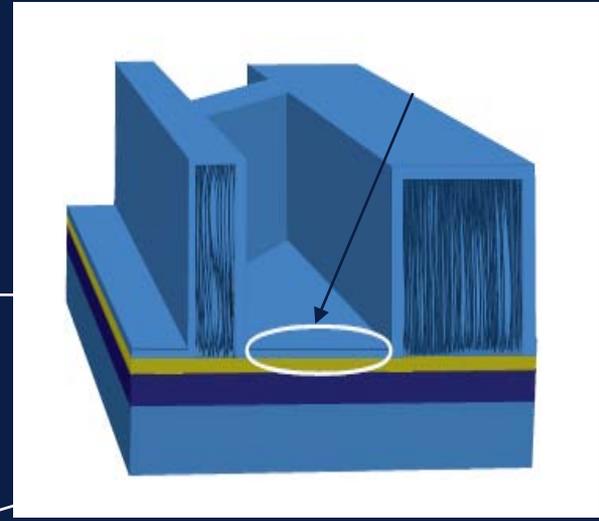
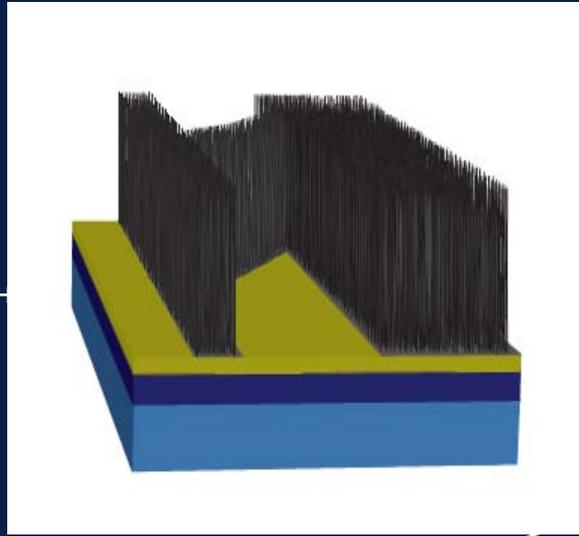
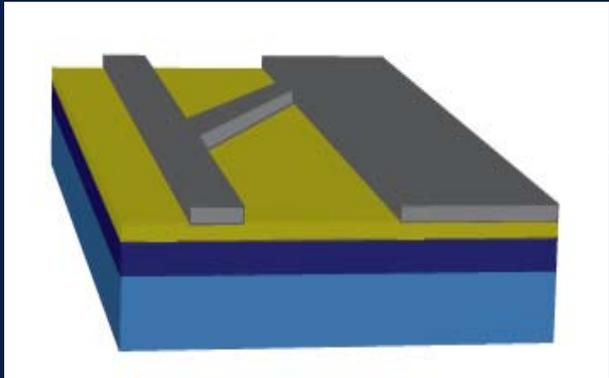




- Nanocomposite**
- Carbon nanotubes**
- Fe**
- Al₂O₃**
- SiO₂**
- Si**

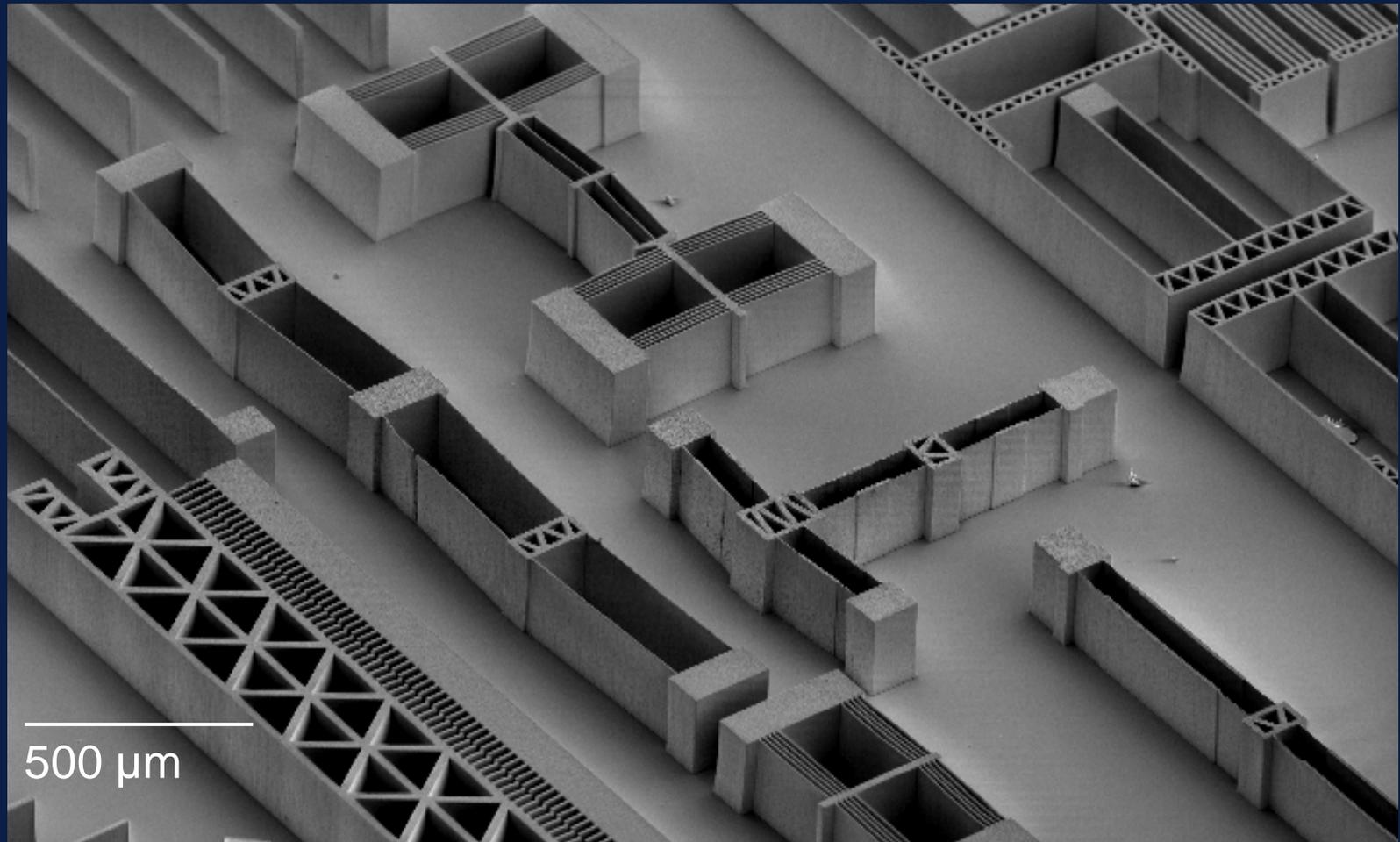


VACNT Composite MEMS Process



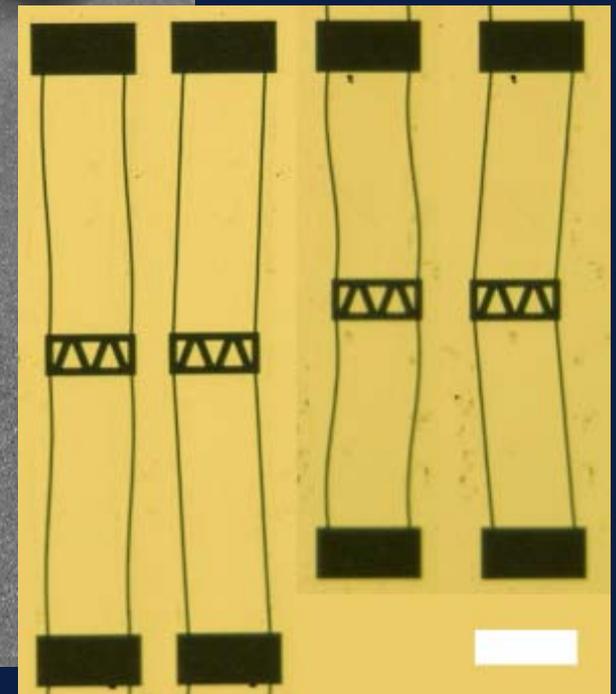
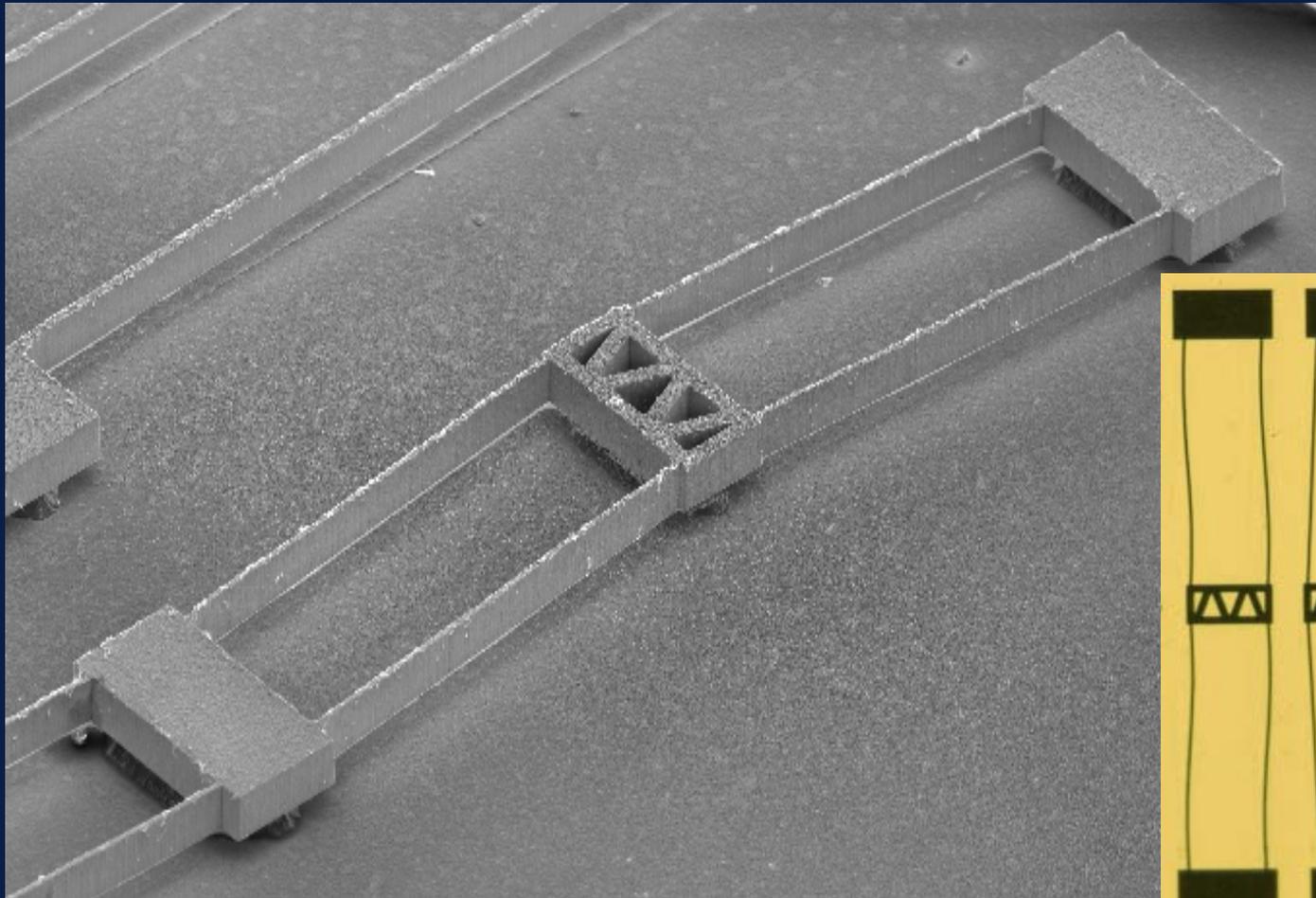


