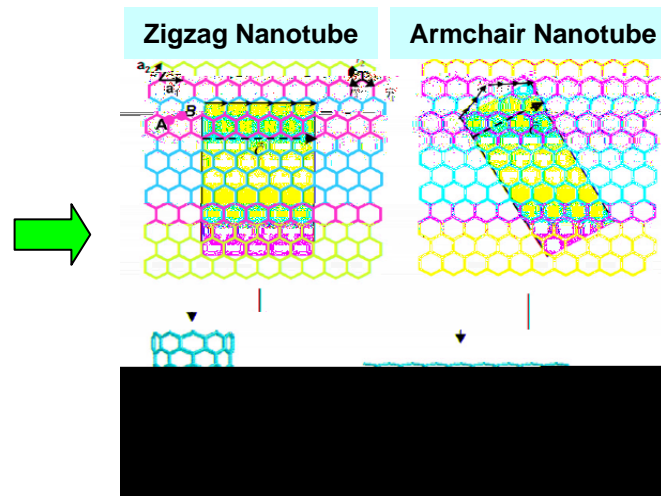
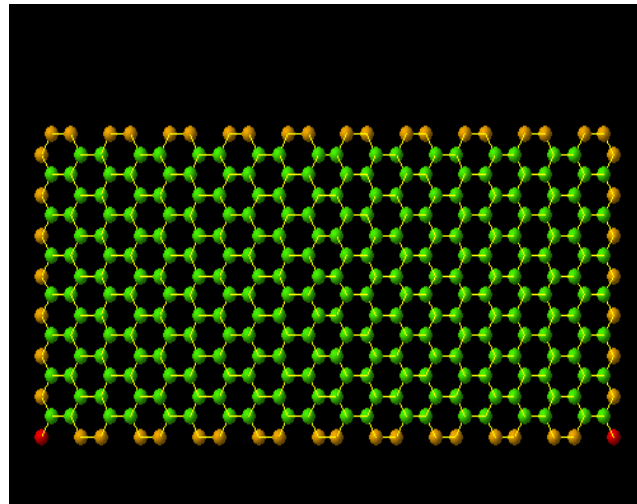


Structure Controlled Growth of Single-walled Carbon Nanotubes on Surface

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College of Chemistry and Molecular Engineering
Peking University, Beijing 100871, P. R. CHINA
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Introduction of Single-walled Carbon Nanotubes

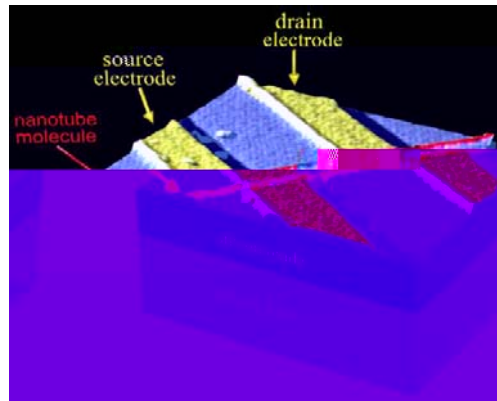


$$\mathbf{C}_h = n\mathbf{a}_1 + m\mathbf{a}_2 \equiv (n,m)$$

$$d_t = \sqrt{3}a_{C-C}(n^2 + m^2 + nm)^{1/2} / \pi$$

$$\theta = \tan^{-1}\left(\frac{\sqrt{3}m}{2n+m}\right)$$

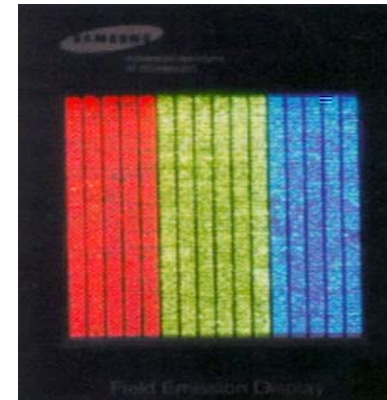
$$n-m = \begin{cases} 3p & \text{metal} \\ 3p \pm 1 & \text{semiconductor} \end{cases}$$



Nanotube transistor



Nanotube RAM

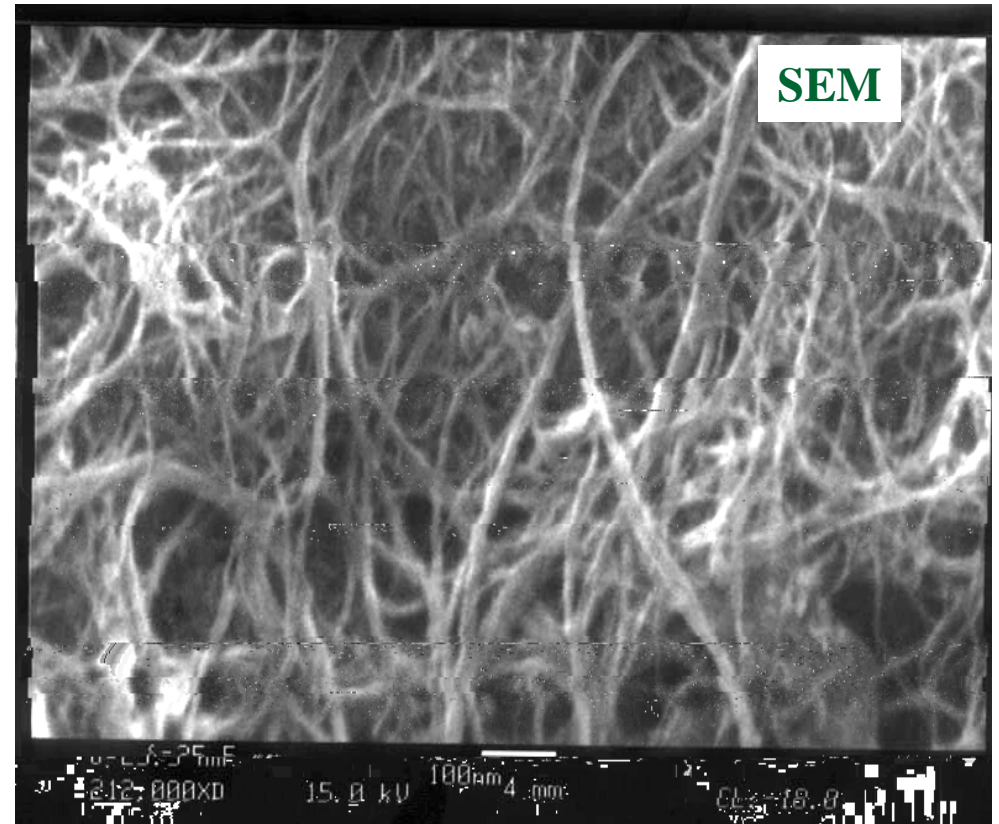
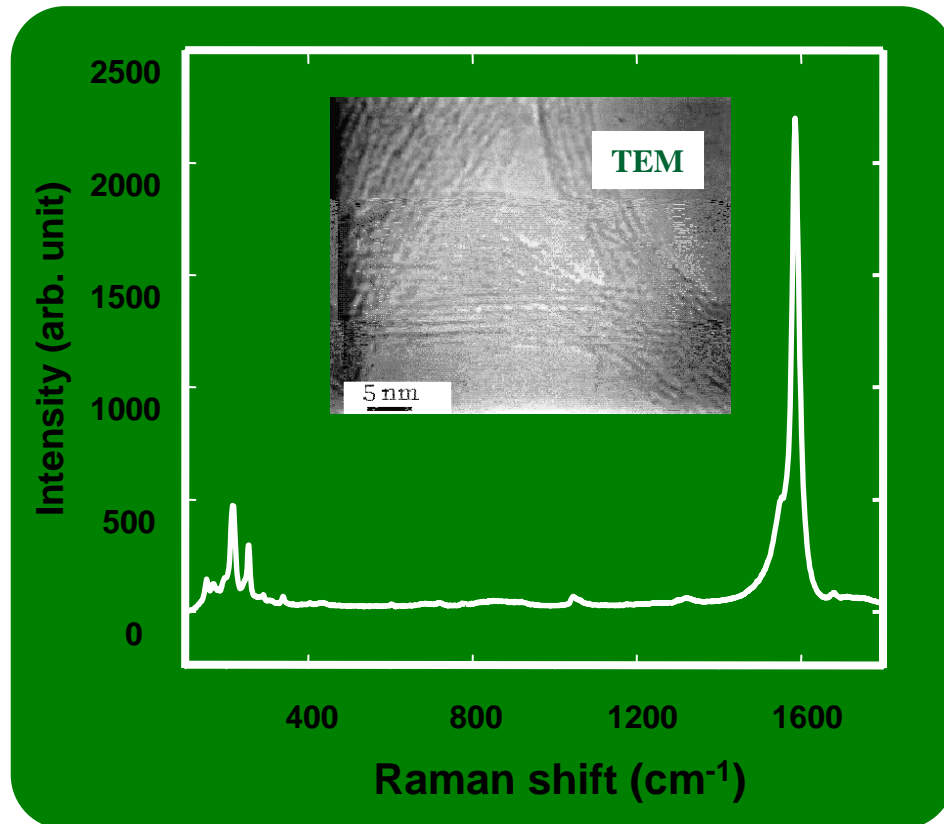


Flat panel display



Nanotube interconnect

A Scalable CVD Synthesis of High-Purity SWNTs with Porous MgO as Support Materials

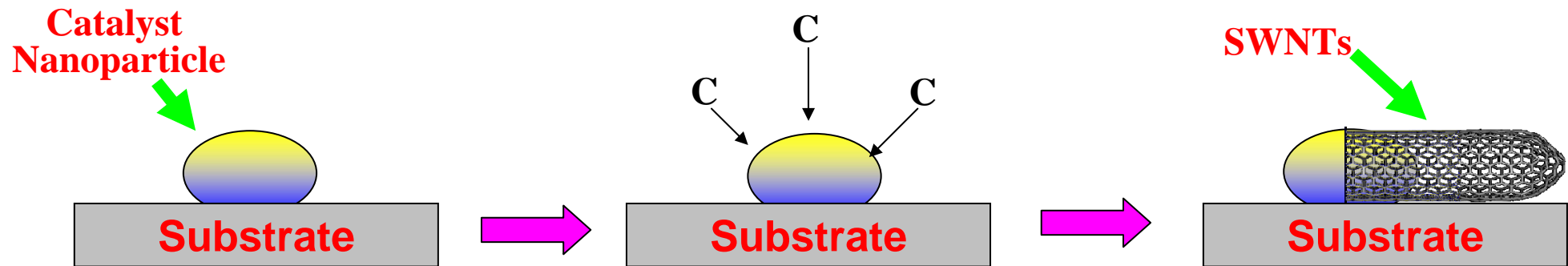


Support: MgO; Catalysts: Fe; Carbon Source: CH₄

J. Zhang et. al. , J Mater Chem, 12(4): 1179-1183, 2002 Carbon, 40(12): 2282-2284, 2002 Carbon, 40(14): 2693-2698, 2002

Surface Growth of SWNTs by CVD

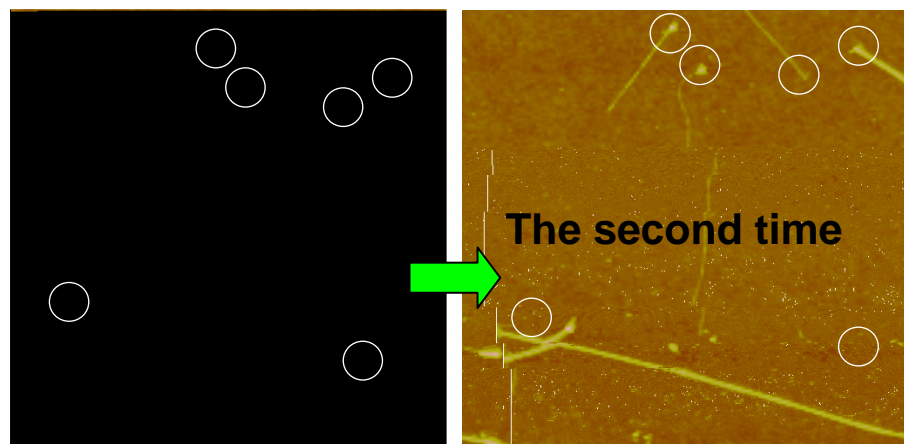
Growth Process:



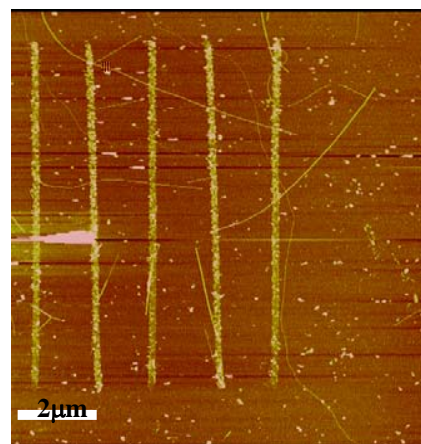
Questions:

1. Growing SWNTs on Surface Directly with Controlled Density, Position and Orientation
2. Growing SWNTs on Surface with Controlled Diameter
3. Growing SWNTs on Surface with Controlled Metallic and Semi-conducting Properties
4. Growing SWNTs on Surface with Controlled Chirality

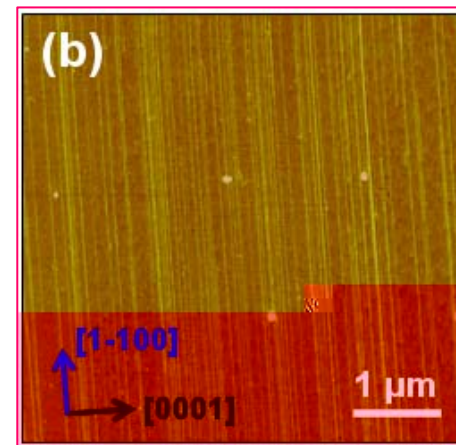
Controlled CVD Growth of SWNTs on Surface