VACNT Templated Microstructures



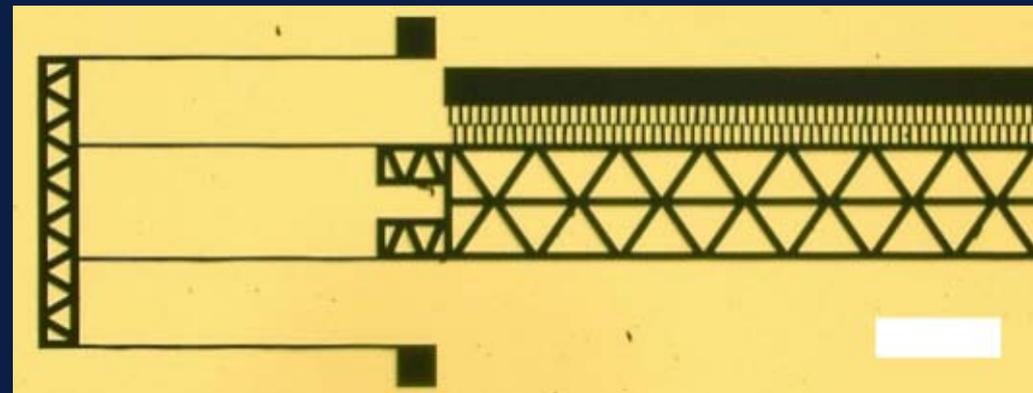
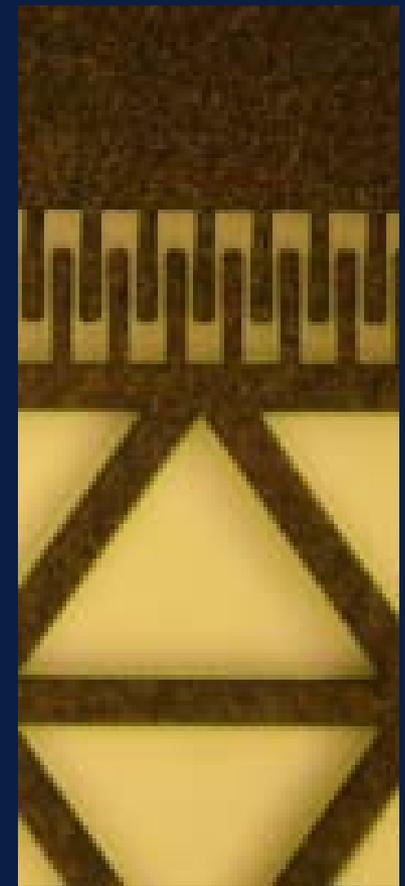
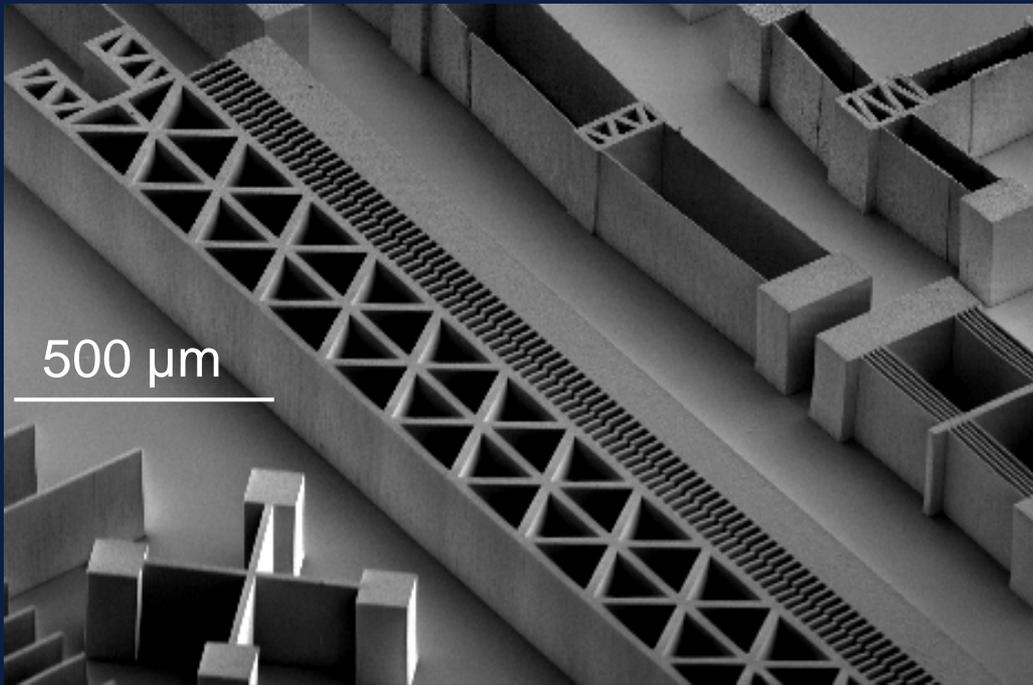


Bi-stable Mechanism



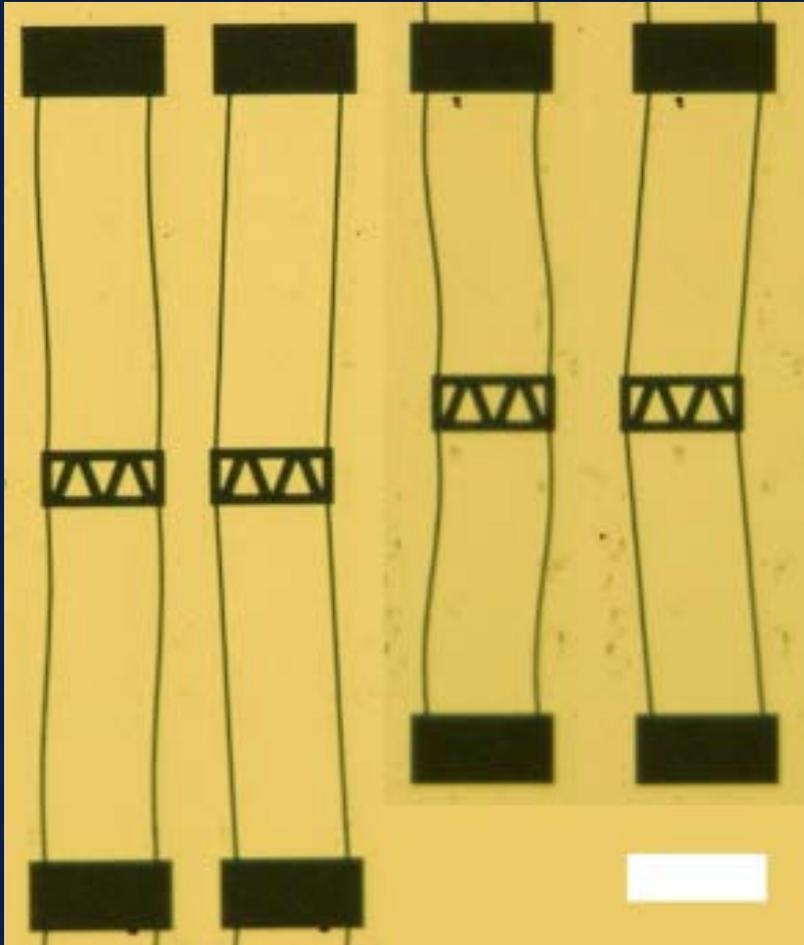


Comb Drive

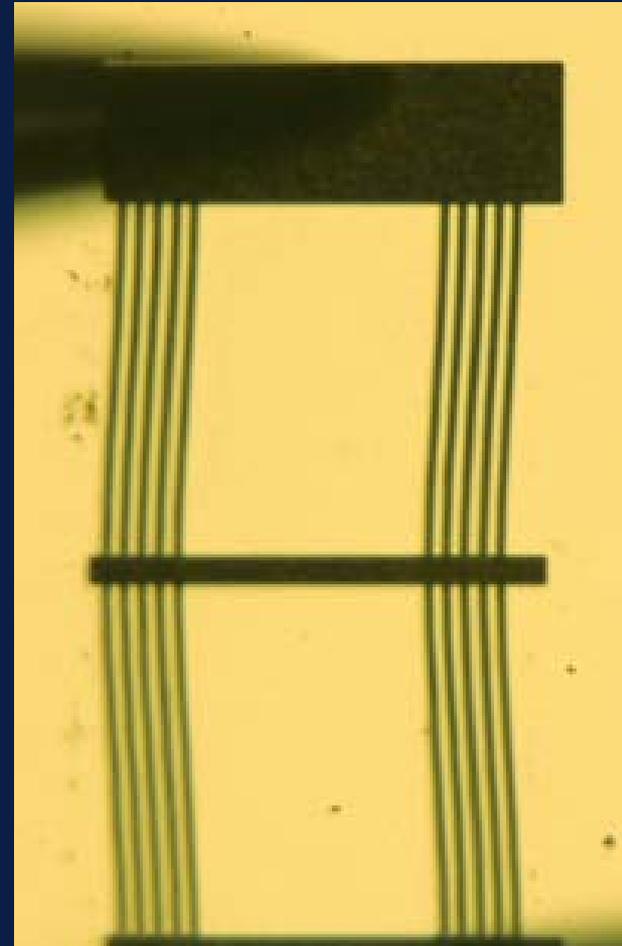




Bistable Mechanisms

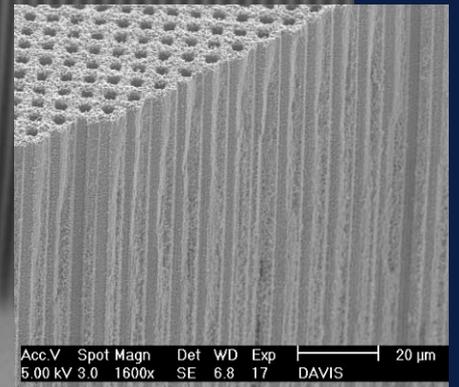
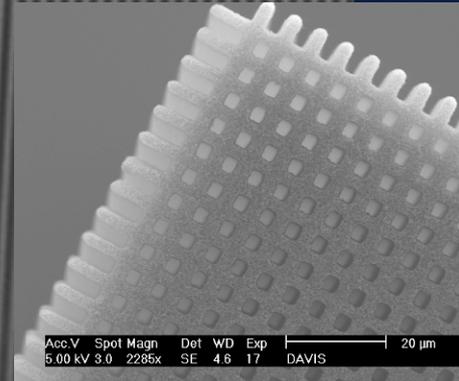
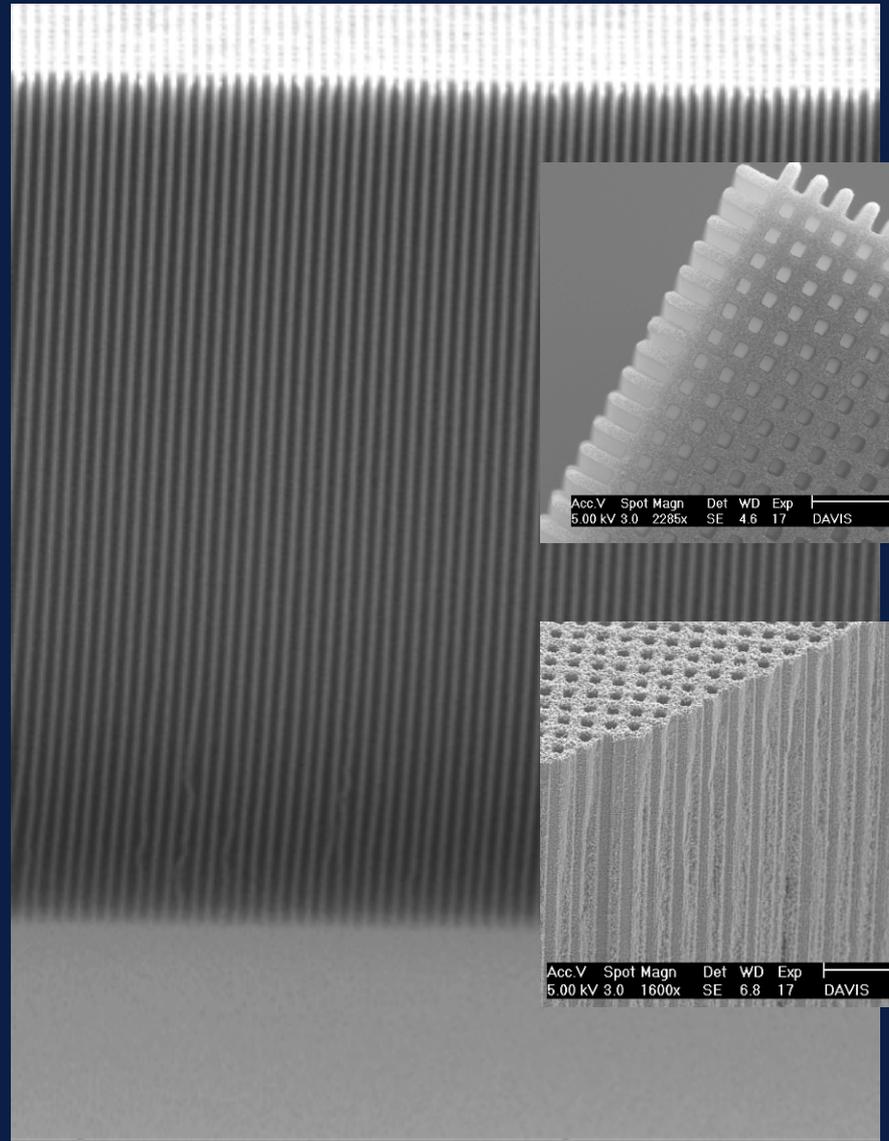
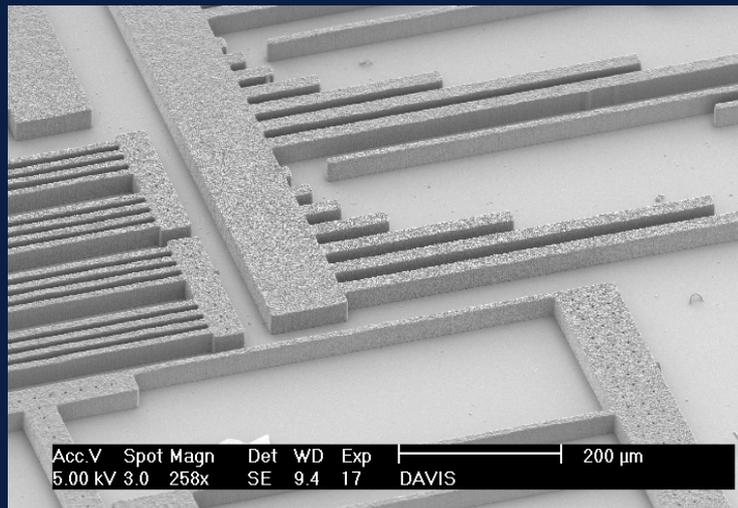
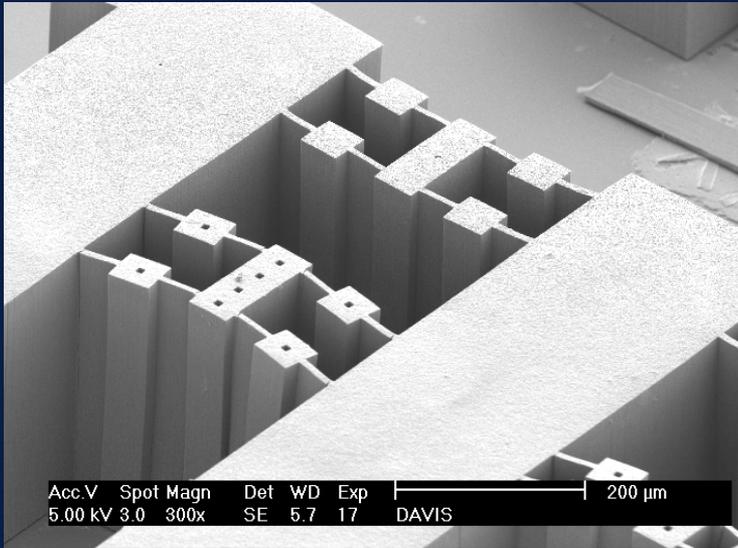


Thermomechanical In-Plane Microactuator (TIM)





Solid High Aspect Ratio Structures

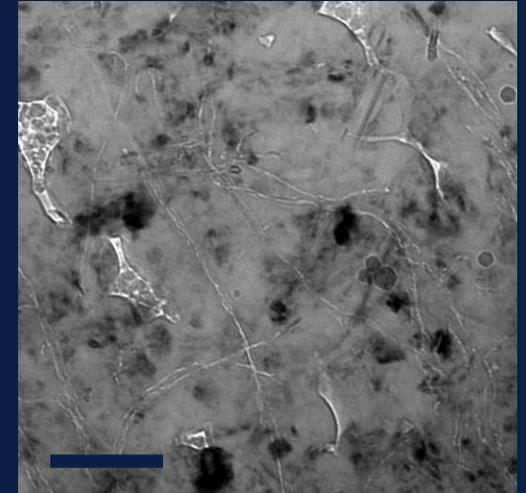
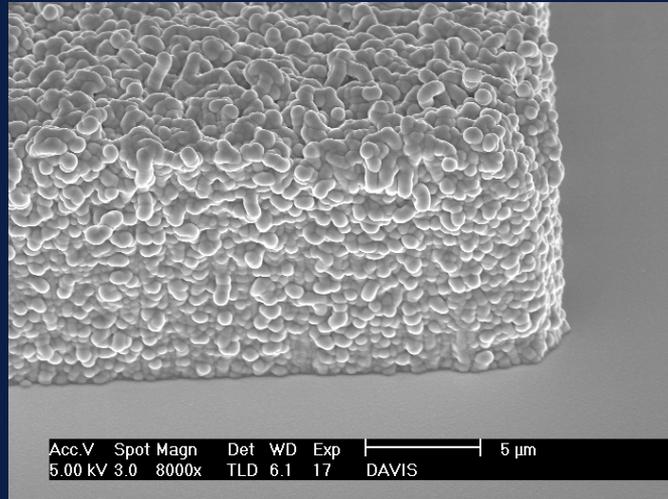


200 μ m
DAVIS

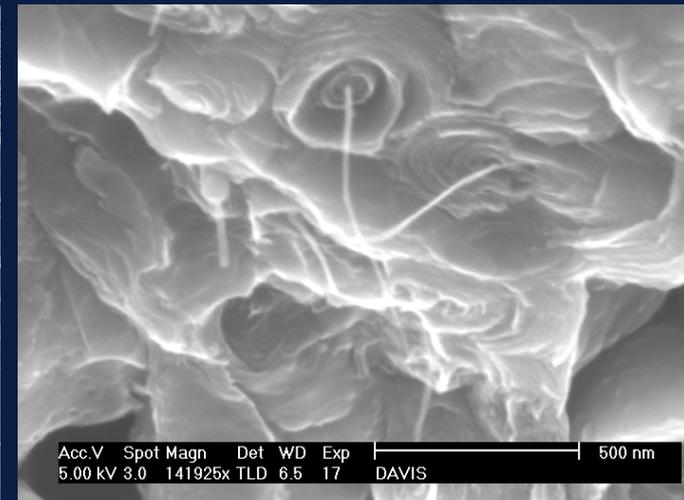
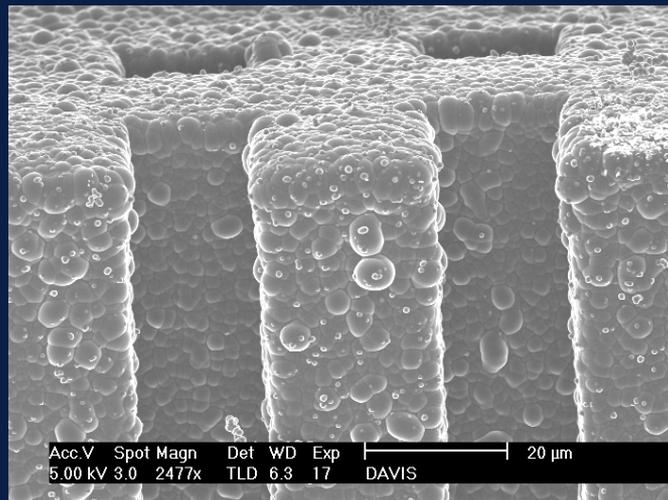


A variety of materials?

Filled with
amorphous Si:



Filled with
amorphous C:





High aspect ratio structures in a variety of materials?

Si, SiNx, C and SiO₂



FROM: *Chemical Vapor Deposition*, ed. Jong-Hee Park, ASM International (2001)

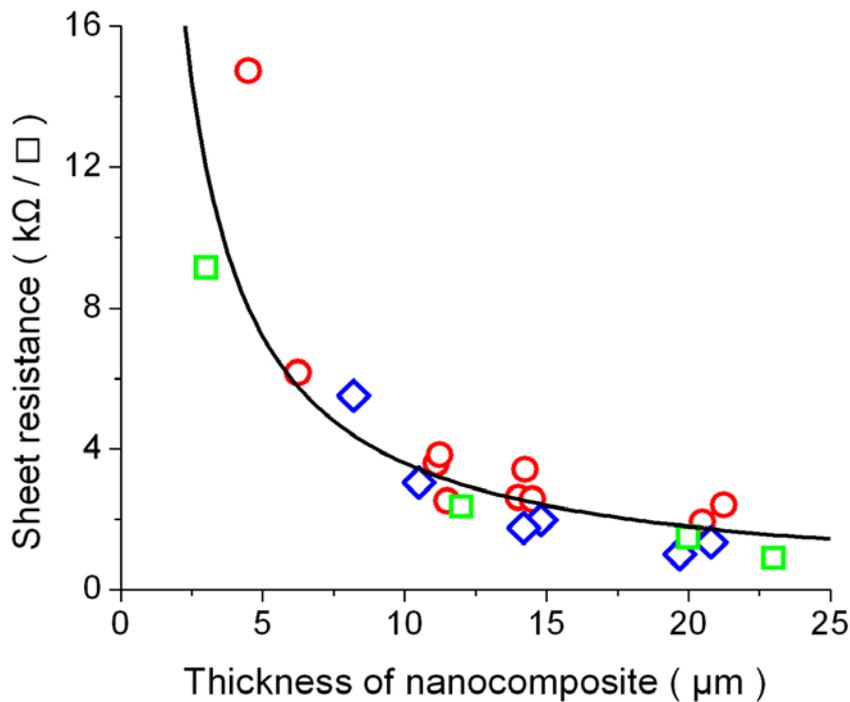


Properties: Resistivity

- Isolated nanotubes: Can exhibit ballistic conduction over distances of several microns
- Undoped poly-Si: $\rho \sim 10^2 \Omega \text{ cm}$
- Si-coated nanotubes: $\rho \sim ?$
- Coat tubes with insulator \rightarrow Conductive MEMS made from insulating materials?



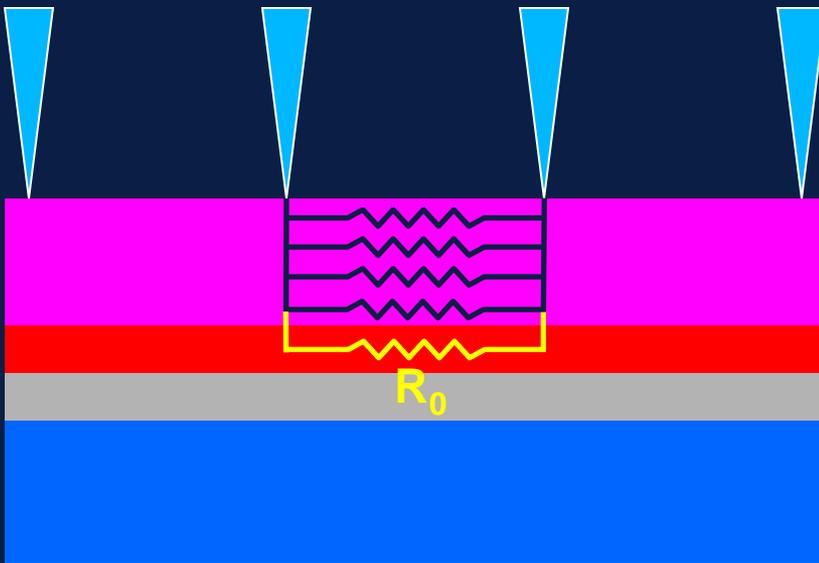
Properties: Resistivity



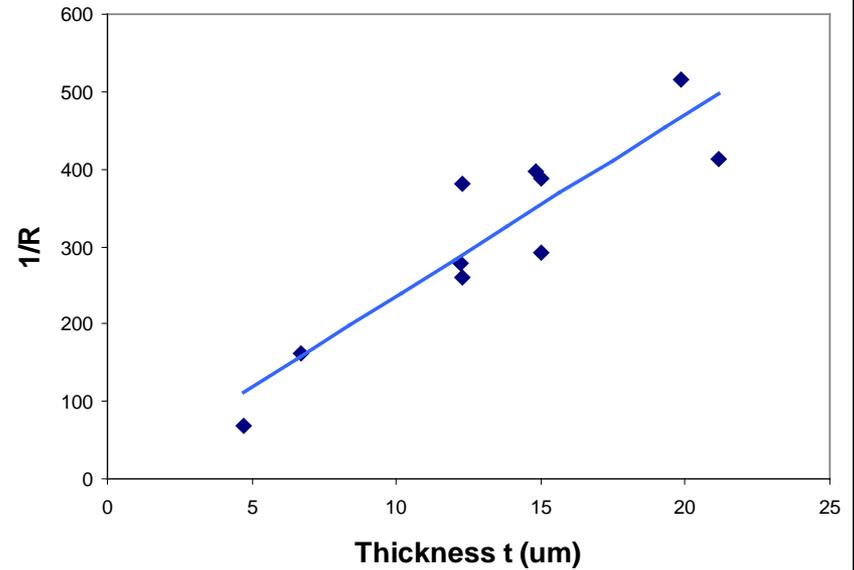
Sheet resistance versus thickness for silicon-filled forests (red circles), silicon nitride-filled forests (green squares), and 20 nm of silicon followed by filling with silicon nitride (blue diamonds) reveals the expected inverse proportionality relationship. The solid line is calculated for an infinitely thin sample with resistivity of $3.6 \Omega \text{ cm}$.



Properties: Resistivity



Sheet conductivity versus thickness



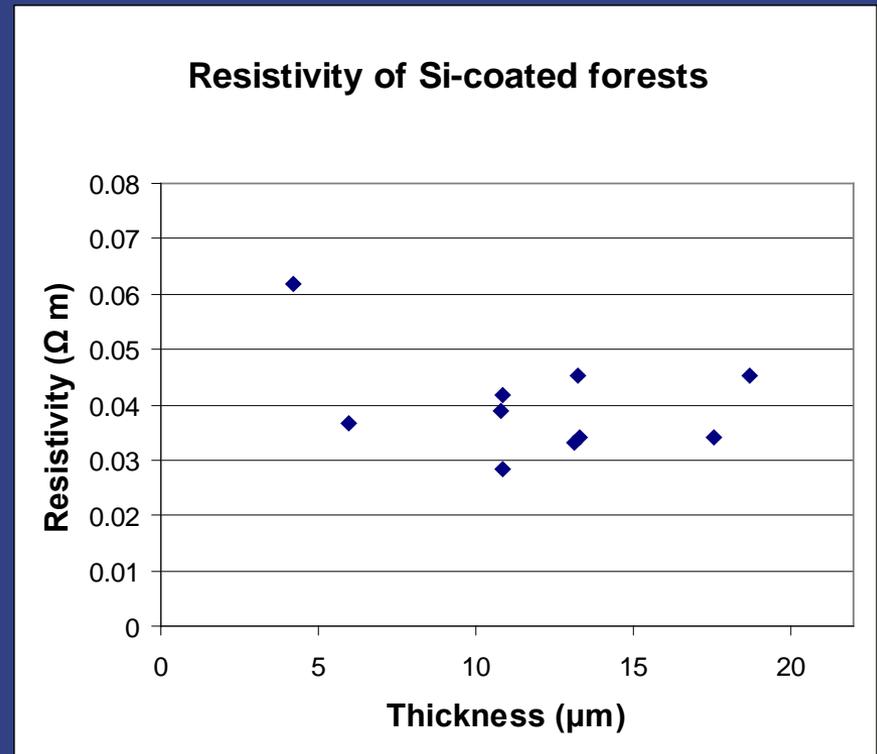
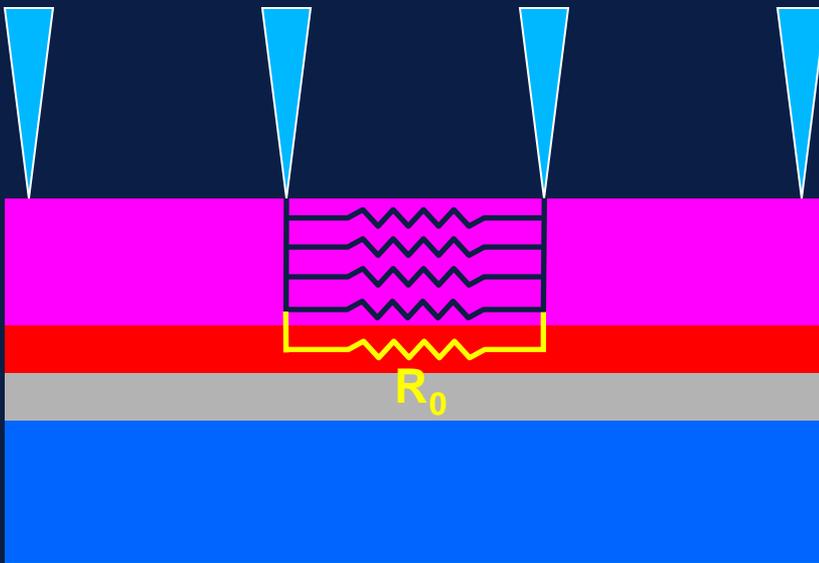
$$R_{sheet} = \frac{K_1}{t + K_2} \approx \frac{K_1}{t}$$