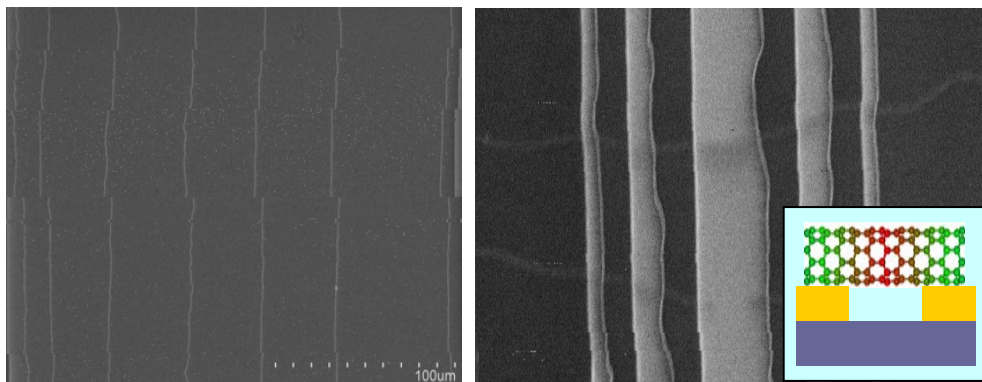
J. Phys. Chem. B. 2004, 108, 12665



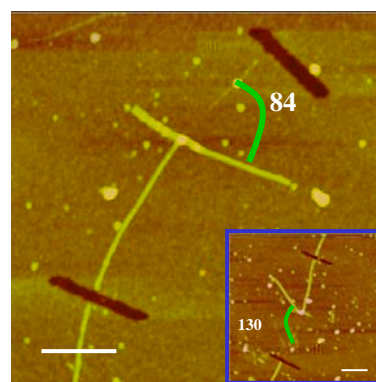
J. Phys. Chem. B. 2005, 109, 10946



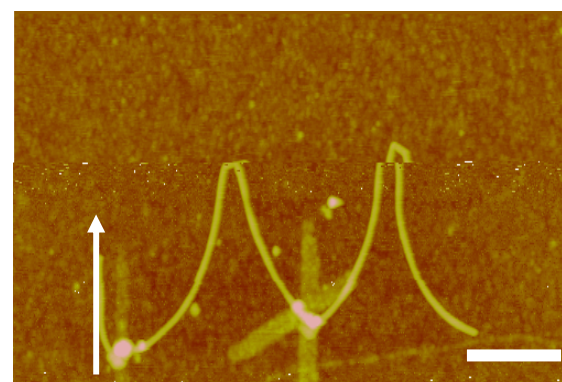
J. Phys. Chem. C. 2008, 112, 8319



J. Am. Chem. Soc. 2005, 127, 17156



J. Am. Chem. Soc. 2005, 127, 8268.
J. Phys. Chem. B. 109 (2005) 2657-2665

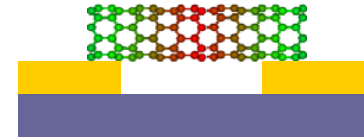


Challenges for the Application of Carbon Nanotubes in Future Device

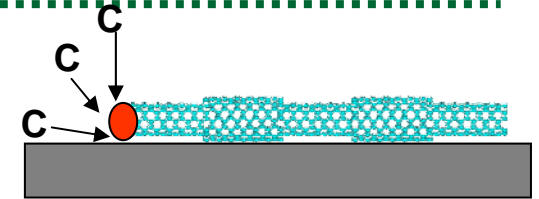
- 1 How to achieve a structure-controlled synthesis of nanotubes ?
 - Diameter
 - Lattice geometry (armchair, zigzag, chirality)
 - Semiconducting or Metallic Nanotubes
 - 2 How to fabricate a desired device structure ?
 - Controlled surface growth
 - Manipulation
 - 3 What architecture should the nanotube device have ?
 - 4 How to integrate trillions of individual nanotube devices ?
-

Outline

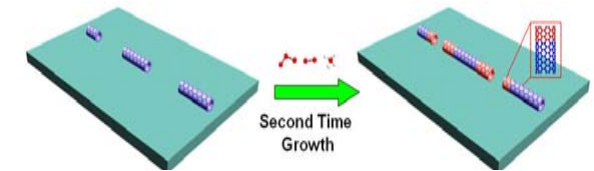
- ✓ **Control of local deformation of SWNTs**
—— Growth on designed surface



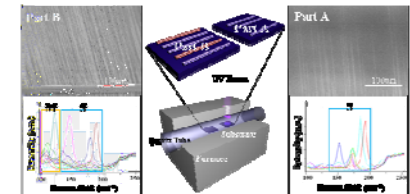
- ✓ **Control of local tube diameters of SWNTs**
—— Temperature-oscillation growth



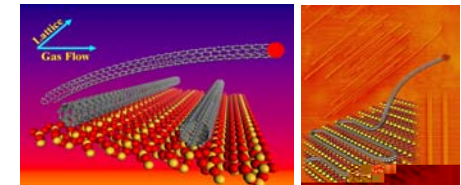
- ✓ **Control of chirality of SWNTs**
—— Cloning growth



- ✓ **Control of metallic-/semiconducting- of SWNTs**
—— UV irradiation assistance CVD growth



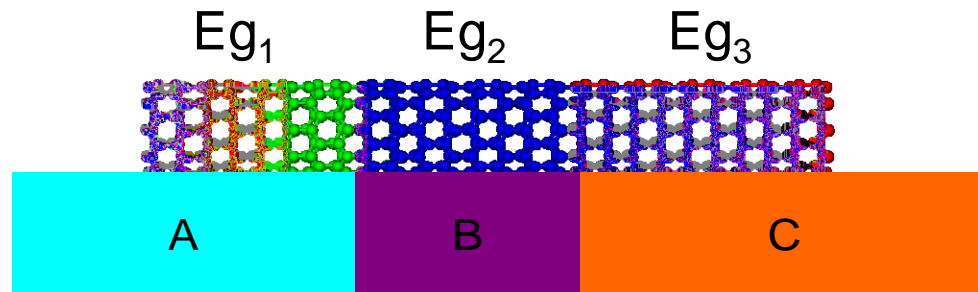
- ✓ **Control of local conformation and architectures**
—— Cooperative growth of floating and lattice oriented modes



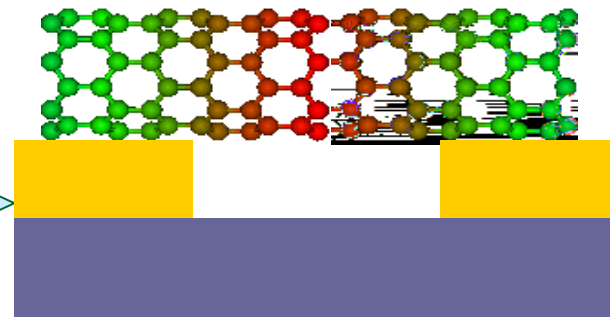
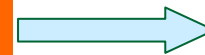
Control of local deformation of SWNTs

—— Growth on designed surface

Substrate-Induced Band Structure Variation

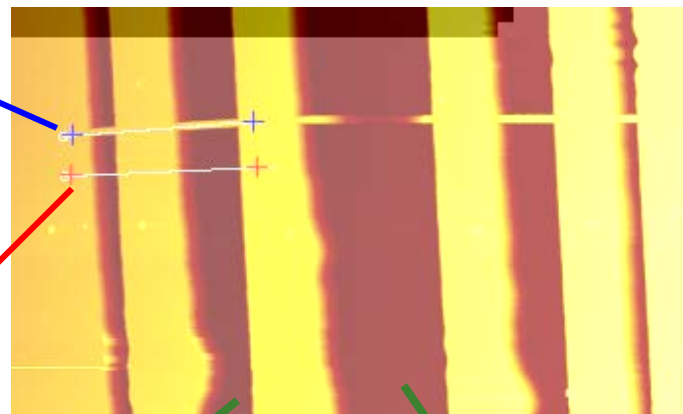


The goal



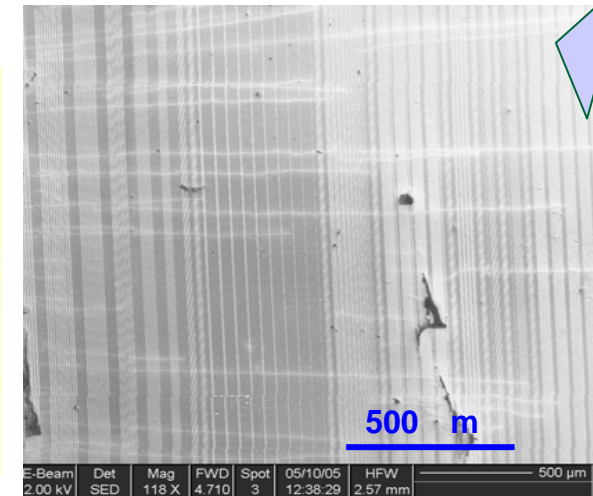
First experimental system

AFM image



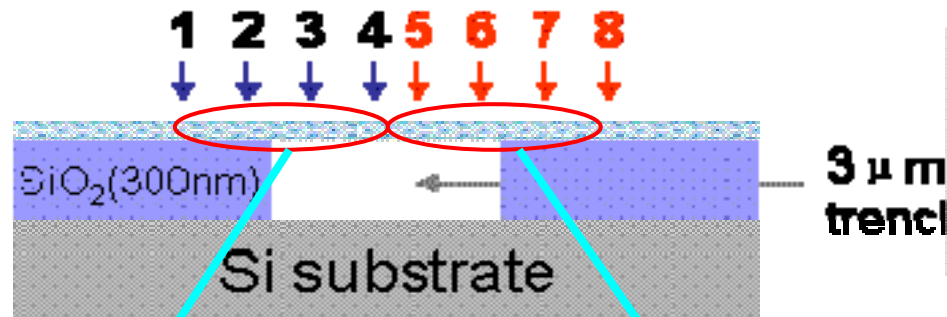
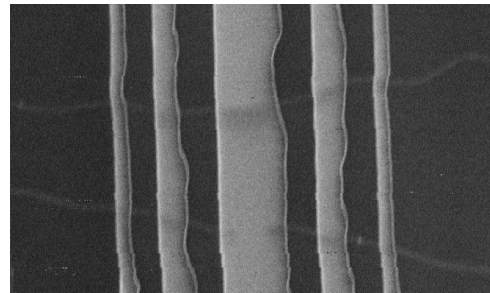
3 μm

6 μm

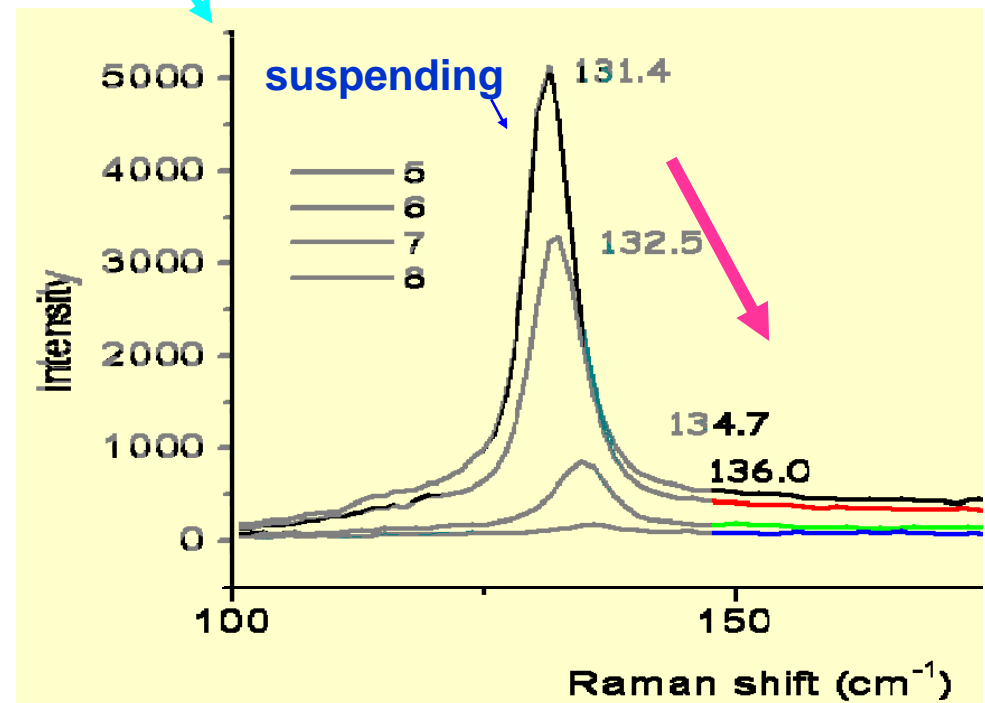
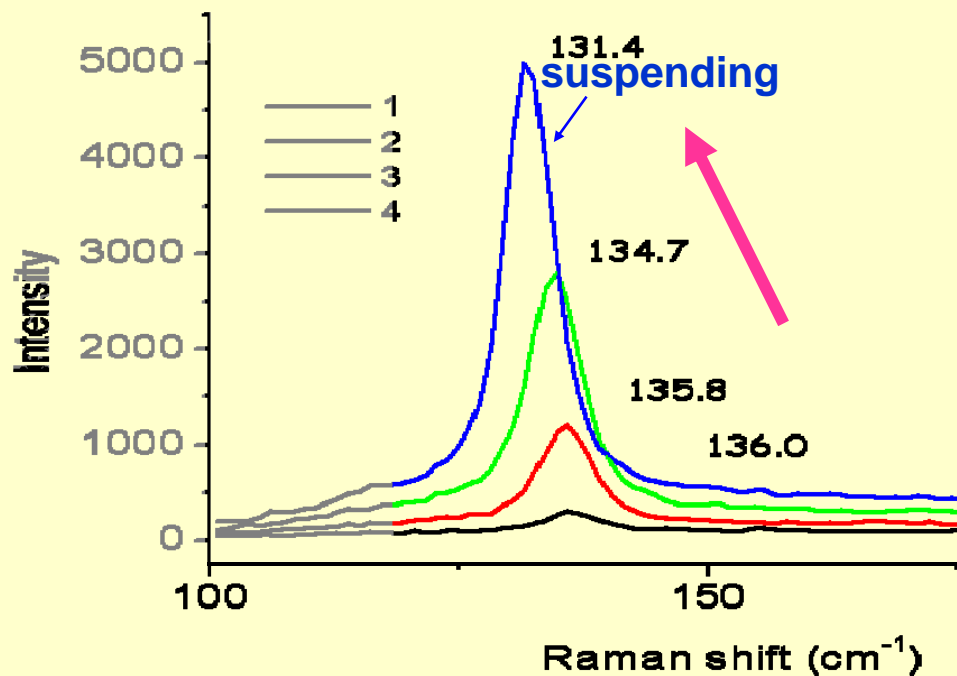