$$K_1 = 42.6 \Omega \text{ m}$$

$$K_2 = 0.04 \mu\text{m}$$



Properties: Resistivity

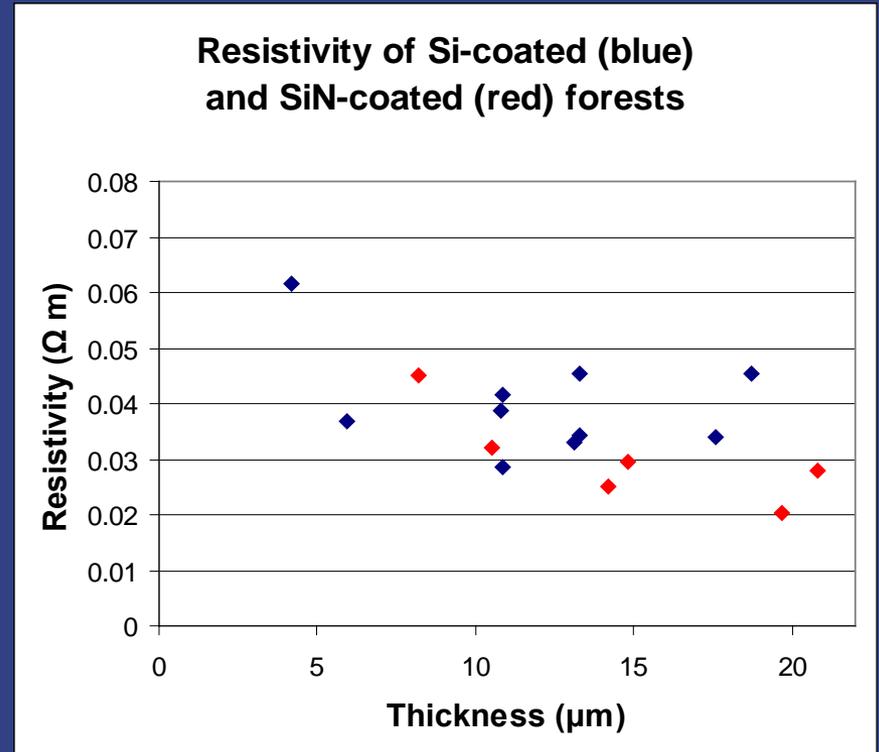
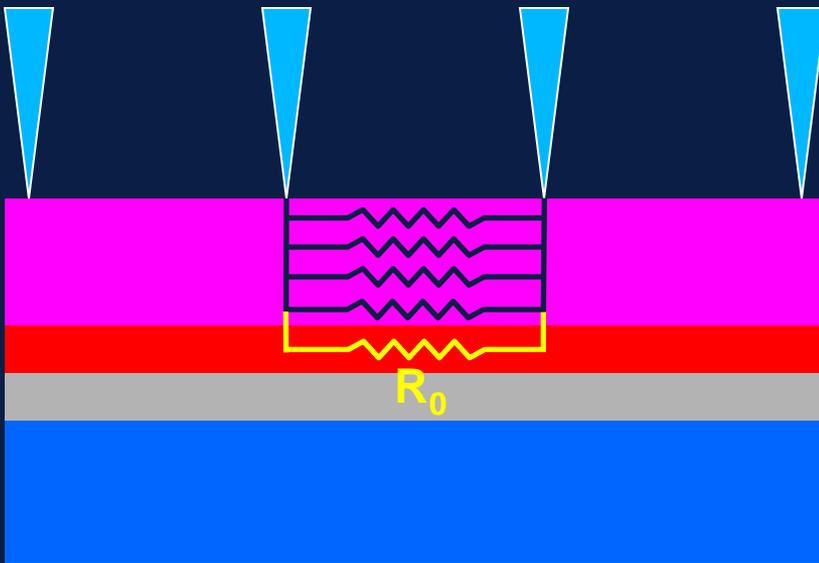


$$\rho_{\text{forest}} \sim 4 \Omega \text{ cm}$$

$$\rho_{\text{poly-Si alone}} \sim 10000 \Omega \text{ m}$$



Properties: Resistivity

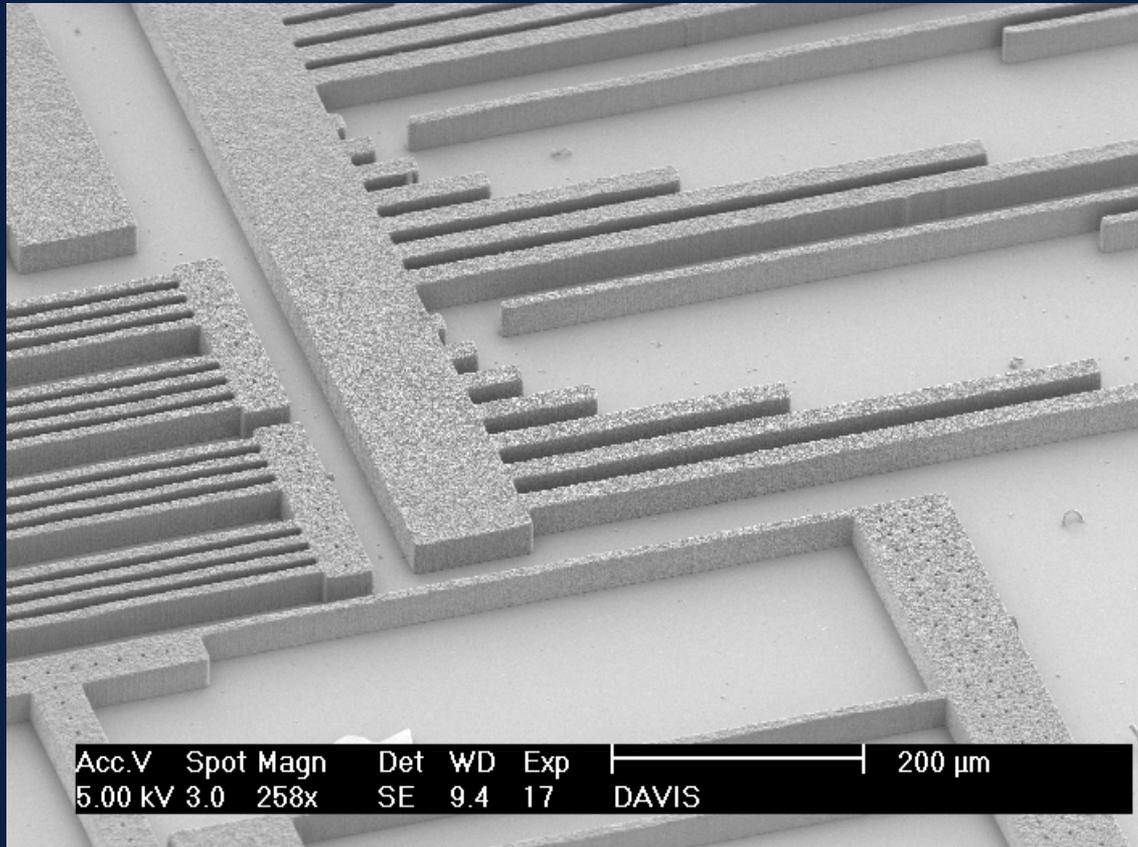


$$\rho_{\text{forest}} \sim 4 \Omega\text{-cm}$$

Approximately the same resistivity as previously reported for other CNT-composites



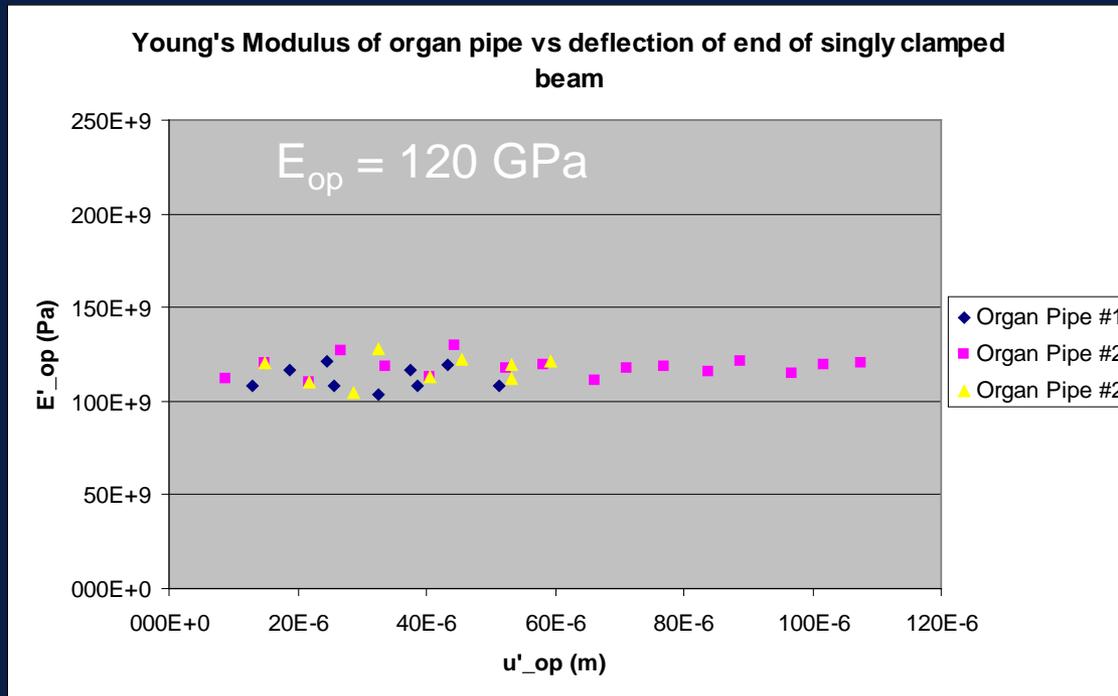
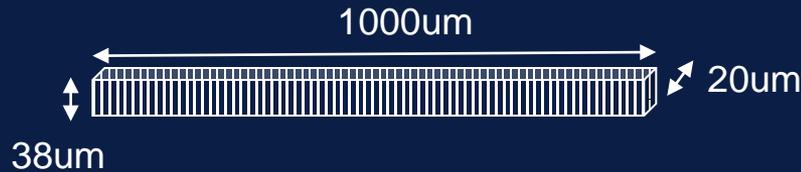
Mechanical Characterization



Filled forests are solid and well adhered (can withstand the scotch tape test)



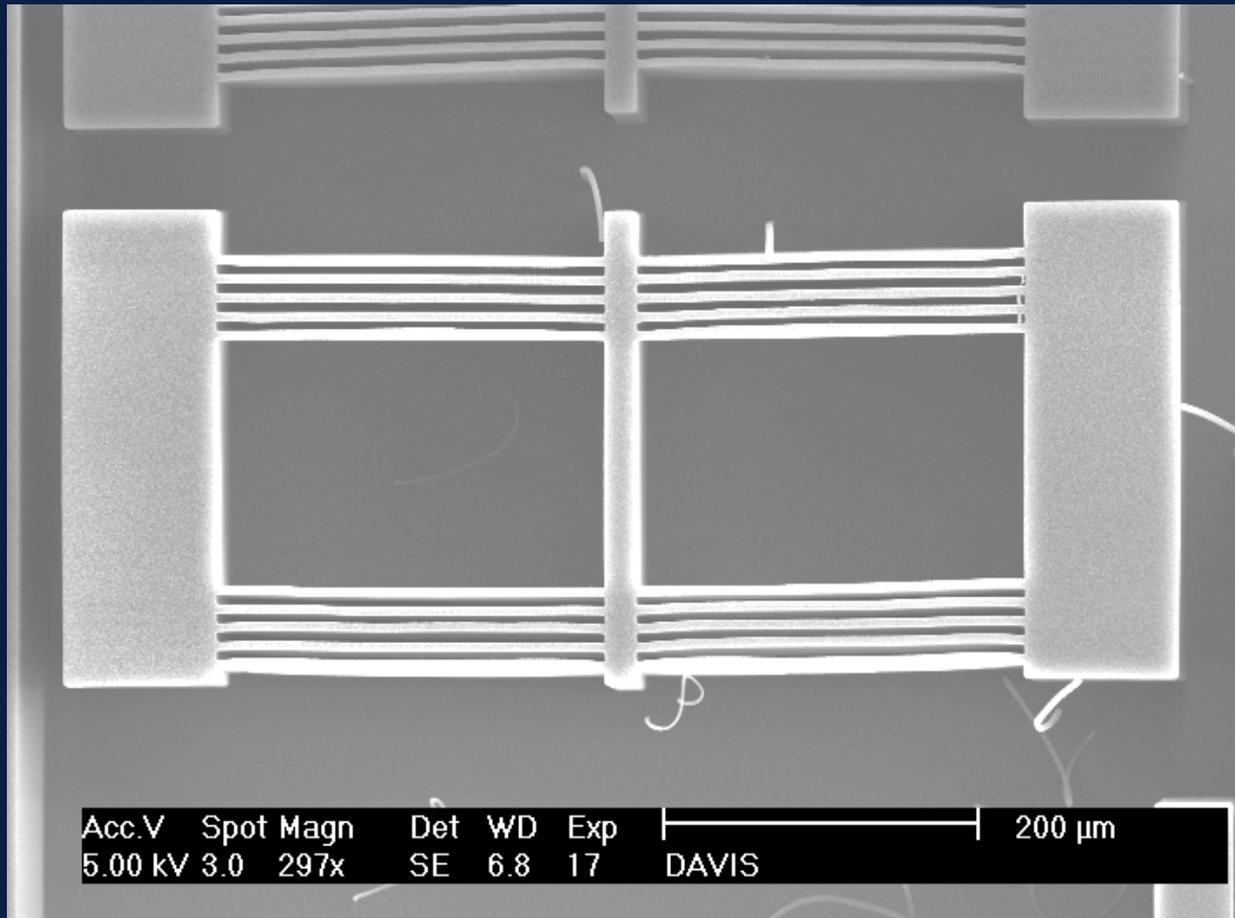
Beam Bending Measurement of Elastic Modulus