SEM image

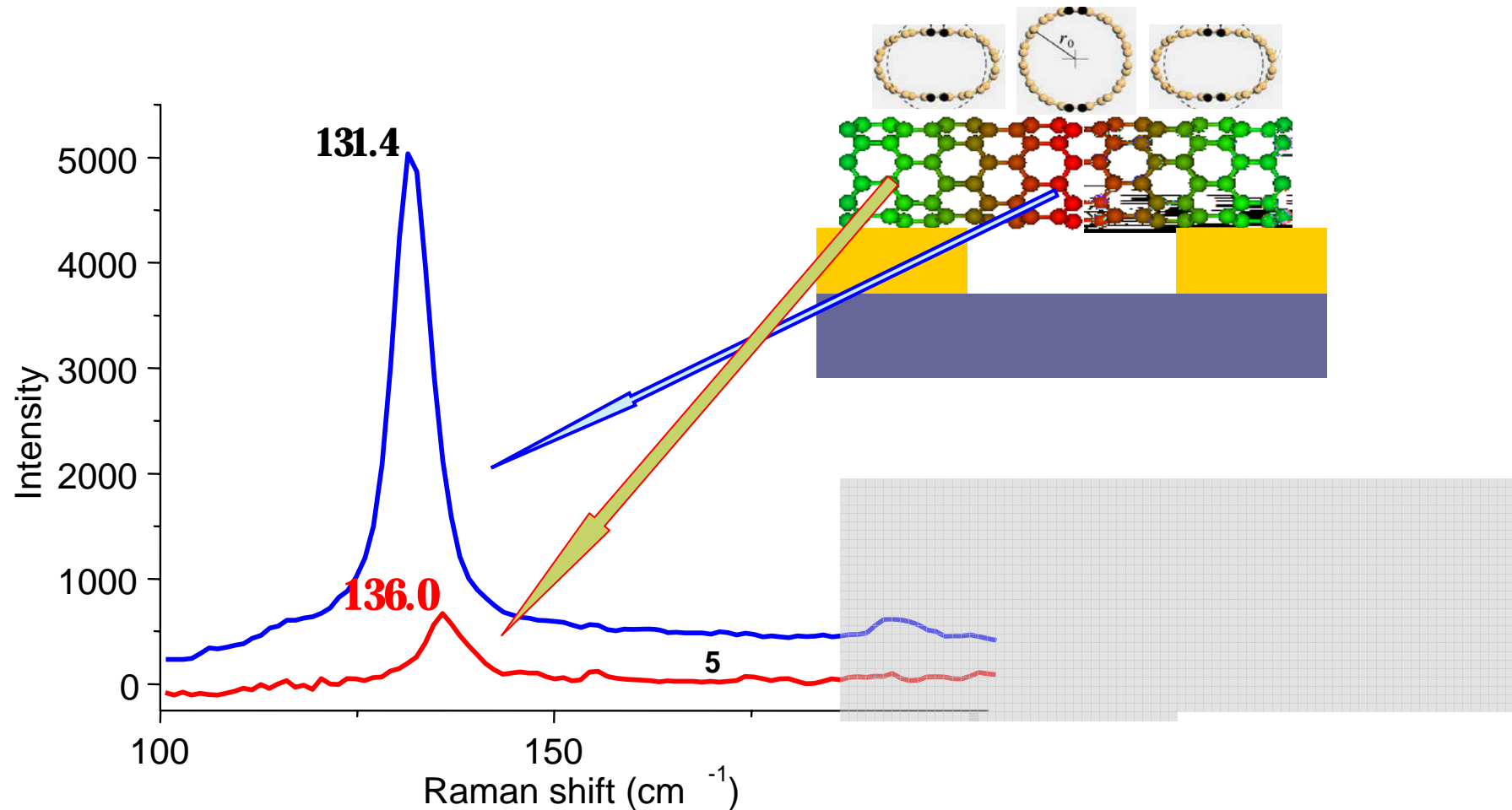
Raman Mapping of Partially Suspended SWNT



- laser=632.8nm
- $E_{\text{laser}}=1.96\text{eV}$
- Cumulating time:10s

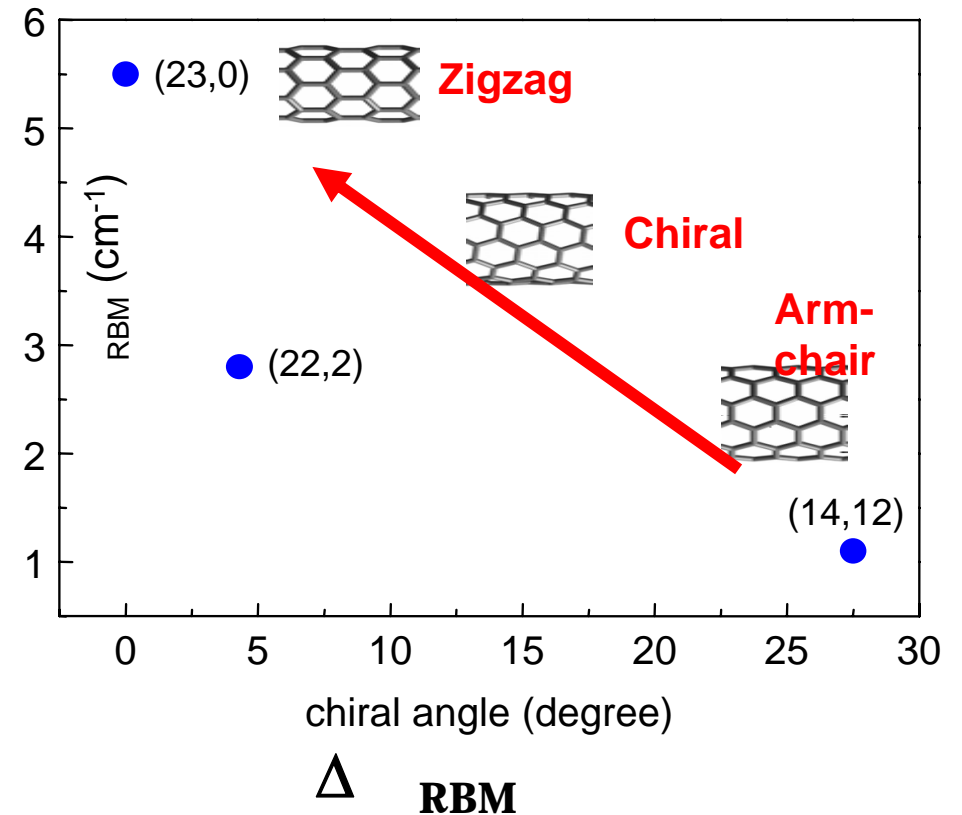
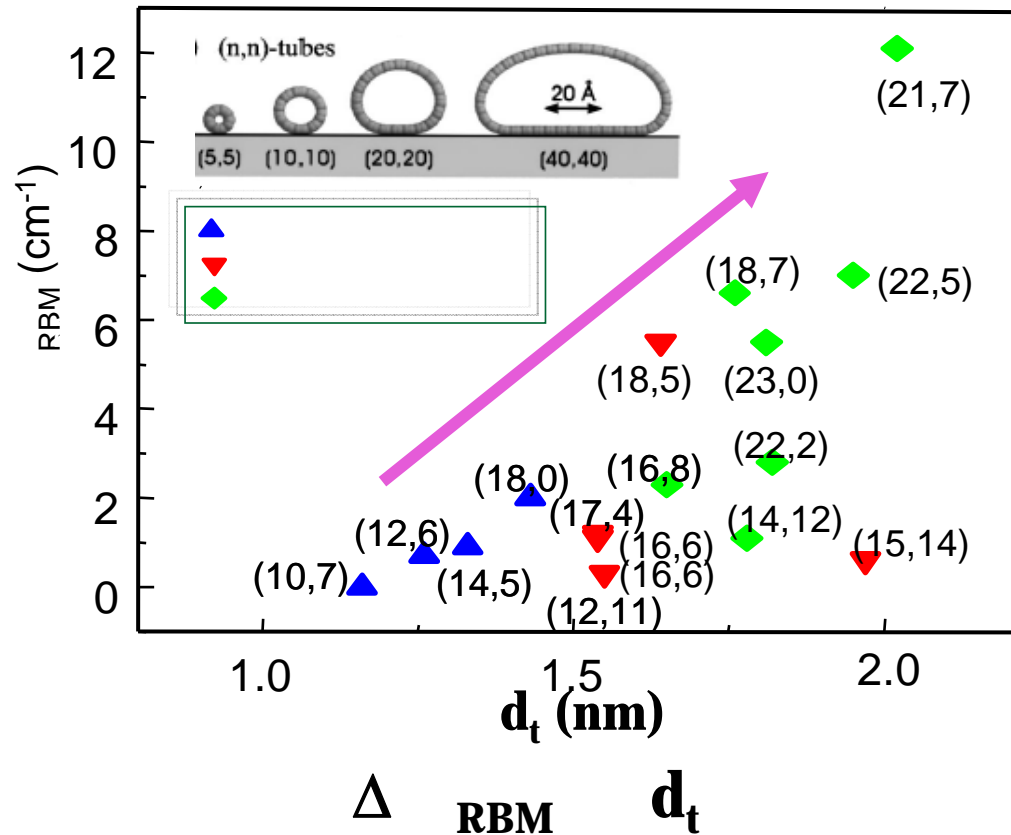


What is the Origin of Raman Spectrum Variation ?



Dependence of Chiral Angle

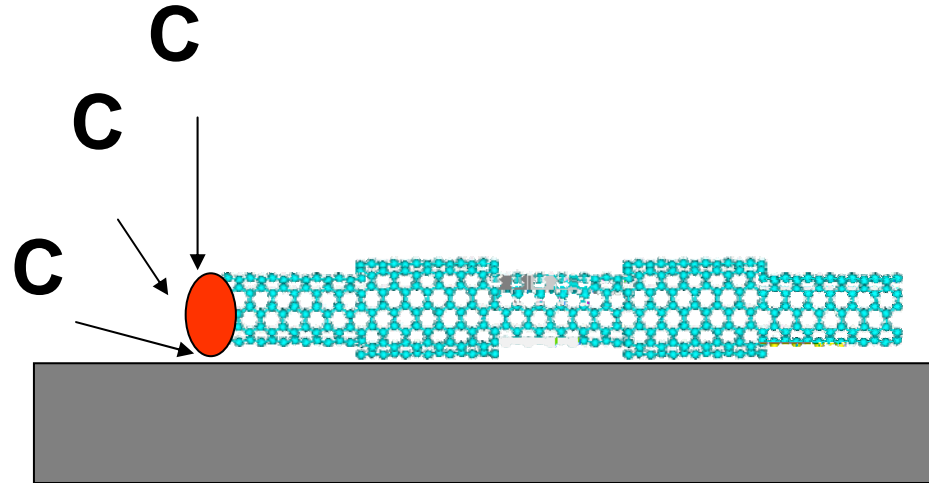
RBM on Diameter and



Good agreement with theoretical work about radial deformation.

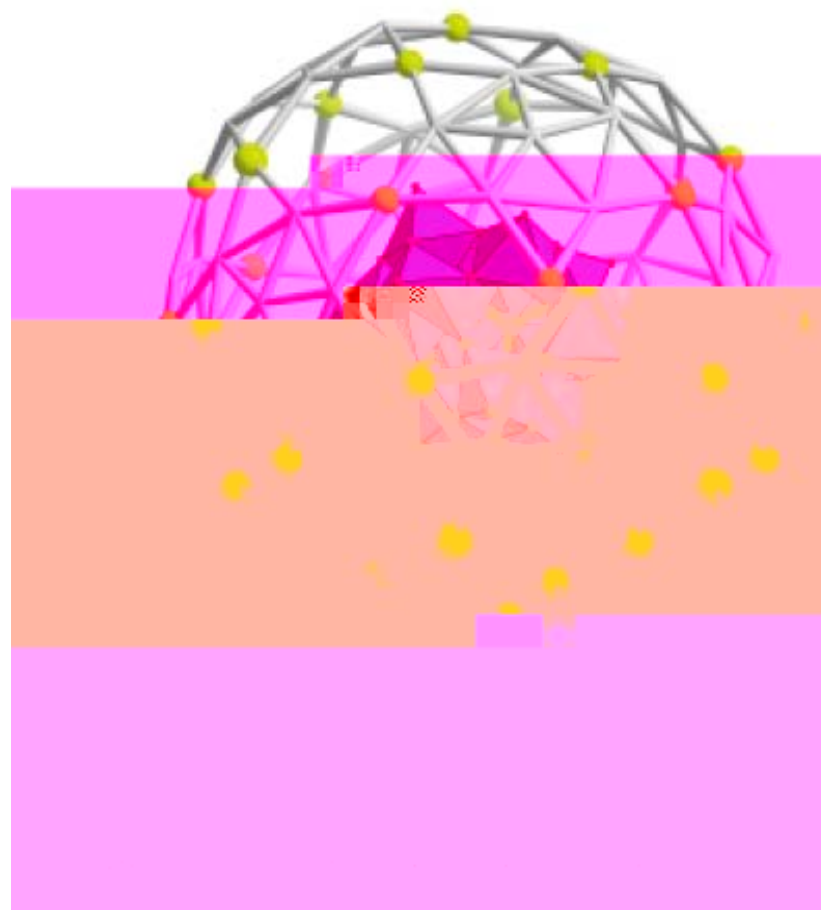
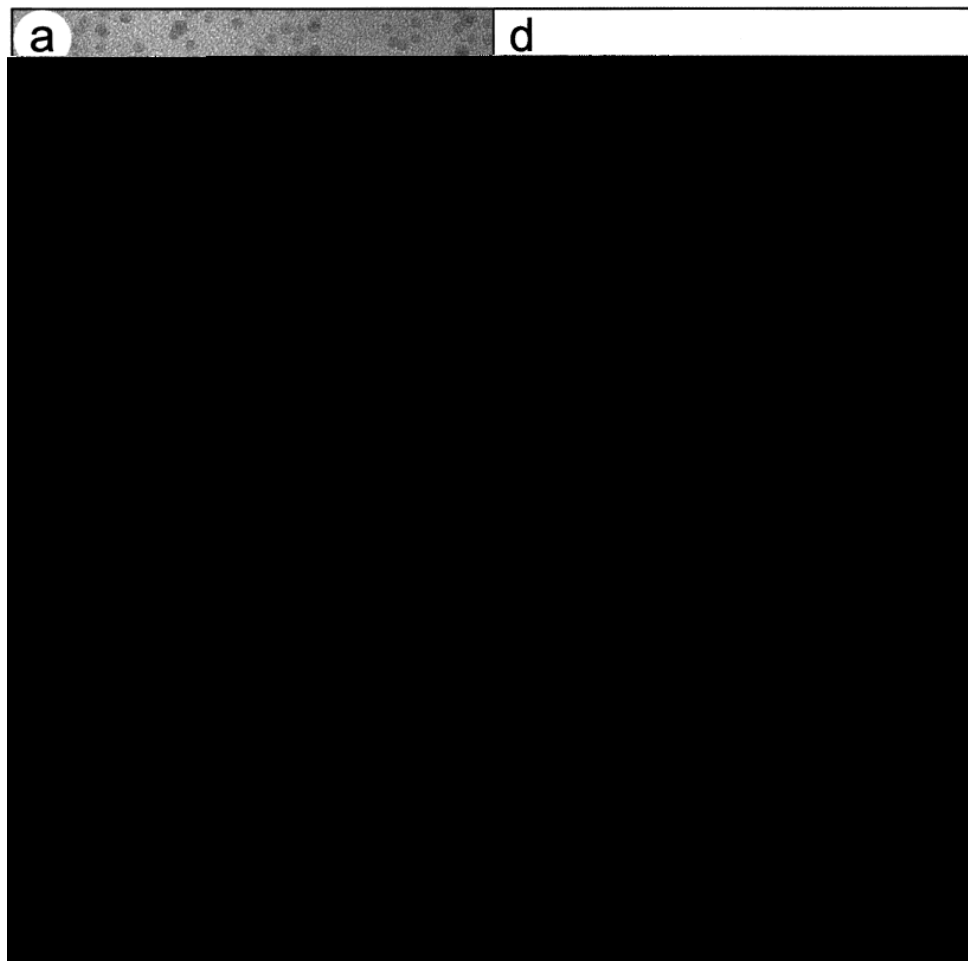
Band Structure Variation between Suspended and Non-suspended SWNT

No.	Location	RBM ^(c) m ⁻¹	I _{AS} /I _S	E _{ii}	E _{ii} (eV)	ΔE _{ii} (meV) (SiO ₂ -Sus)	(n,m)	(2n+m) MOD3
1	Suspended	282.7	0.12	E22S	1.9522	-3.1	(7,5)	1
	SiO ₂	282.7	0.085		1.9491			
2	Suspended	243.6	1.26		1.9736	-4.8	Difficult	
	SiO ₂	244.8	0.95		1.9688			
3	Suspended	200.4	0.13	E11M	1.9514	0	(13,4)/(14,2)/(15,0)	0
	SiO ₂	200.4	0.14		1.9511			
4	Suspended	187.3	0.13	E11M	1.9517	+0.9	(11,8)	0
	SiO ₂	188.3	0.15		1.9526			
5	Suspended	169.7	0.40	E33S	1.9584	-2.1	(14,7)	2
	SiO ₂	169.4	0.28		1.9563			
6	Suspended	159.7	0.94	E33S	1.9640	-0.6	(12,11)/(16,6)/(17,4)	2
	SiO ₂	159.8	0.79		1.9634			
7	Suspended	156.4	0.91	E33S	1.9671	-9.4	(12,11)/(15,8)/(20,1)	2
	SiO ₂	156.7	0.39		1.9577			
8	Suspended	148.3	1.16	E33S	1.9640	-3.7	(16,8)	1
	SiO ₂	148.2	0.60		1.9603			
9	Suspended	129.5	2.11	E44S	1.9698	-14.1	(24,1)/(18,10)/(19,8)/23,3)	1
	SiO ₂	129.9	0.44		1.9557			
10	Suspended	116.8	0.55	E44S	1.9588	+4.3	(25,3)/(23,7)/(20,10)	2
	SiO ₂	116.8	1.20		1.9631			
11	Suspended	115.9	1.50	E44S	1.9648	+2.6	(23,7)/(20,10) (25,3)	2
	SiO ₂	115.7	1.25		1.9672			
12	Suspended	110.7	1.01	E44S	1.9670	+26	(28,0)	2
	SiO ₂	110.8	3.07		1.9810			

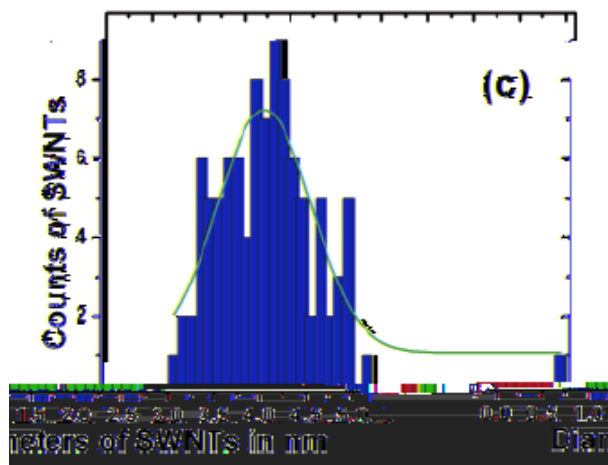
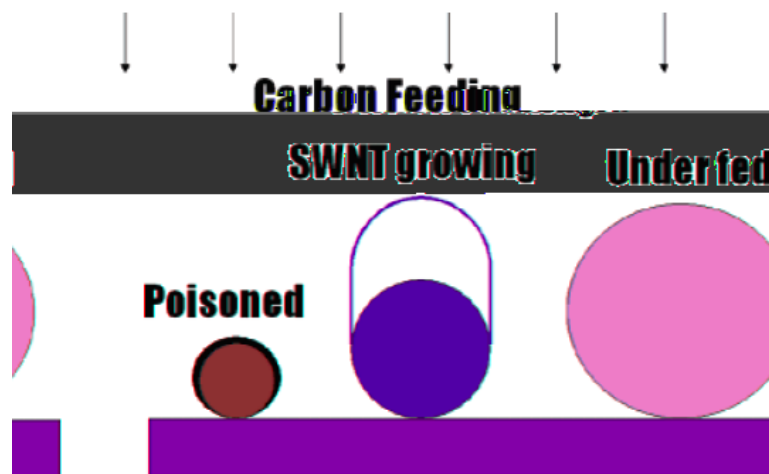


Control of local tube diameters of SWNTs
—— **Temperature-oscillation growth**

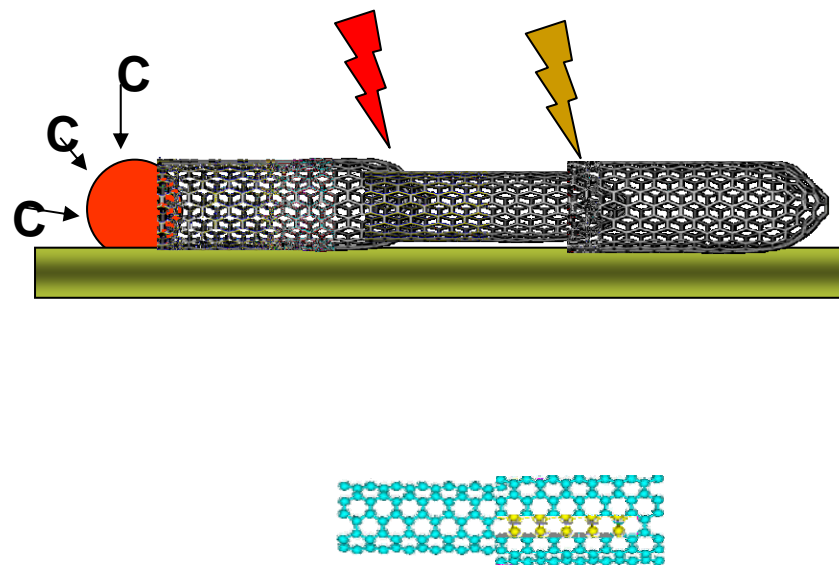
Controlling the Diameter of SWNT by Catalyst Particle



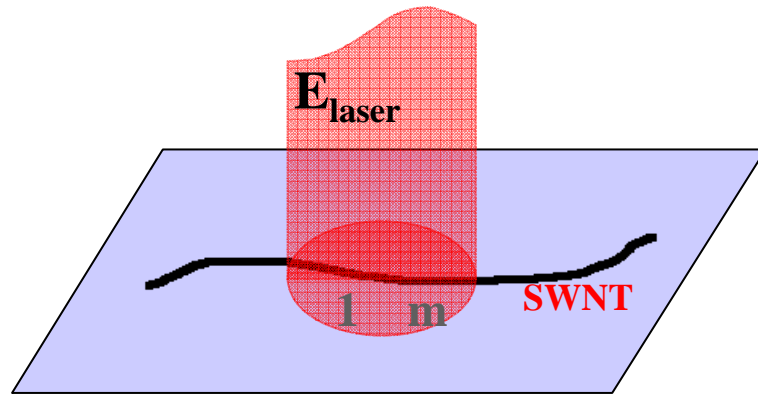
Controlling the Diameter by Carbon Feeding



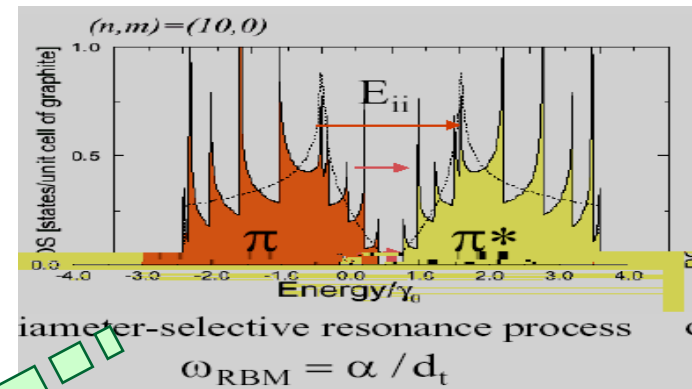
Our Approach: Tune the Diameter of SWNTs by Temperature



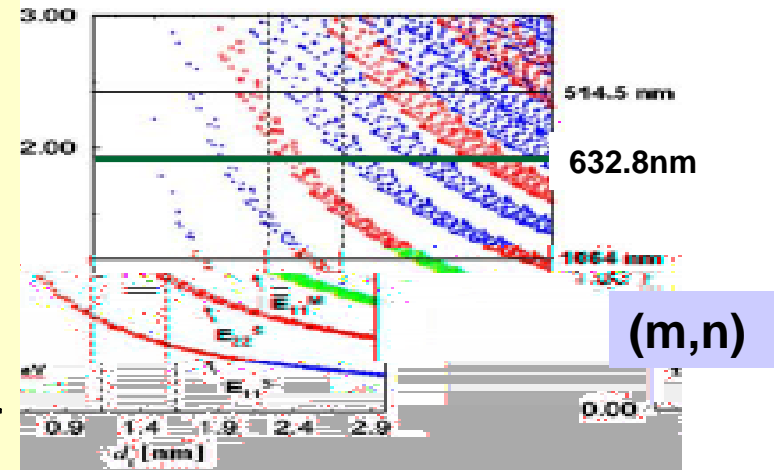
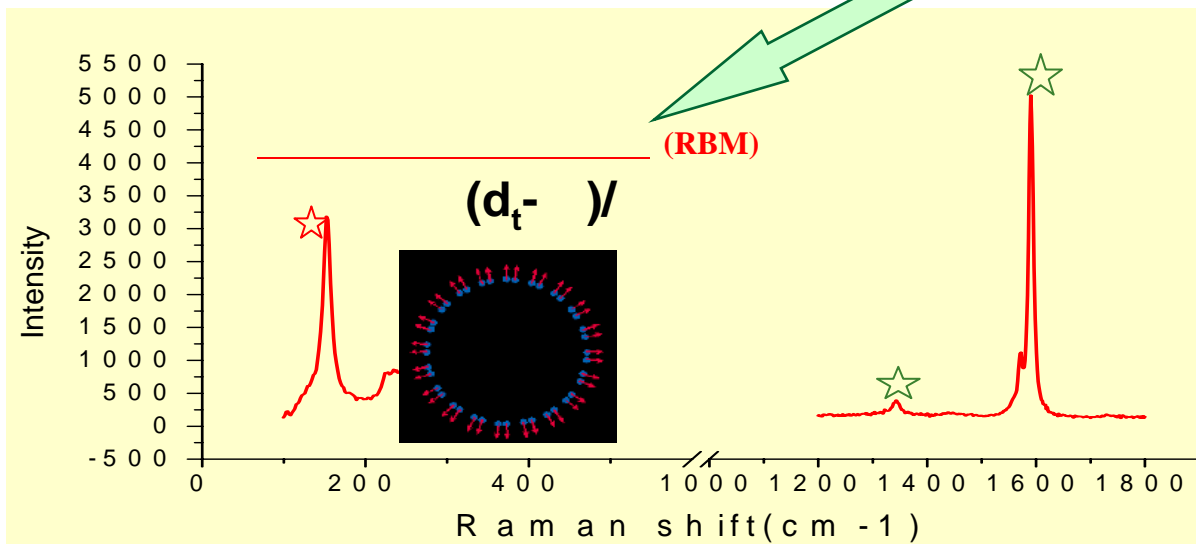
Micro-Resonance Raman Spectrum of Individual SWNTs