Reported bulk polySi modulus ~ 140-210 GPa, dependent on deposition conditions



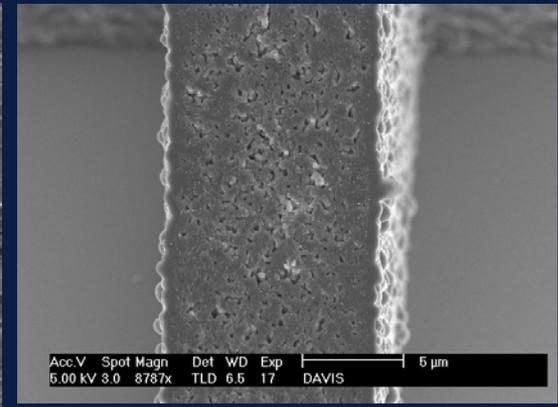
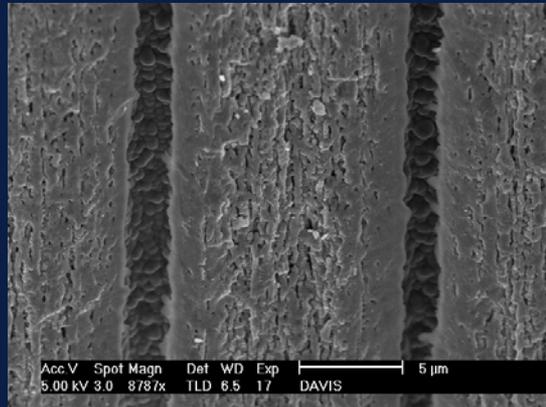
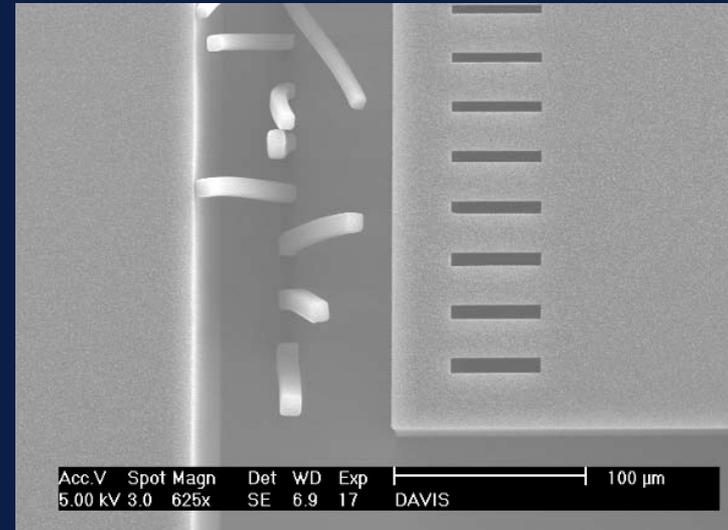
Actuated device: thermomechanical in-plane microactuator (TIM)

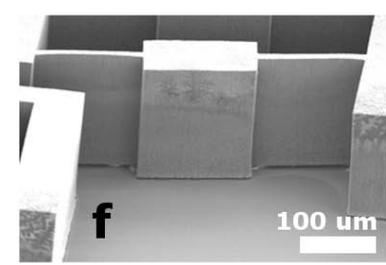
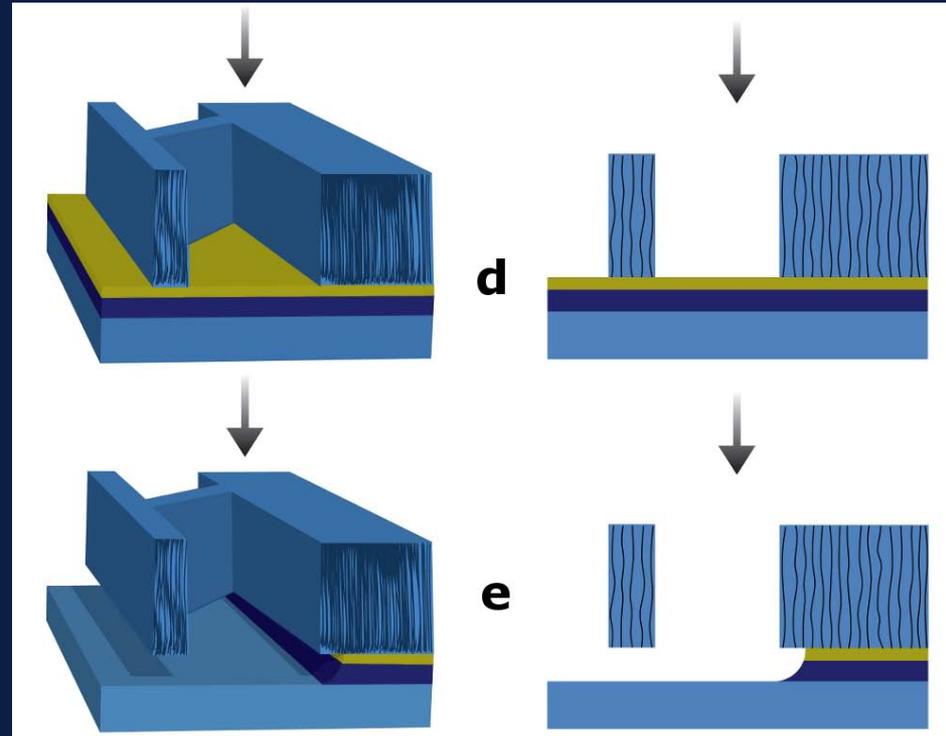
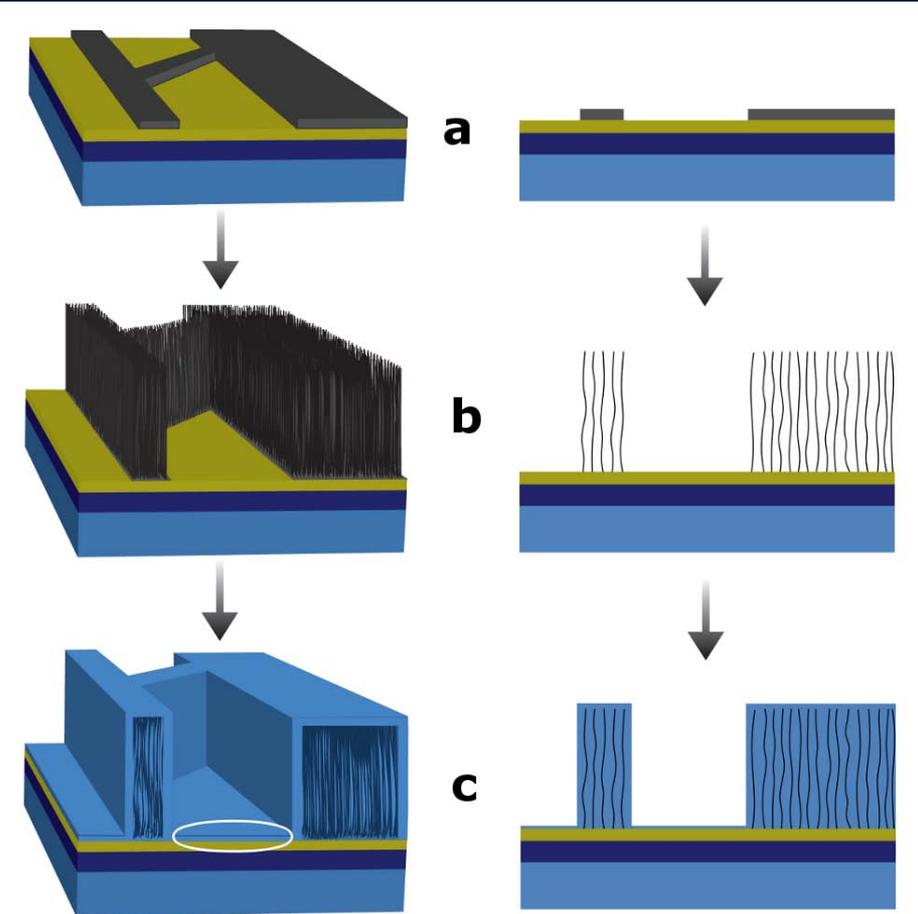




Developing Engineering Design Rules

- Height to width ratio for dimensional stability
- Maximum feature width for filling of forest interior
- Role of geometry
LPCVD fill-factor

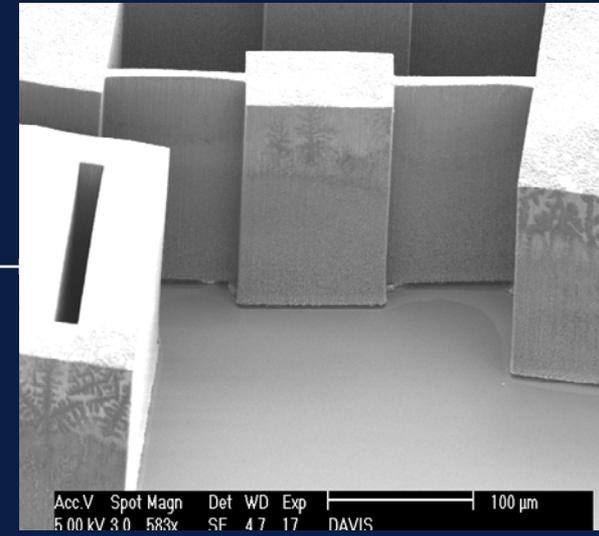
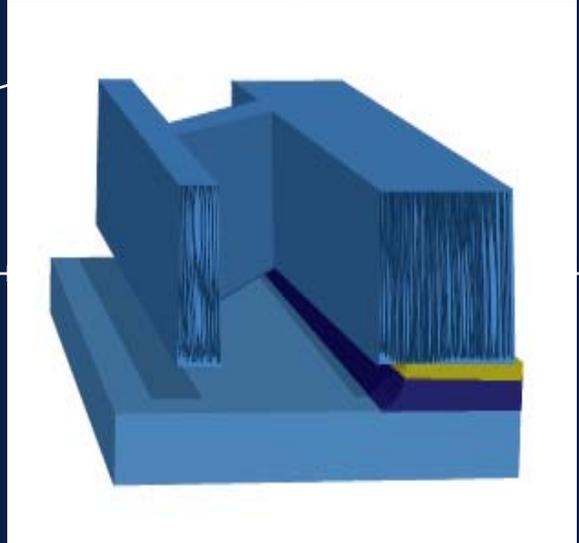
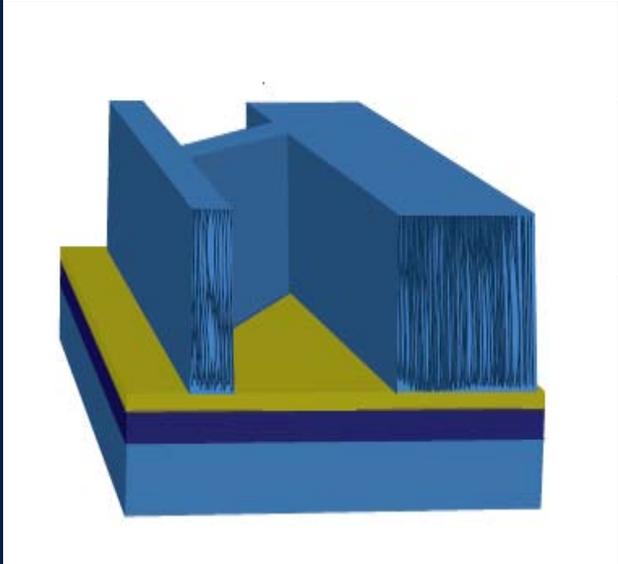
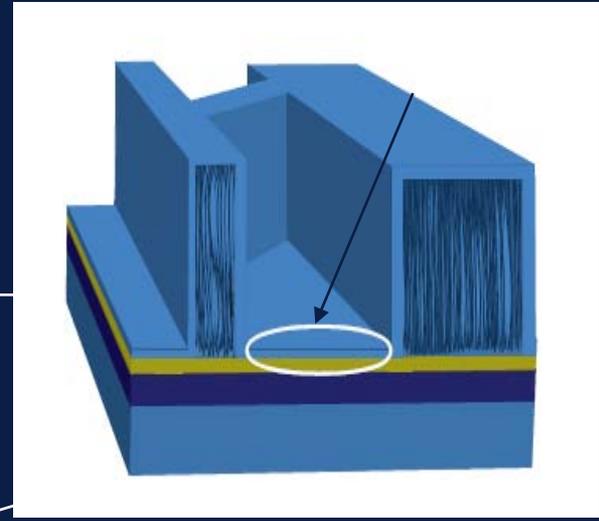
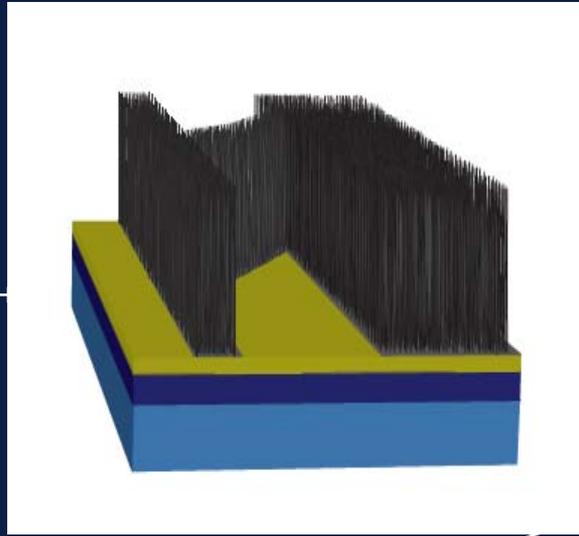
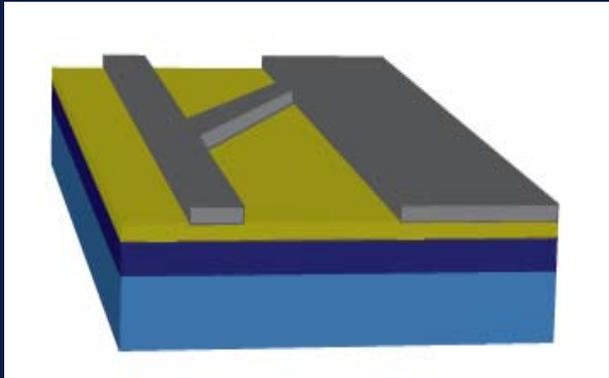