Micro-Raman spectroscopy

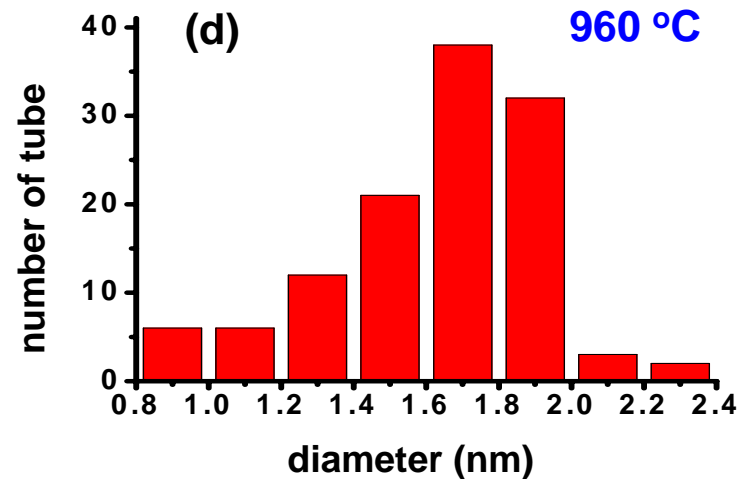
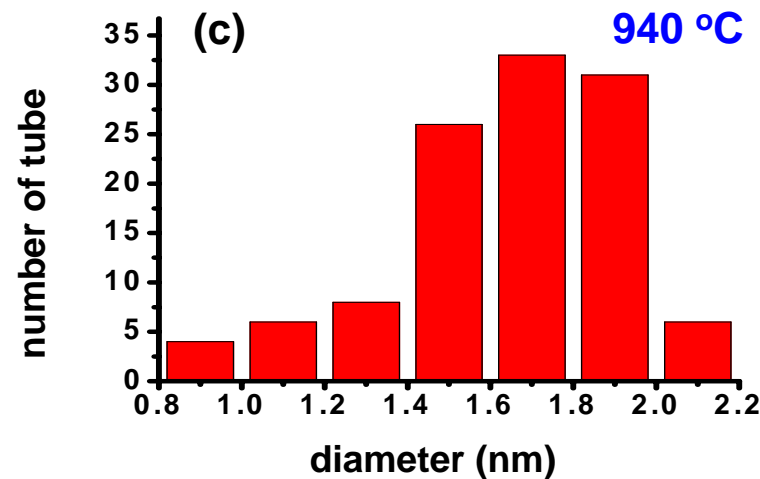
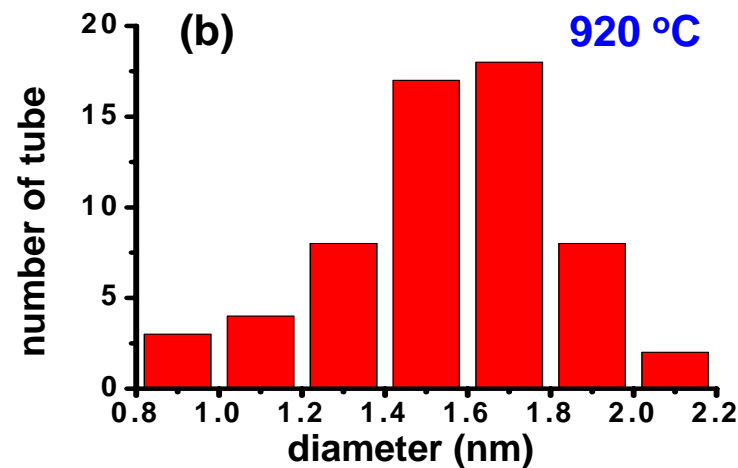
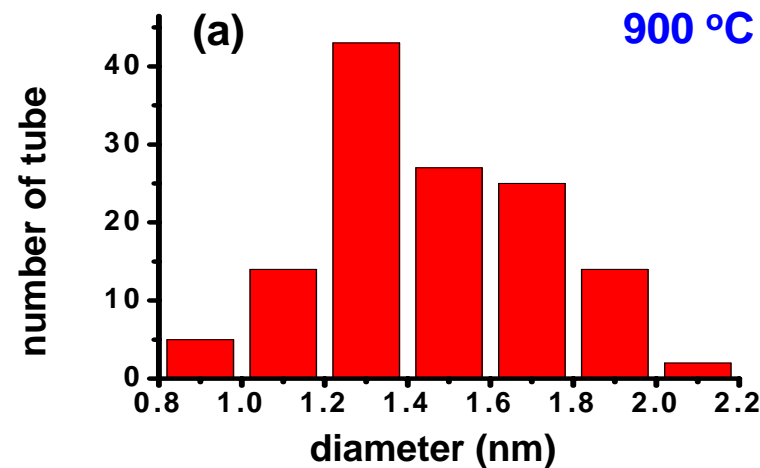


Resonance condition: $E_{\text{laser}} = E_{ii}$



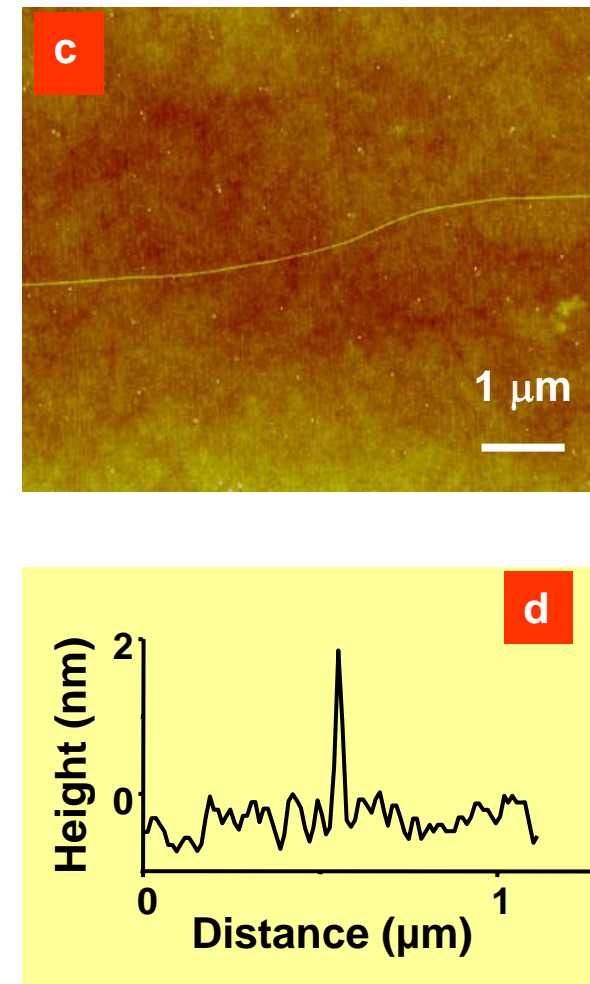
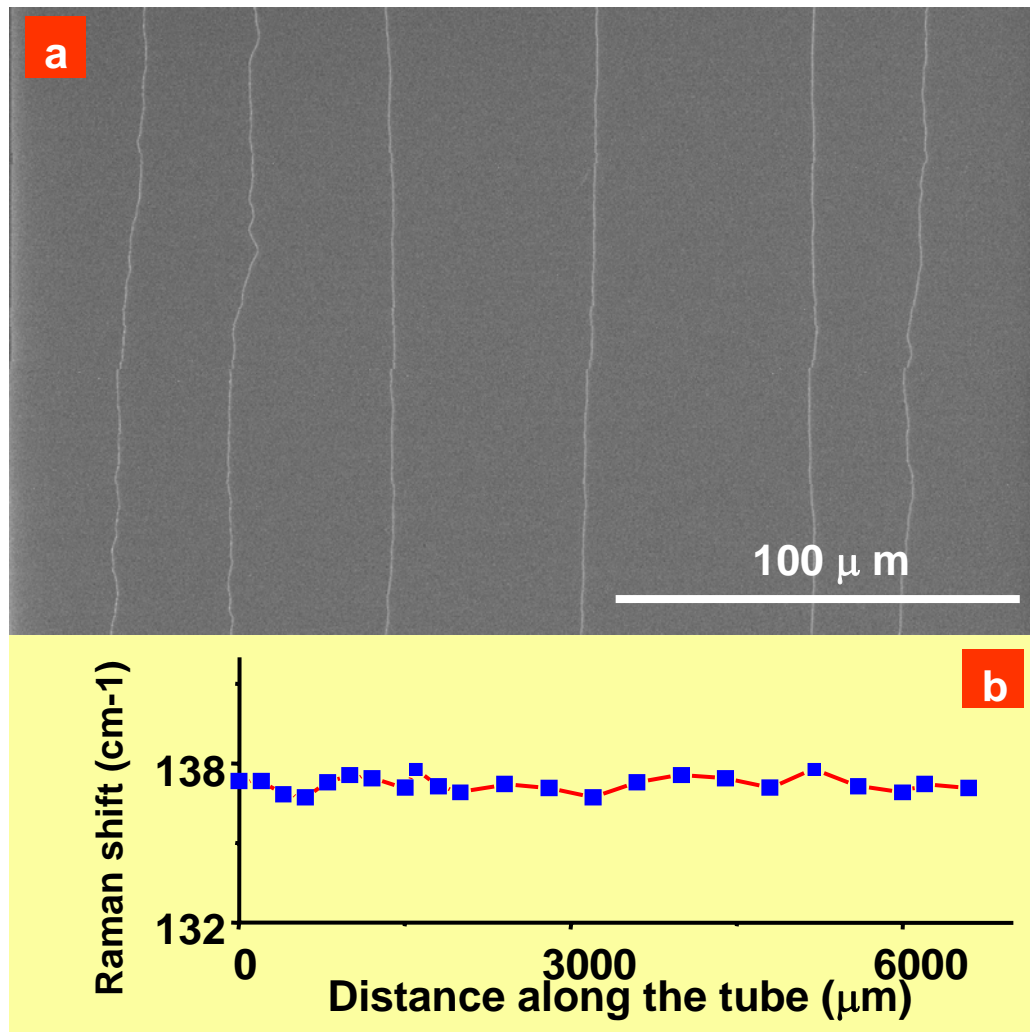
A power tool for both the atomic and electronic structure of SWNT !

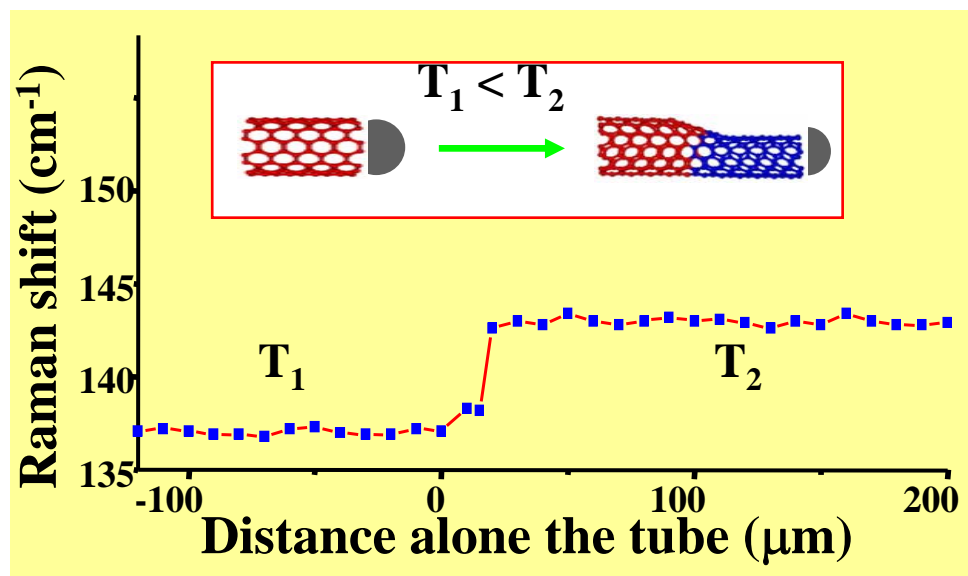
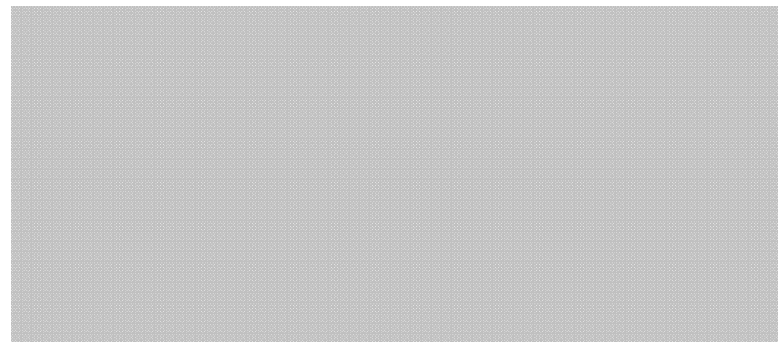
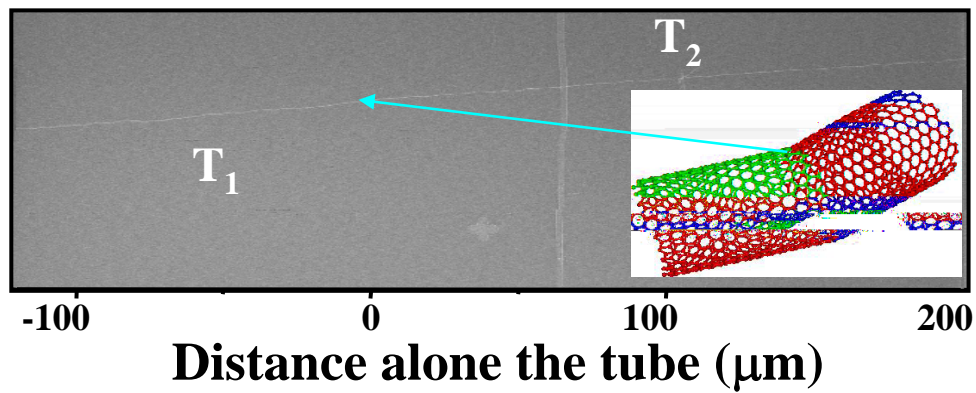
Growing SWNTs at Different Temperature