- Nanocomposite
- Carbon nanotubes
- Fe
- Al₂O₃
- SiO₂
- Si

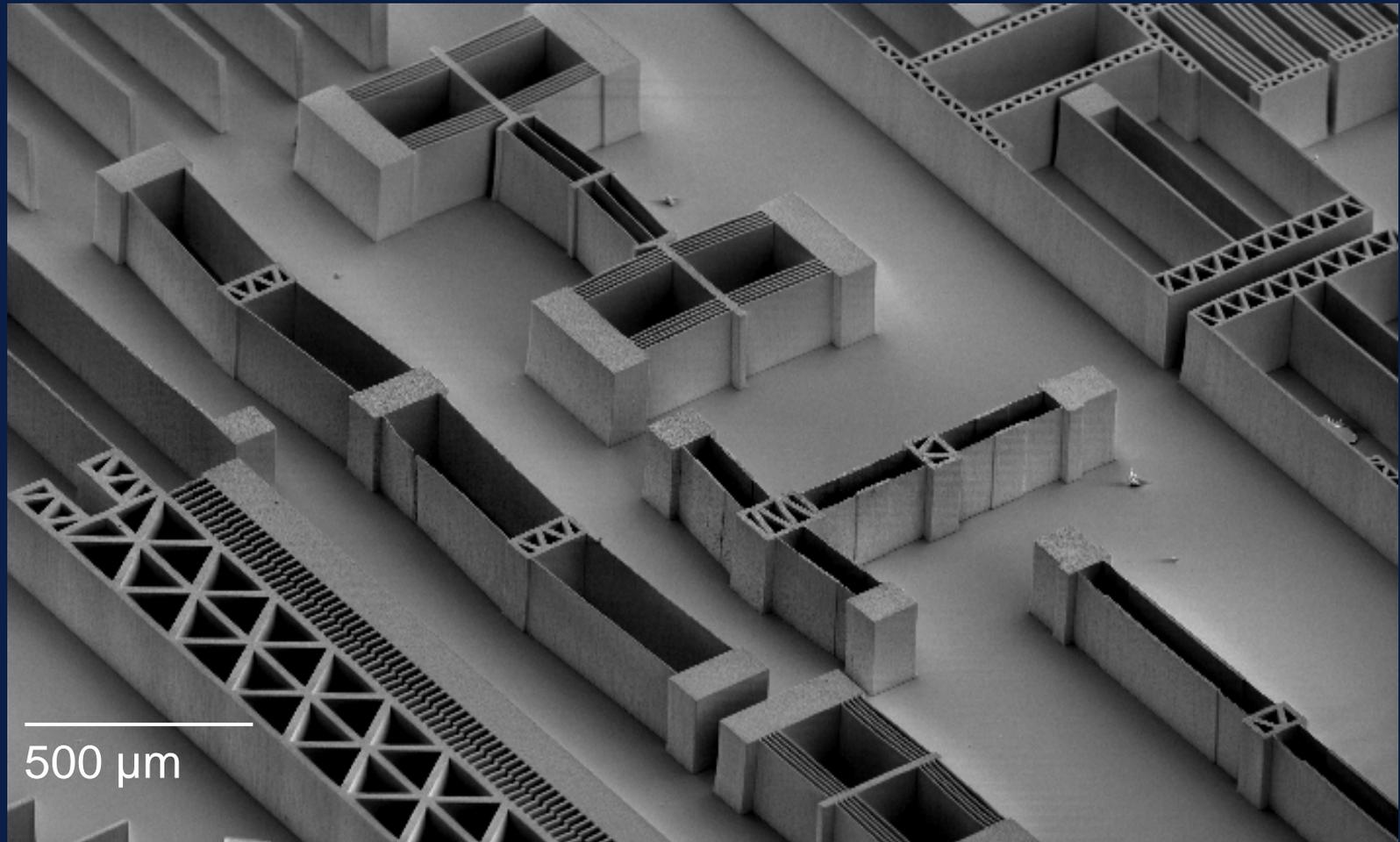


VACNT Composite MEMS Process



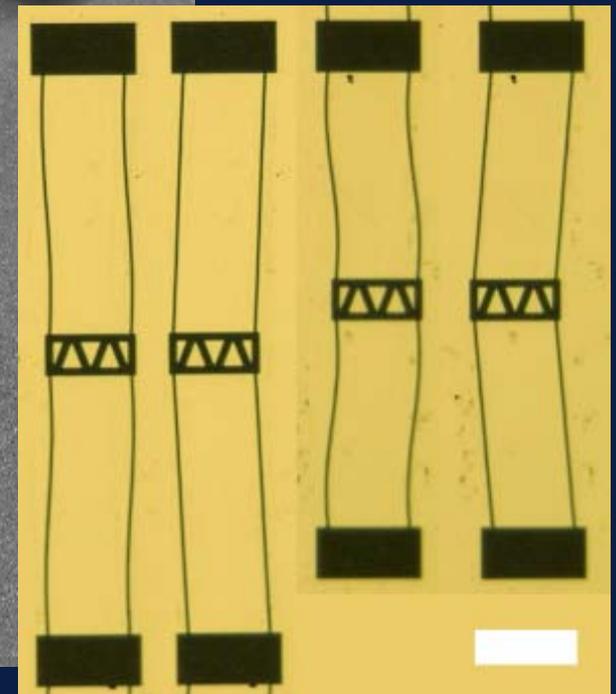
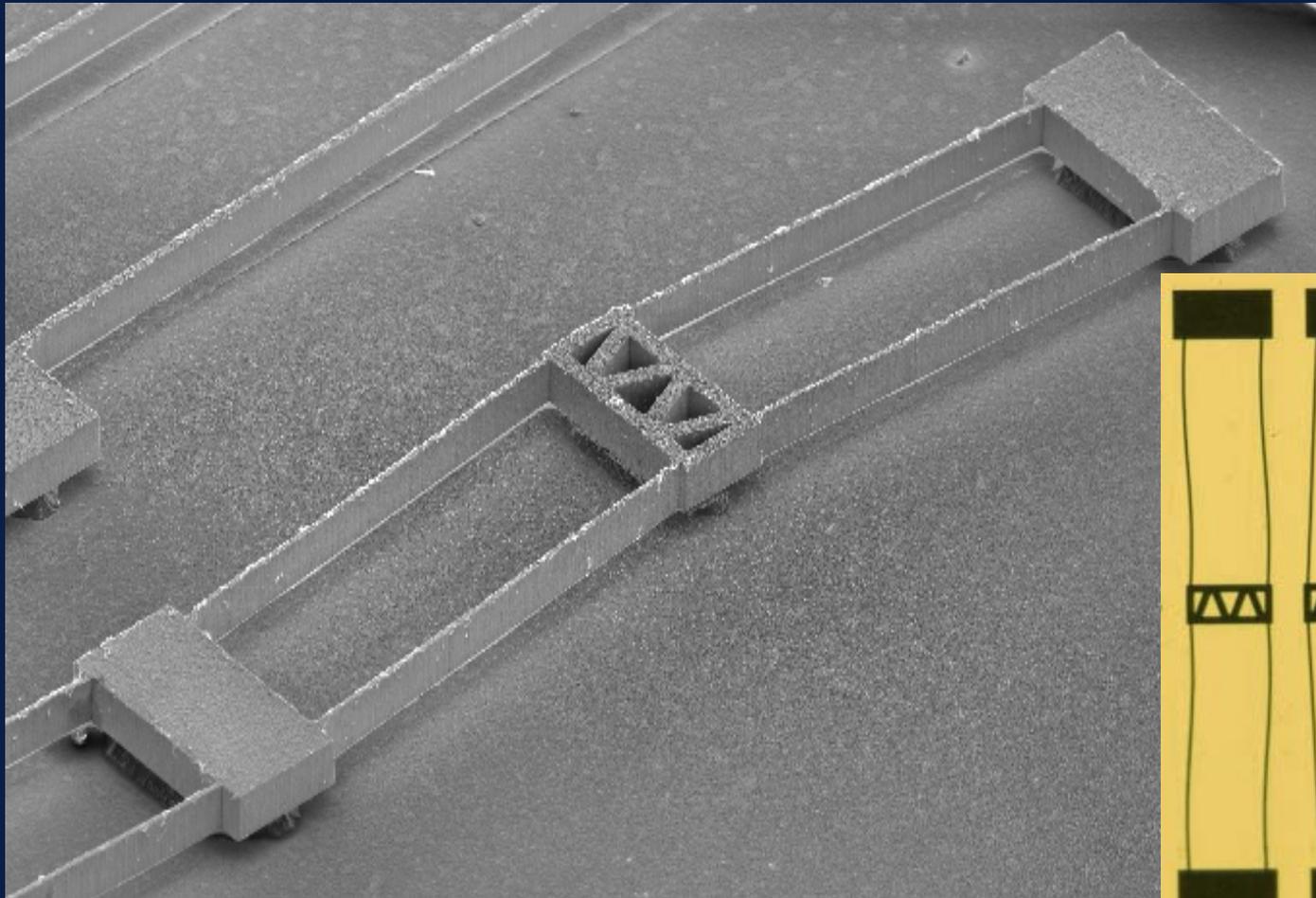


VACNT Templated Microstructures



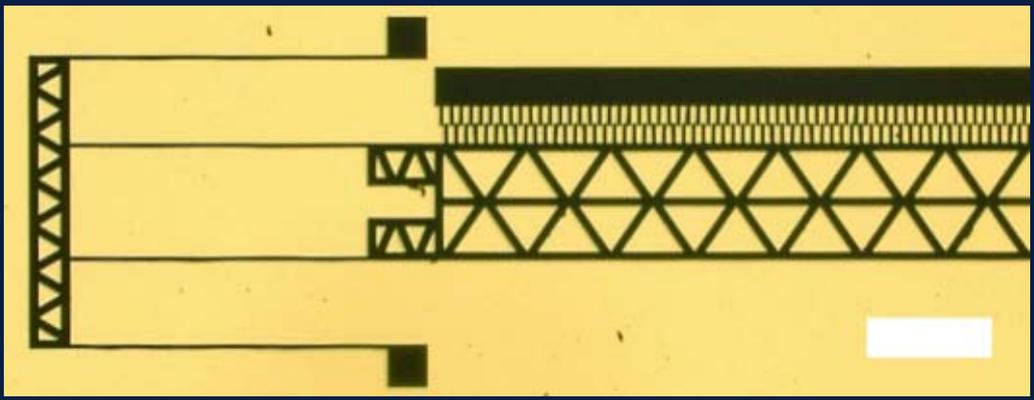
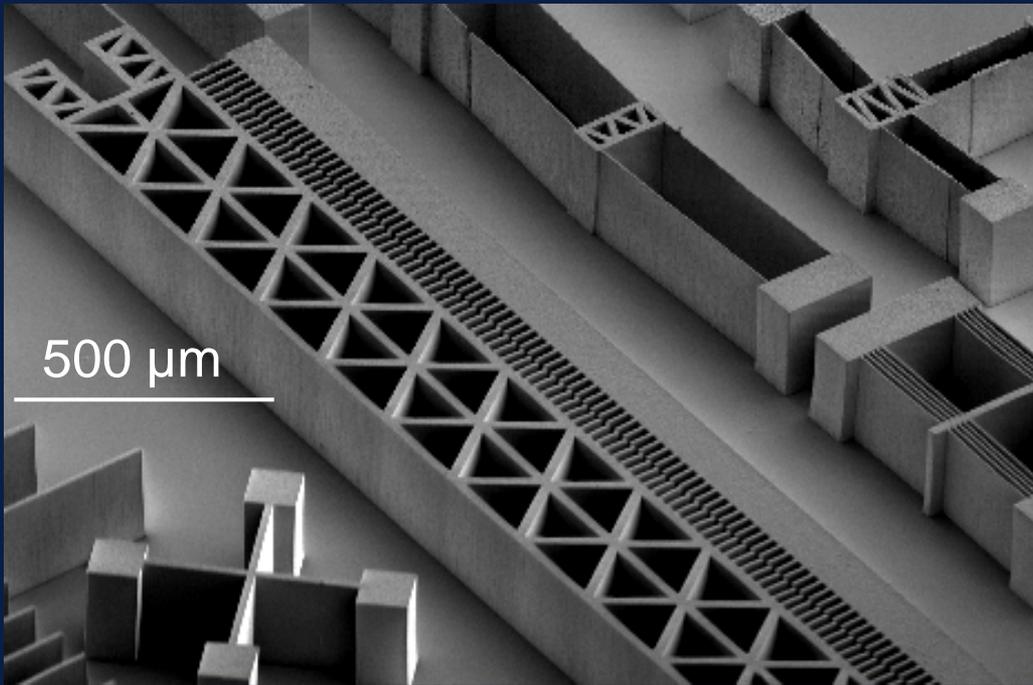