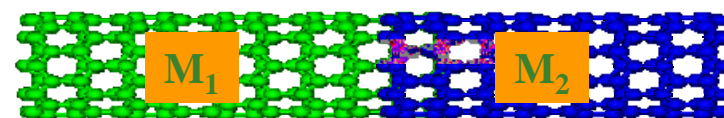
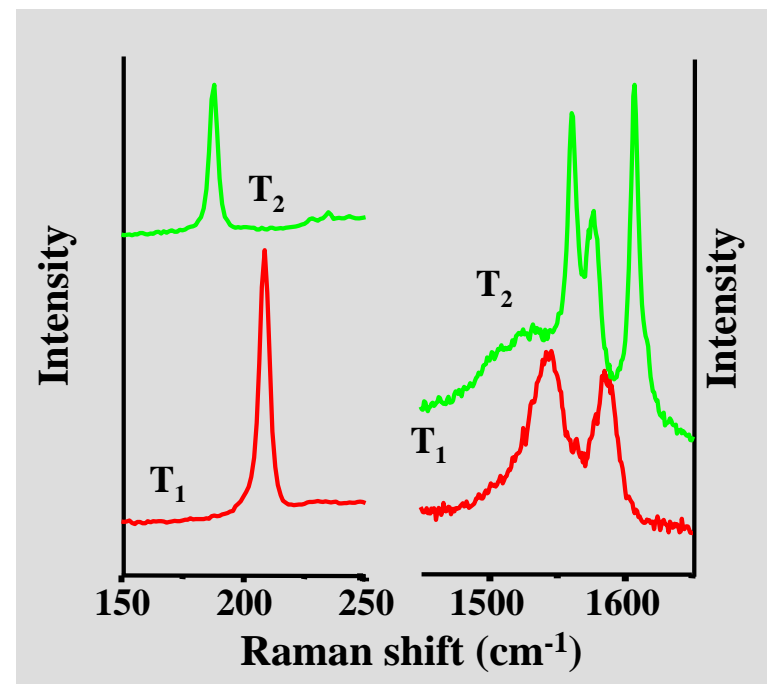
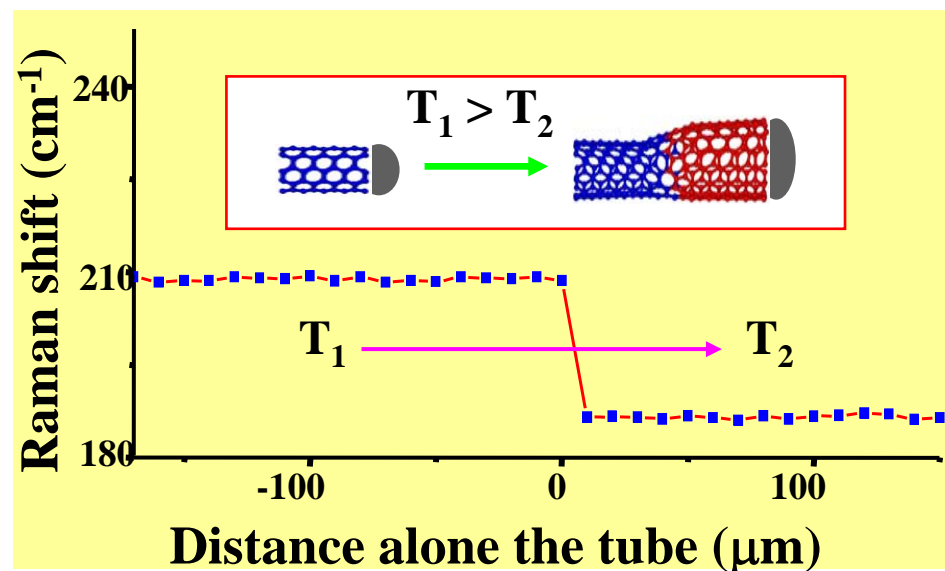
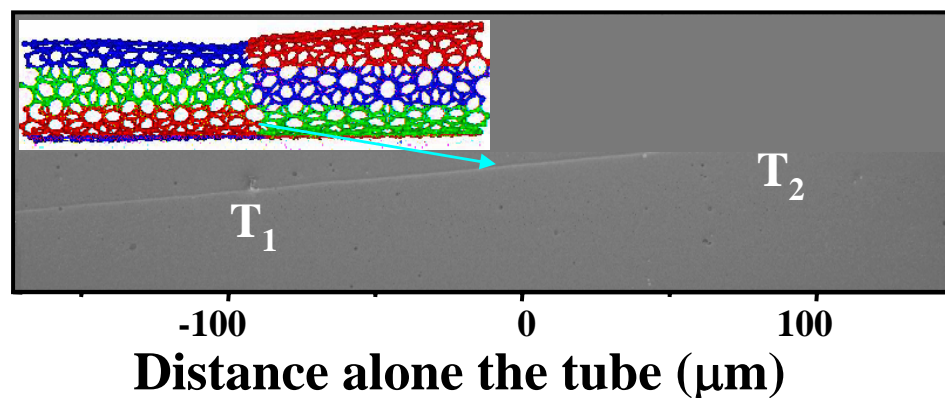
The catalyst nanoparticles might sinter or collide with each other at the high temperature

Constant-temperature CVD





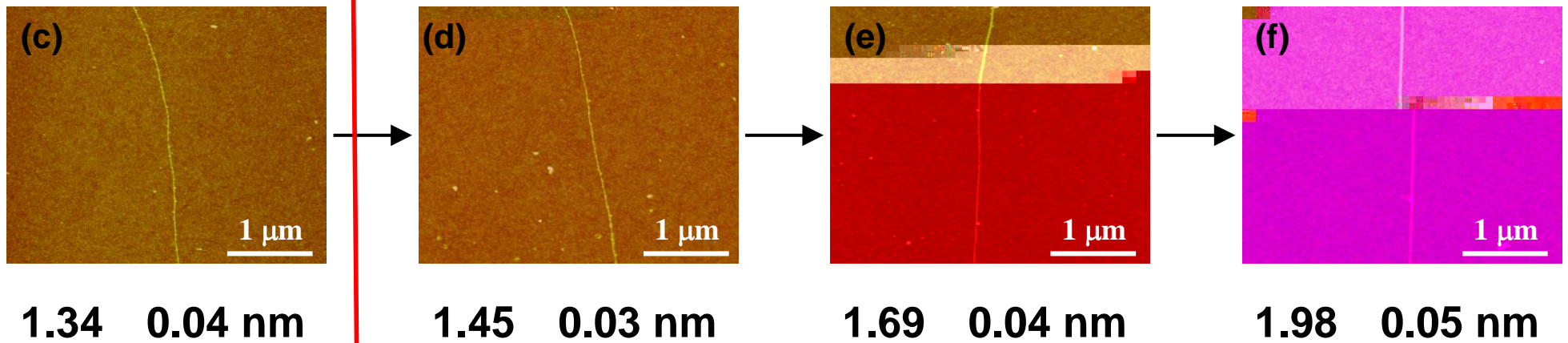
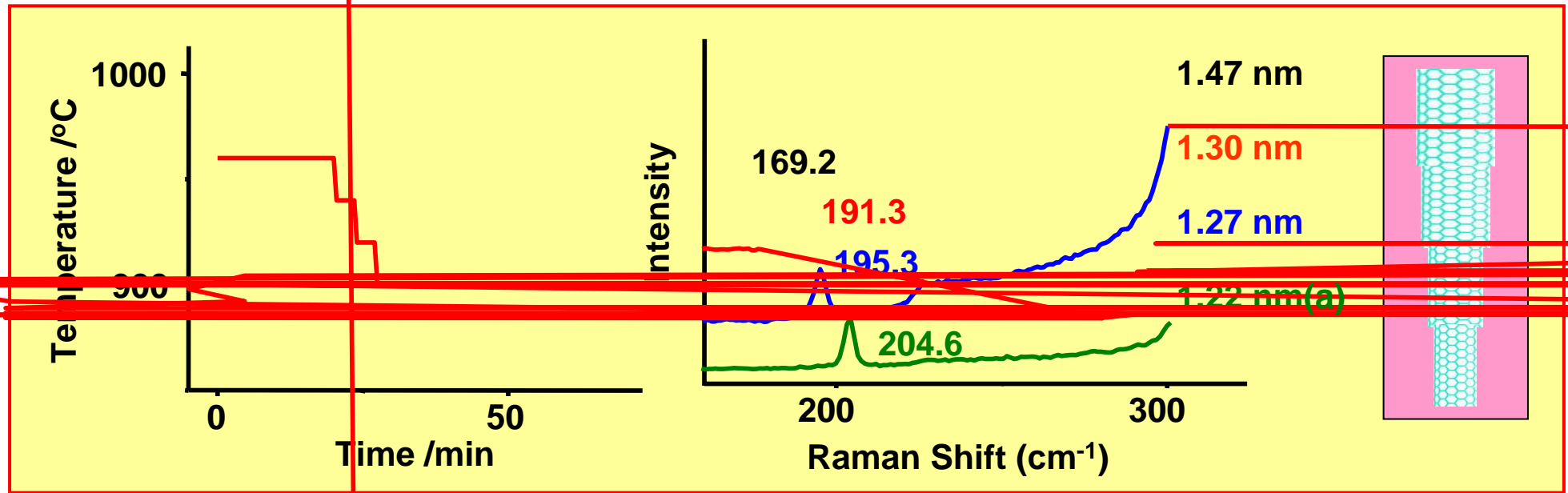
SWNTs Grown by one Time Temperature Oscillated CVD (From 950°C to 900°C)



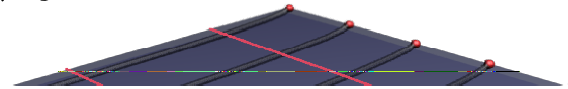
Metallic Metallic Nanojunction

Controlled Thinning of SWNTs via Temperature Step

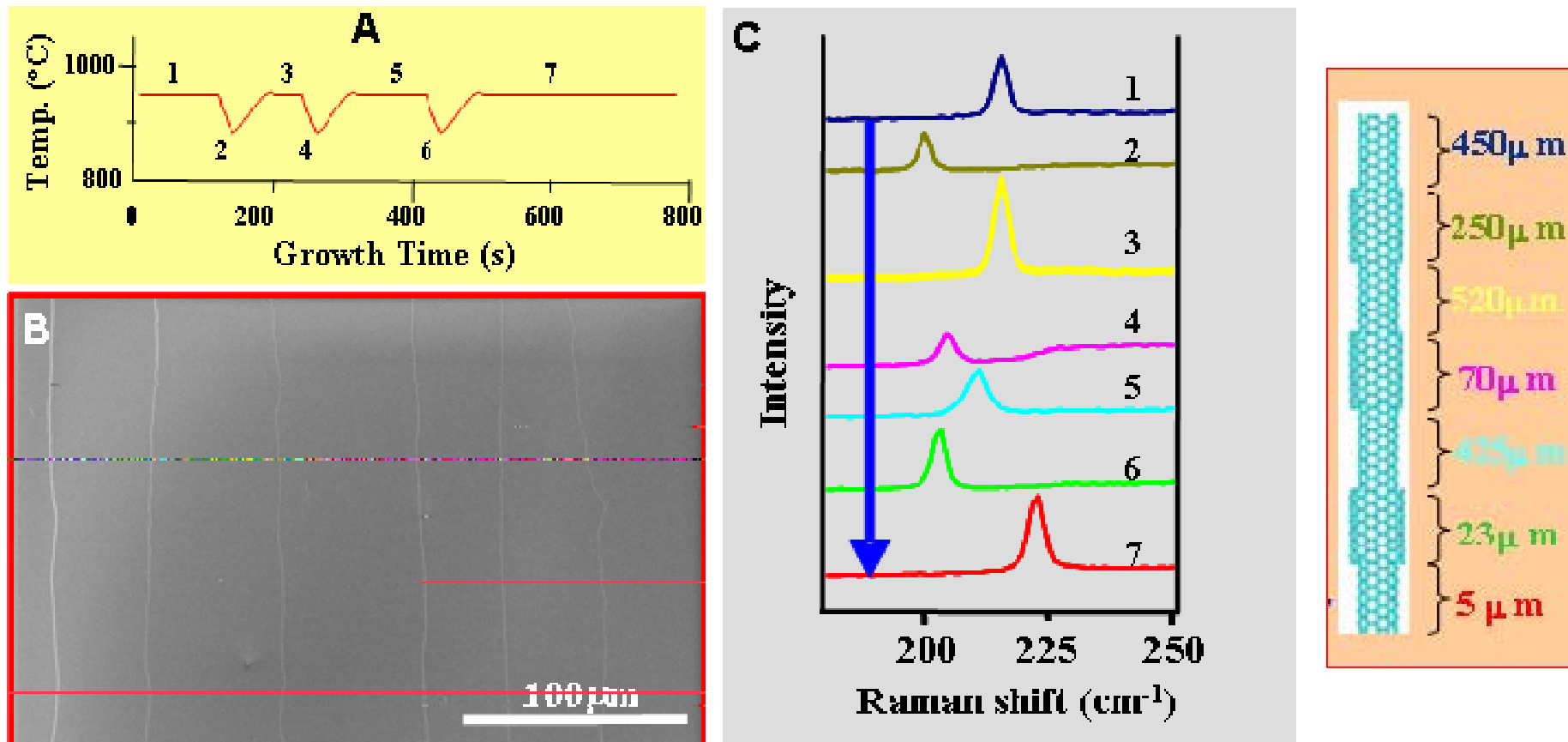
Controlled Thickening of SWNTs via Temperature Step-Down



$900\text{ }^{\circ}\text{C} \rightarrow 920\text{ }^{\circ}\text{C} \rightarrow 940\text{ }^{\circ}\text{C} \rightarrow 960\text{ }^{\circ}\text{C}$



Multiple Intratube Nanojunctions by Repeating Temp. Oscillation



Six intramolecular junctions were induced by three temperature oscillations between 950°C and 880°C during CVD. (A) shows the scheme of temperature oscillation with time; (B) is an SEM image of several parallel ultralong SWNTs grown during the temperature oscillation; (C) shows Raman RBM peak positions along a SWNT, each peak corresponds to a time period in (A).



nanotoday

www.nanotoday.com

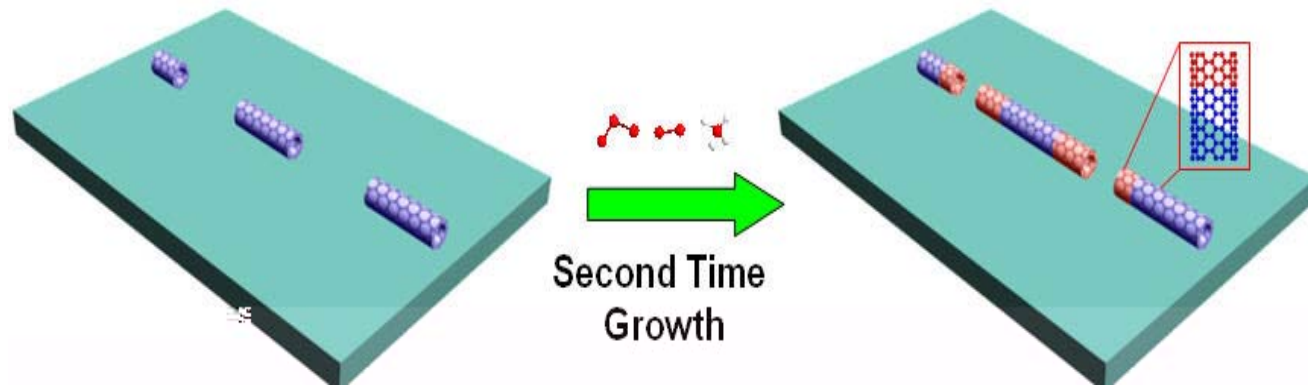
JUNE 2007 | VOL 2 | NO 3

But carbon nanotubes could, in the future, provide more than just thermal management, they could form the basis of electronic devices themselves. To realize such a goal, it will be necessary to find a means of creating intramolecular junctions in a controlled manner. Researchers from Peking University, China and Los Alamos National Laboratory claim to be able to form single-walled carbon nanotube (SWNT)

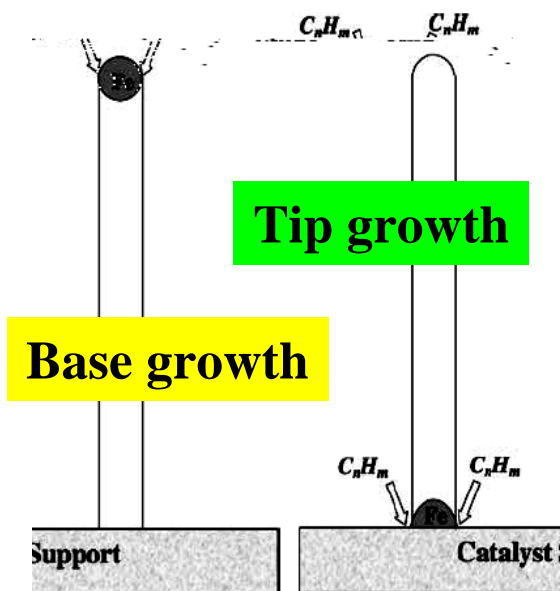
intramolecular junctions simply by varying growth temperature [Yao et al., Nat. Mater. (2007) 6, 293].

It is currently accepted that the size of the catalytic particle used to grow a nanotube determines its diameter. However, the Chinese researchers observe that the diameter of a SWNT varies with growth temperature during catalytic CVD, even with the same catalytic particle. The results show that if the growth temperature is increased from 900°C to 950°C, the diameter of the SWNT decreases by ~4%. Conversely, when the growth temperature is decreased, the SWNT diameter increases. With the change in nanotube diameter comes a change in chirality and, hence, bandgap. However, if the growth temperature is held constant, the researchers observe nanotubes of uniform diameter.

"These strategies provide a potential approach to grow SWNT intramolecular junctions at desired locations, sizes, and orientation," says Jin Zhang of Peking University. If such a simple method could reliably produce SWNT intramolecular junctions, it could be a significant step toward next-generation, carbon-nanotube-based electronic circuits and devices.



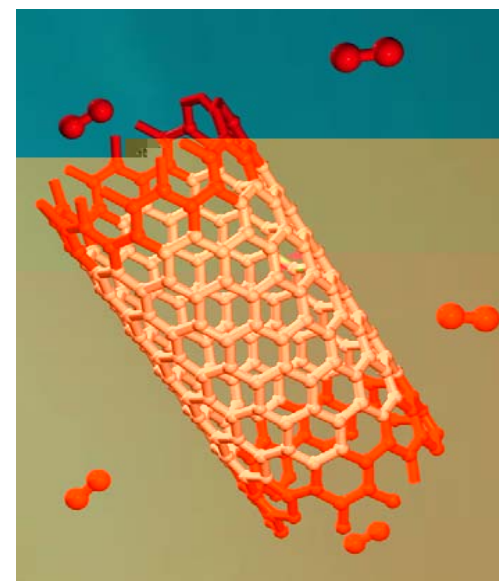
Control of chirality of SWNTs
—— Cloning growth



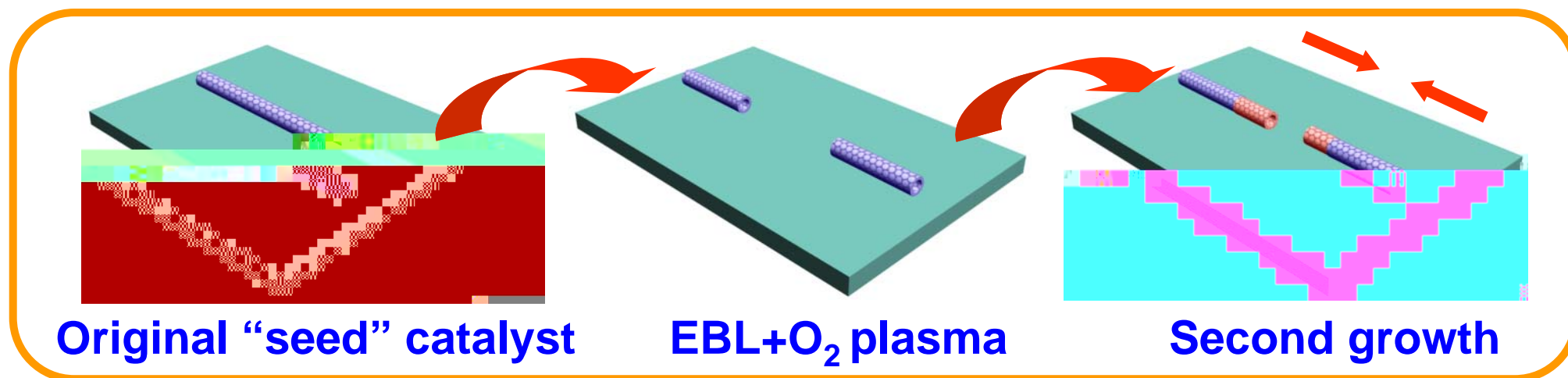
Growth catalyst:

Fe, Co, Ni, Cu, Ag, Au, Pt,
Pd, C_{60} , Si/SiC, Ge/SiC,
 SiO_2 , TiO_2 , Al_2O_3 , Rare
earth oxides

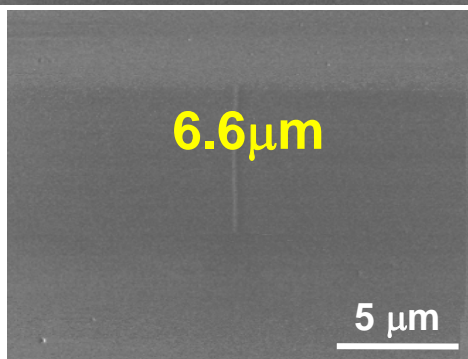
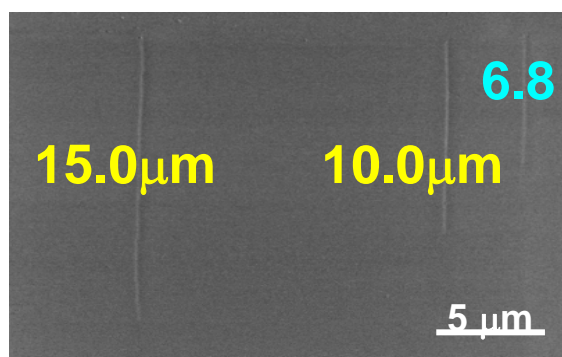
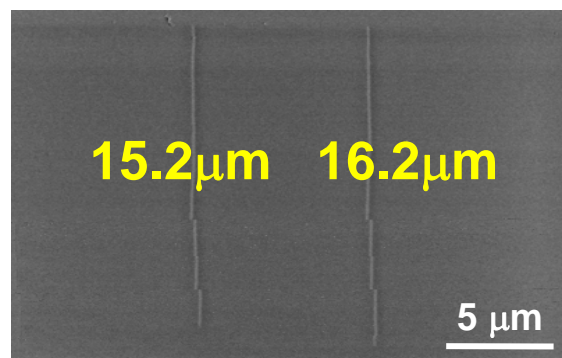
Why not the chemically-
active open-cap nanotube ?



Open-end growth mode



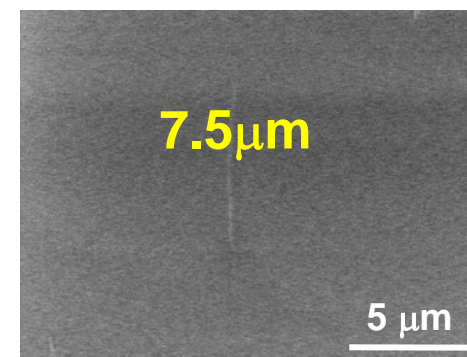
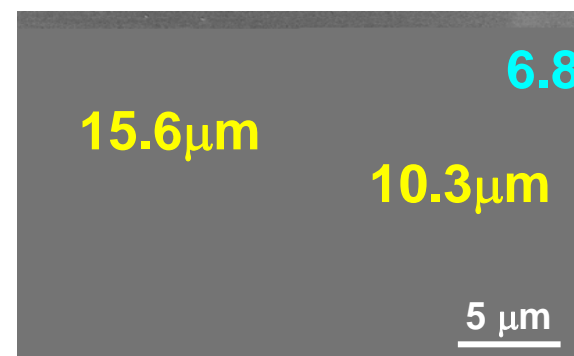
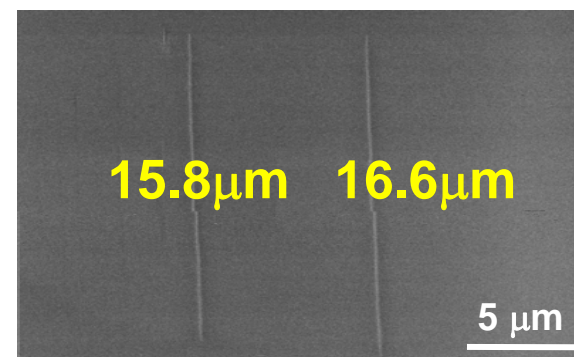
Cloning SWNTs on Quartz Surface