Bi-stable Mechanism



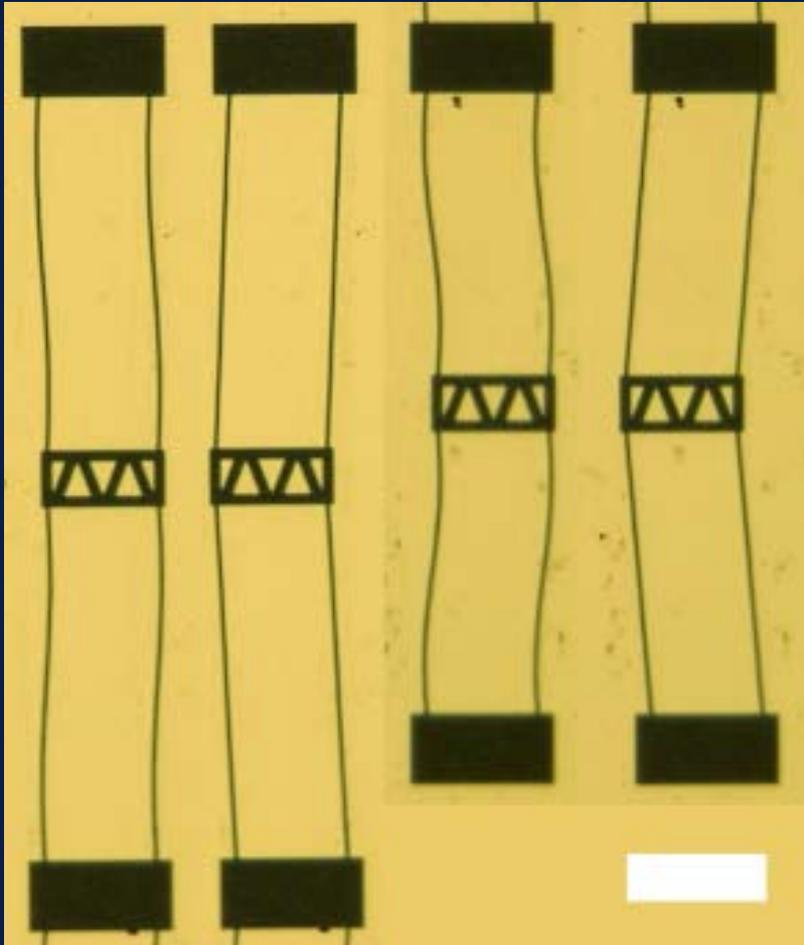


Comb Drive

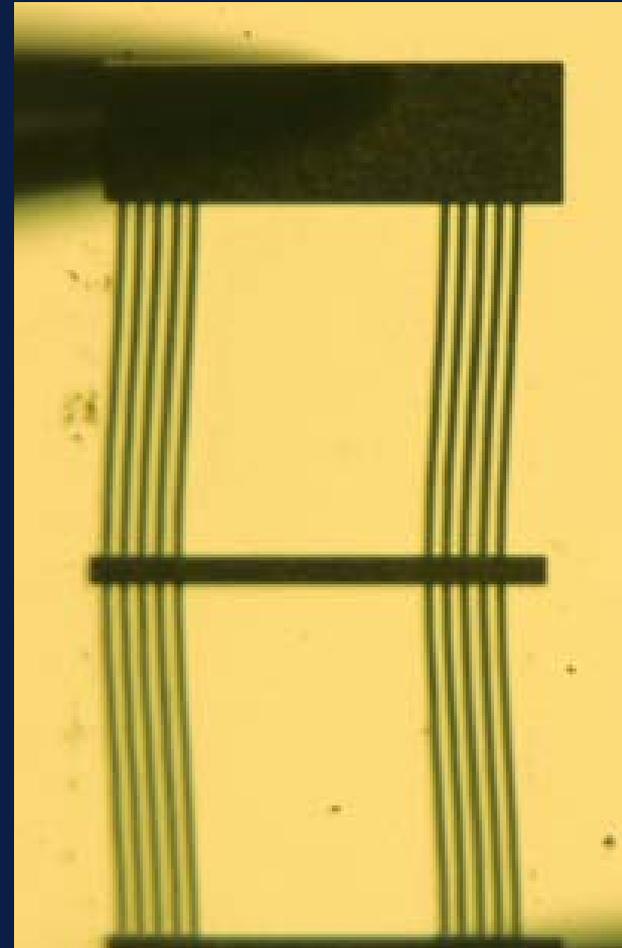




Bistable Mechanisms



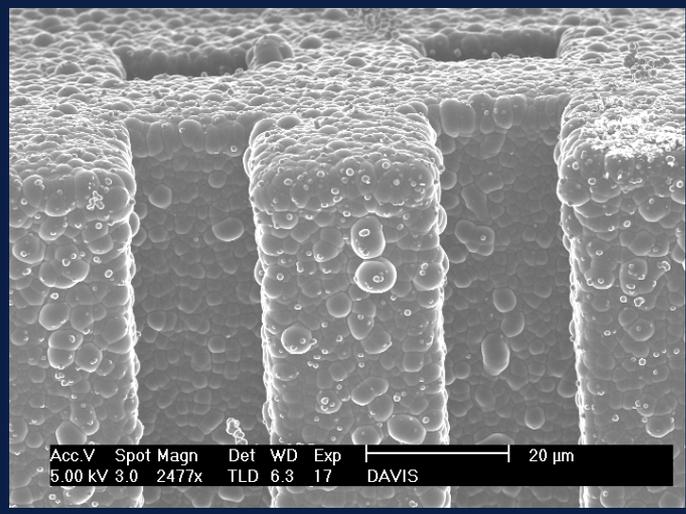
Thermomechanical In-Plane Microactuator (TIM)





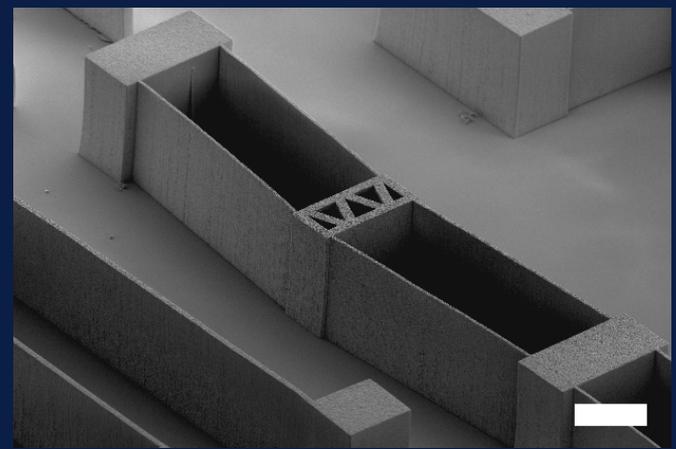
Other infiltration materials

Filled with amorphous C:



Fills most completely

Filled with Silicon nitride:

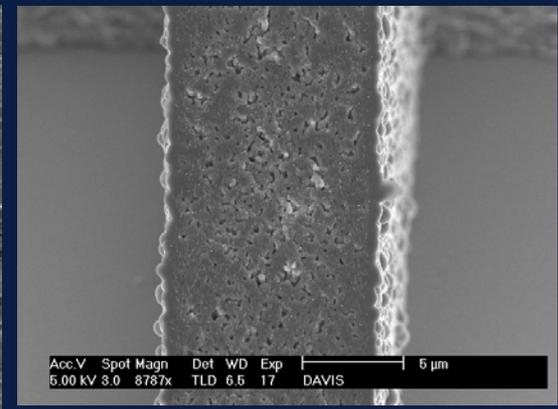
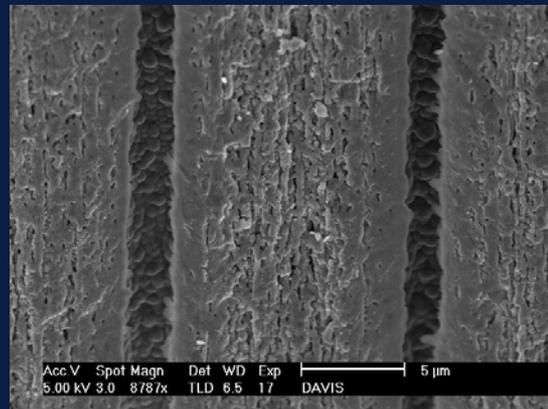
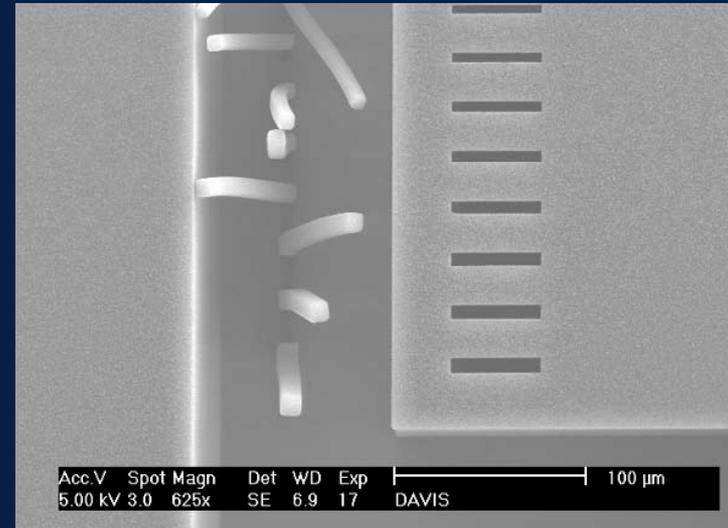


Forms stable high aspect walls



Developing Design Rules

- Height to width ratio for dimensional stability
- Maximum feature width for filling of forest interior
- Role of geometry
LPCVD fill-factor

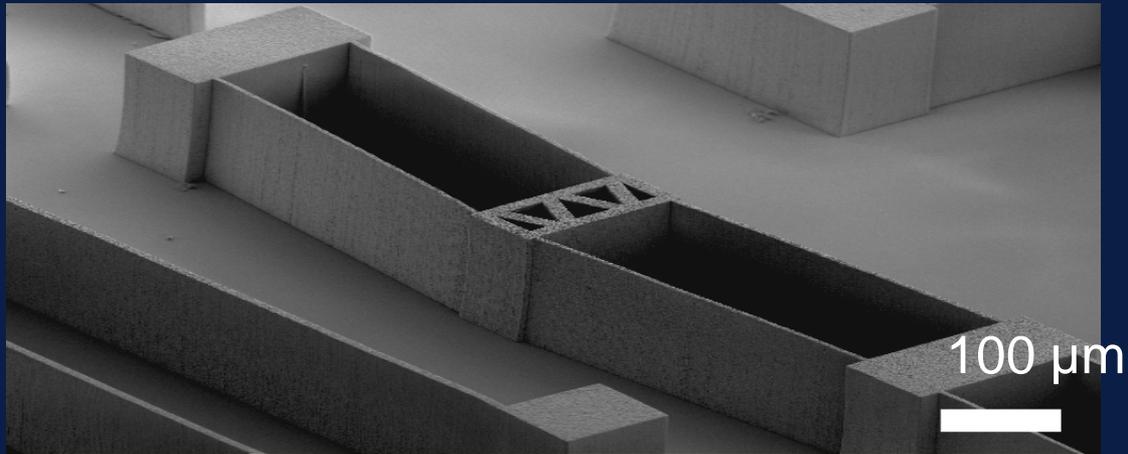




End



Nanostructured Materials as templates for fabrication

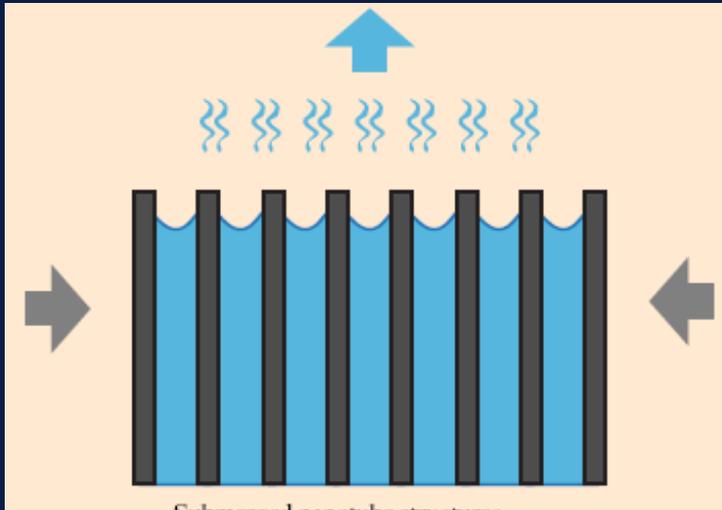




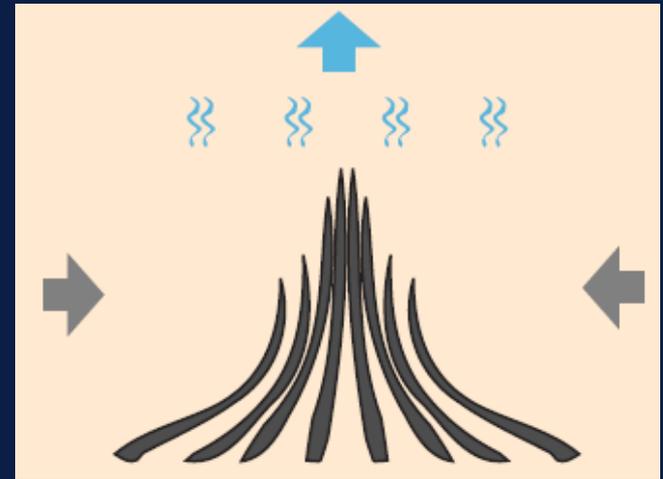
Dense Nanotube Structures



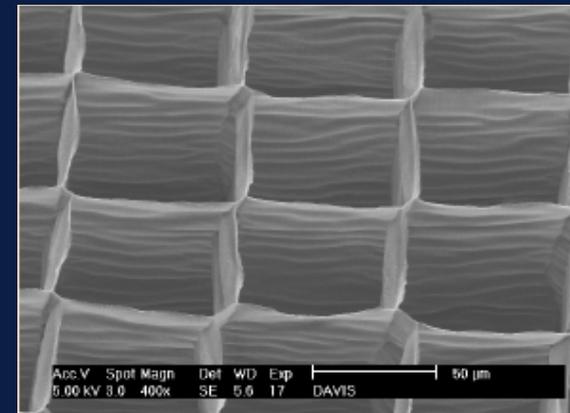
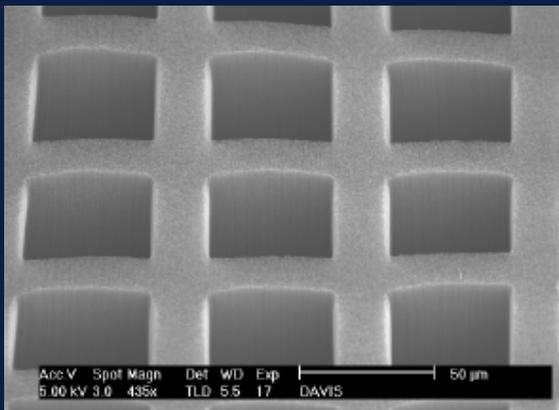
Liquid Induced Densification



Submerged nanotube structures

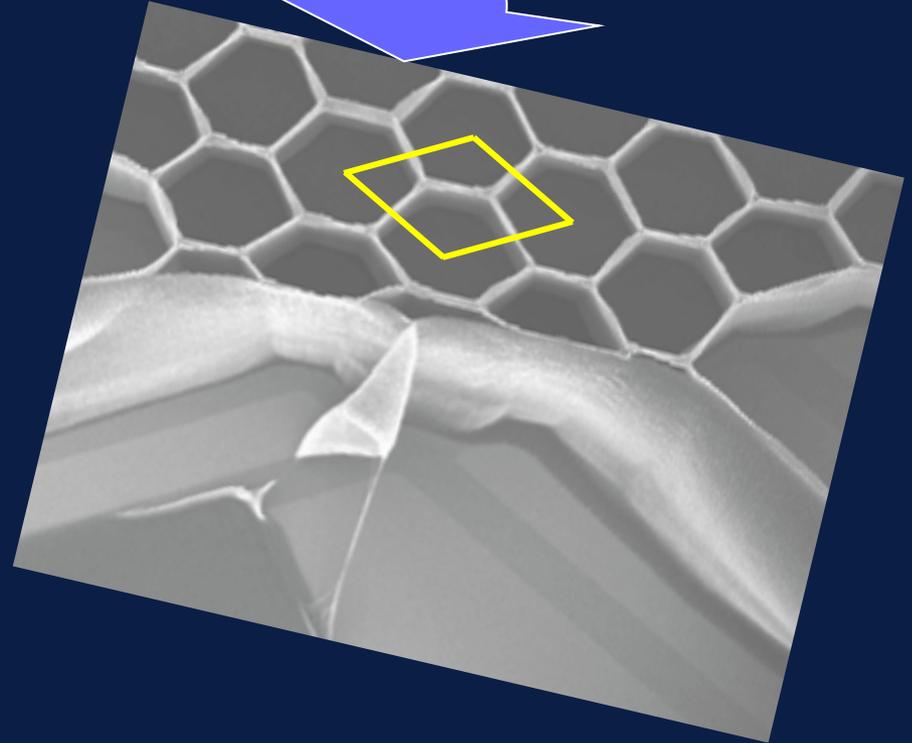
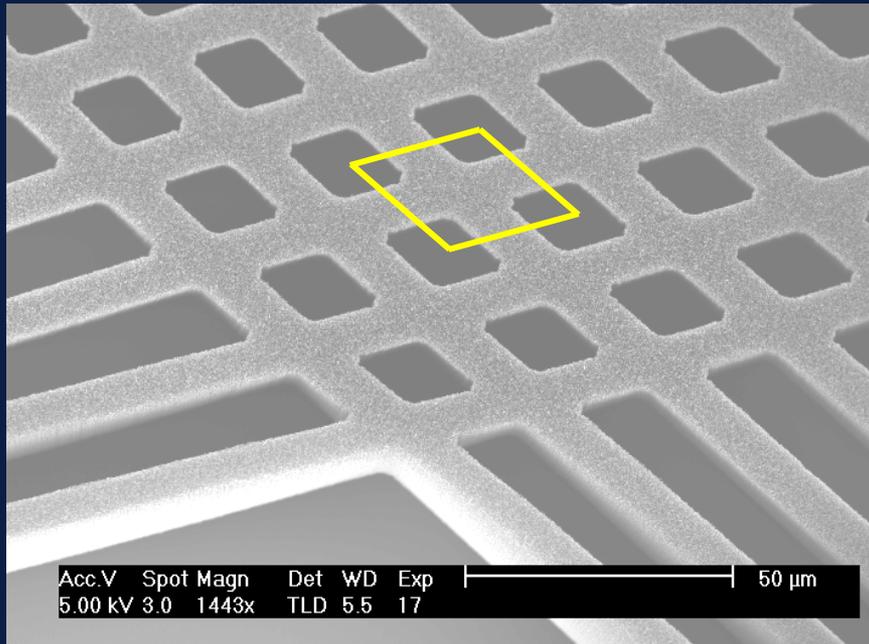
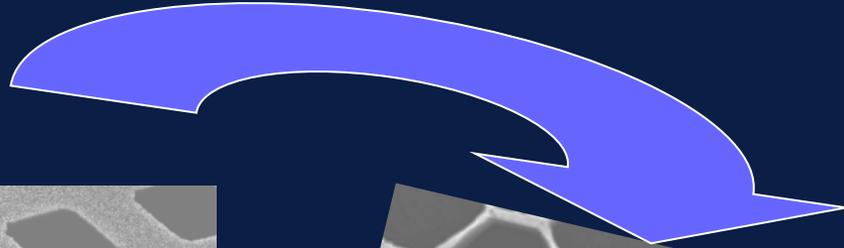


Dried structure





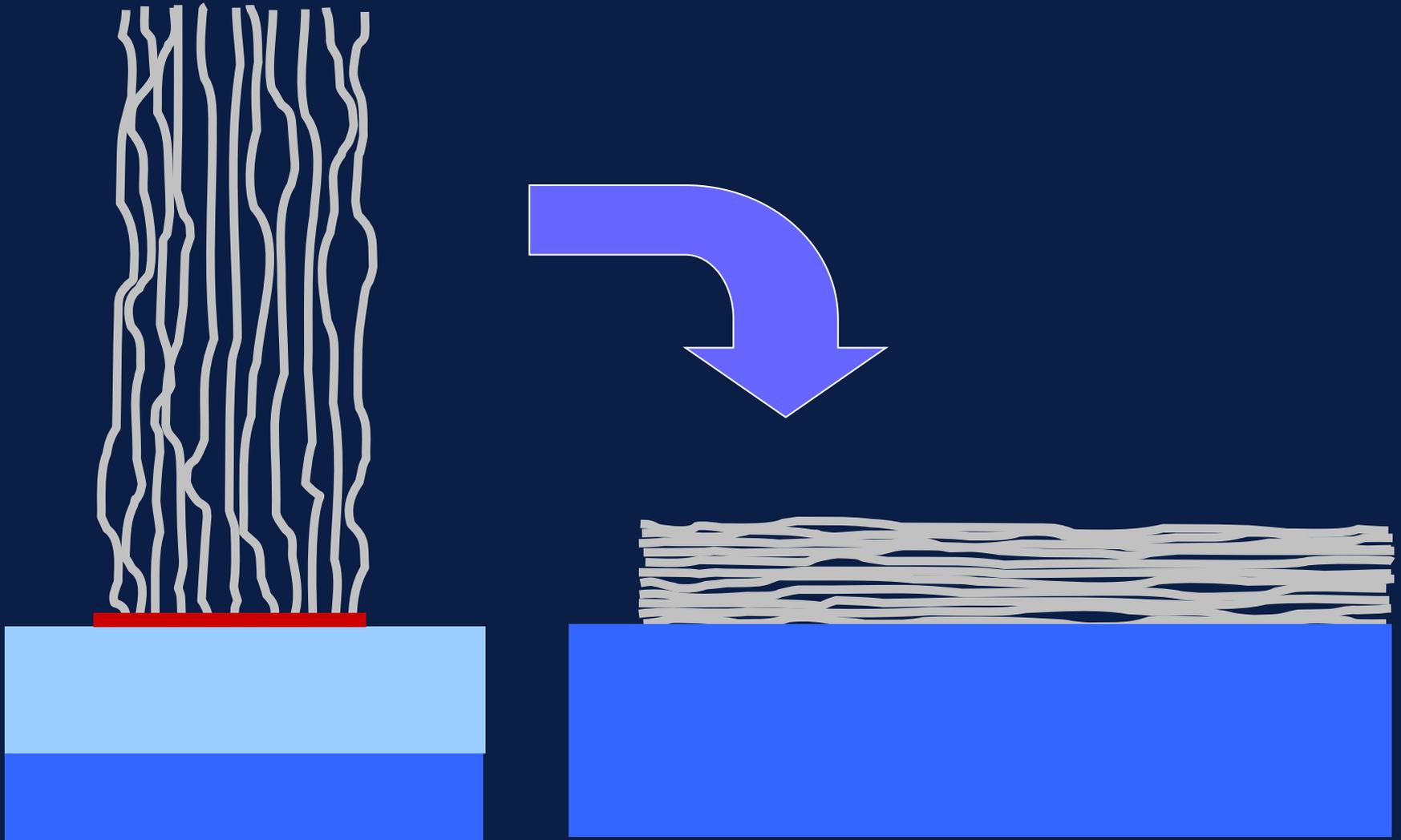
Surface forces dominates



Unequal angles become equal angles.
Final structure depends on initial structure and surface forces

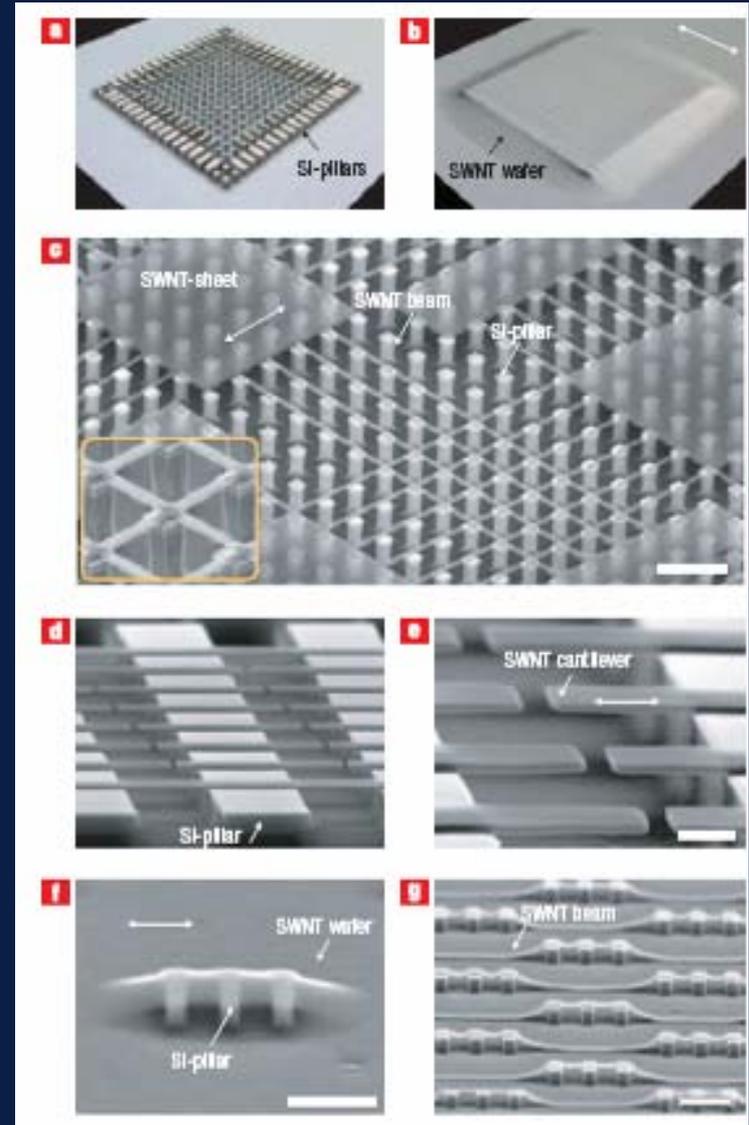
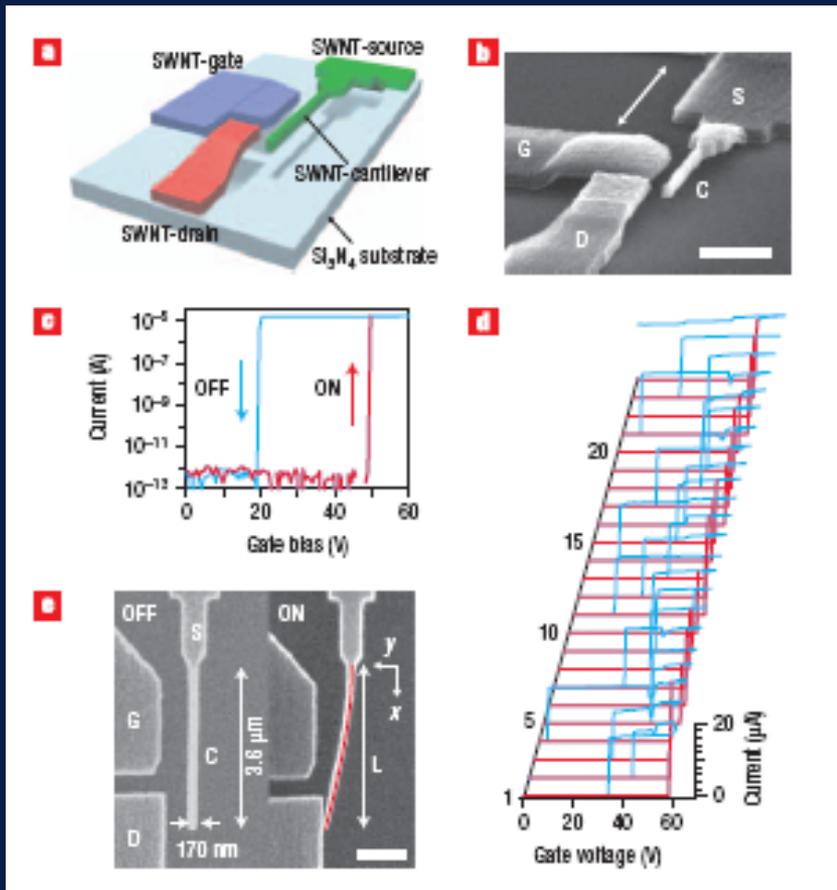


Horizontal Aligned CNT films





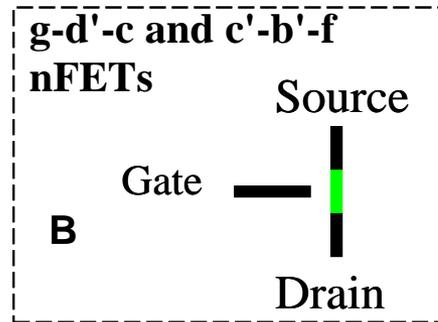
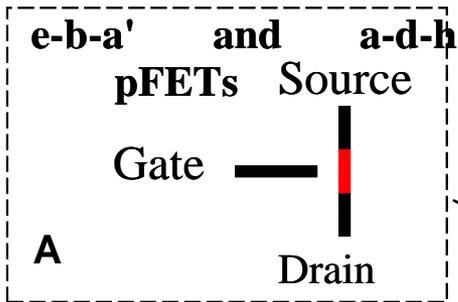
AIST Japan group working on in-plane aligned CNT MEMS



Yuhei Hayamizu, Takeo Yamada, Kohei Mizuno, Robert C. Davis, Don N. Futaba, Motoyuki Yumura, & Kenji Hata **Nature Nanotechnology** 3, 241 (2008).



Complementary logic gate geometry



DNA assembly of MC