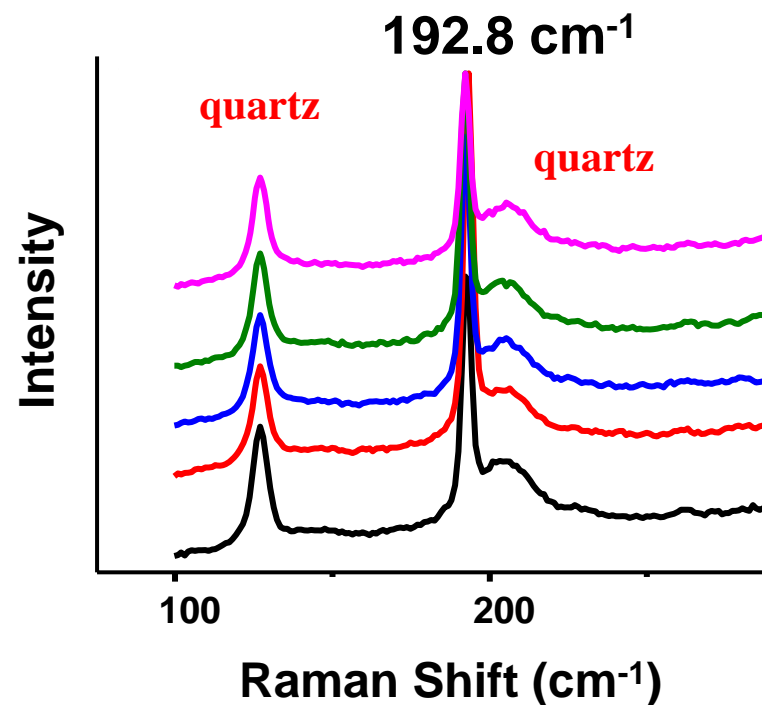
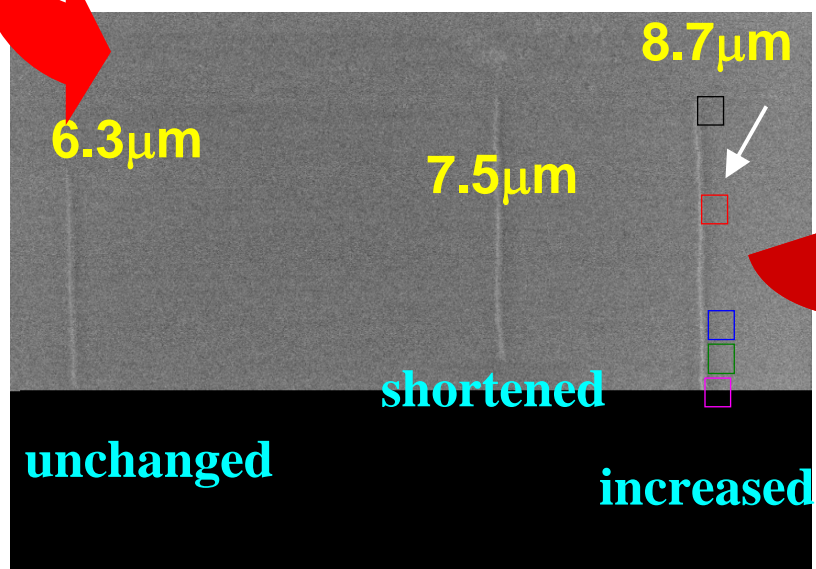
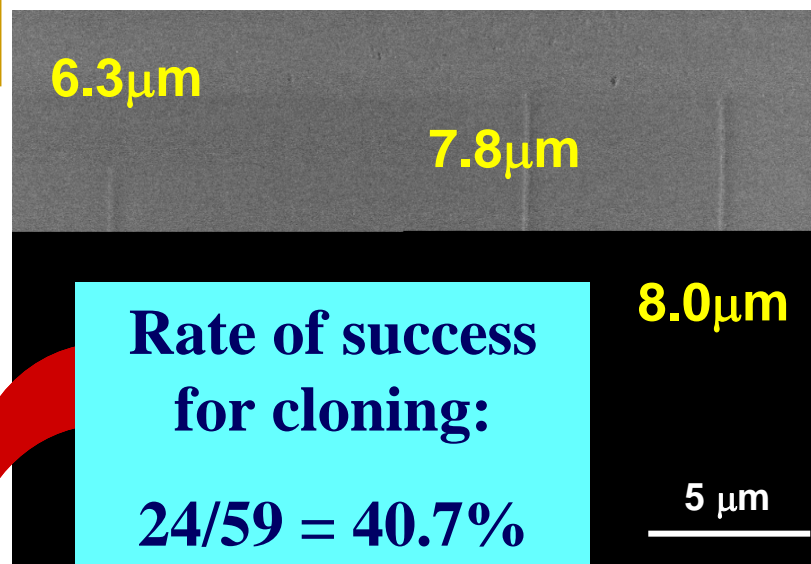
Before growth

Tube length
increase:

0 μm – 1 μm

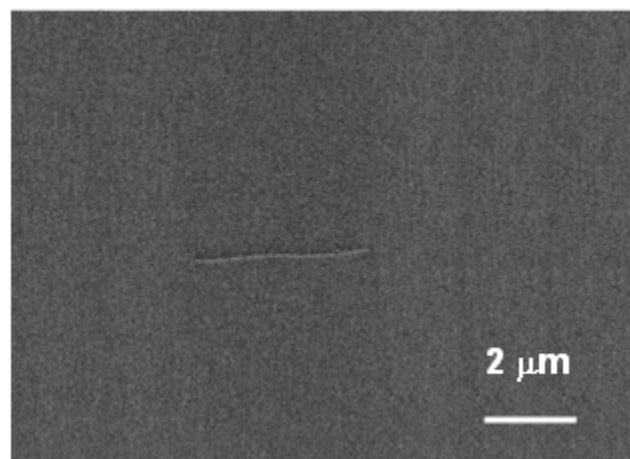
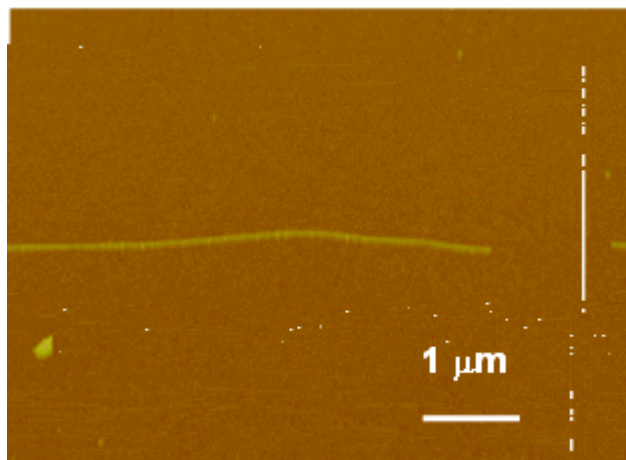


After growth



**Tube diameter & chirality
keep unchanged after
cloning growth**

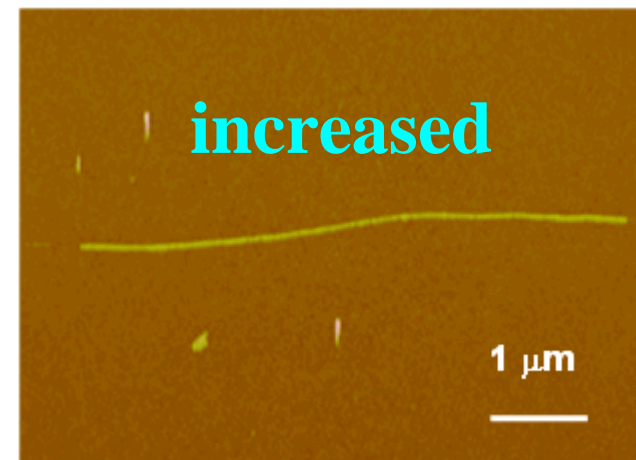
Cloning SWNTs on SiO₂/Si Surface



Before growth

Tube length
increase:

0 μm – 4.6 μm

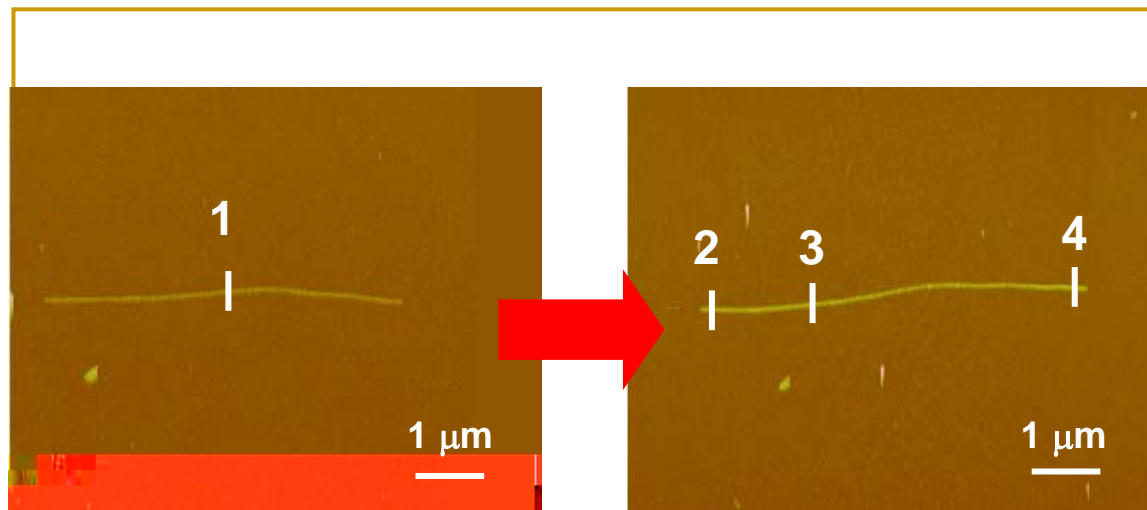


After growth

Rate of success
for cloning:

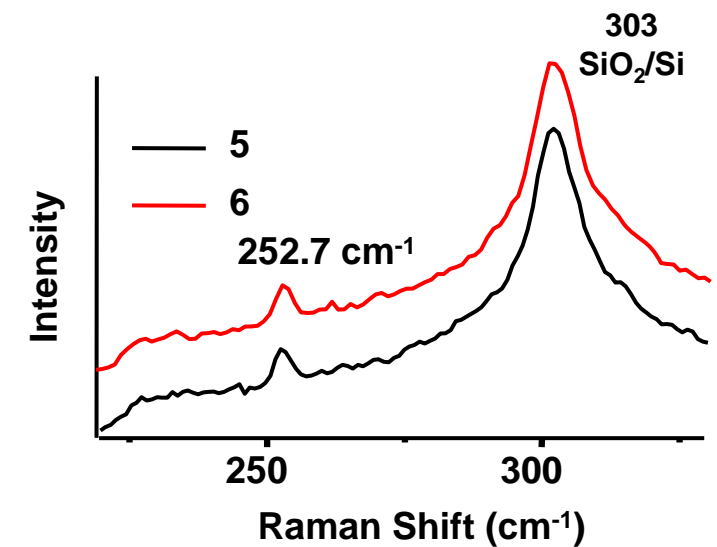
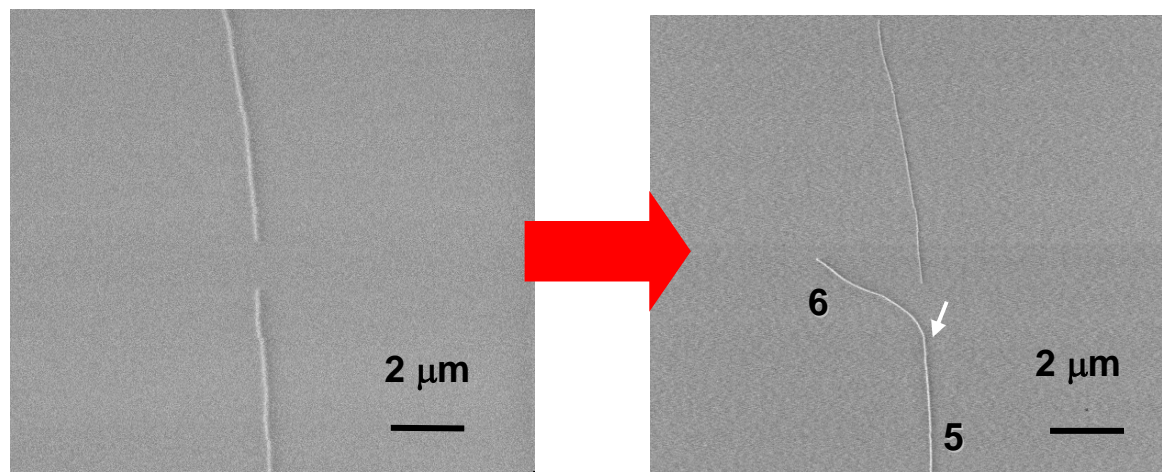
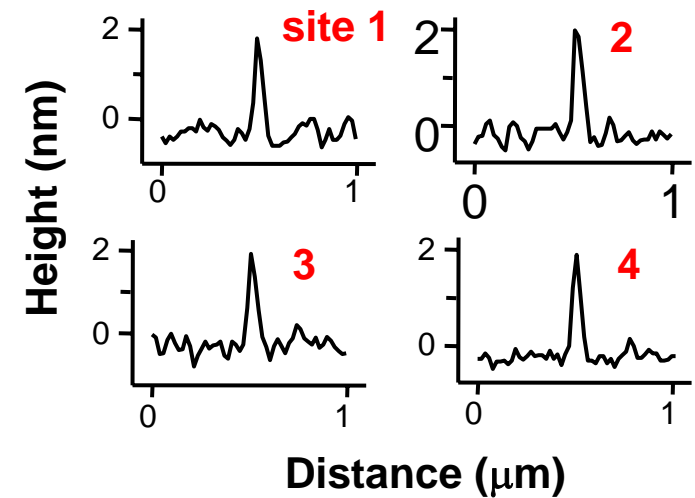
$56/600 = 9.3\%$

- $CH_4/C_2H_4/H_2=100/5/300$
- Growth $T=975^\circ C$ for 15min



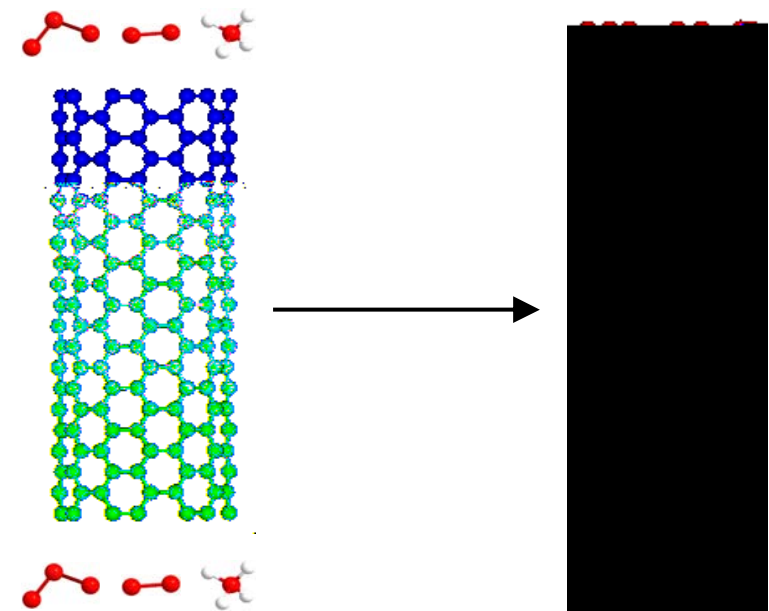
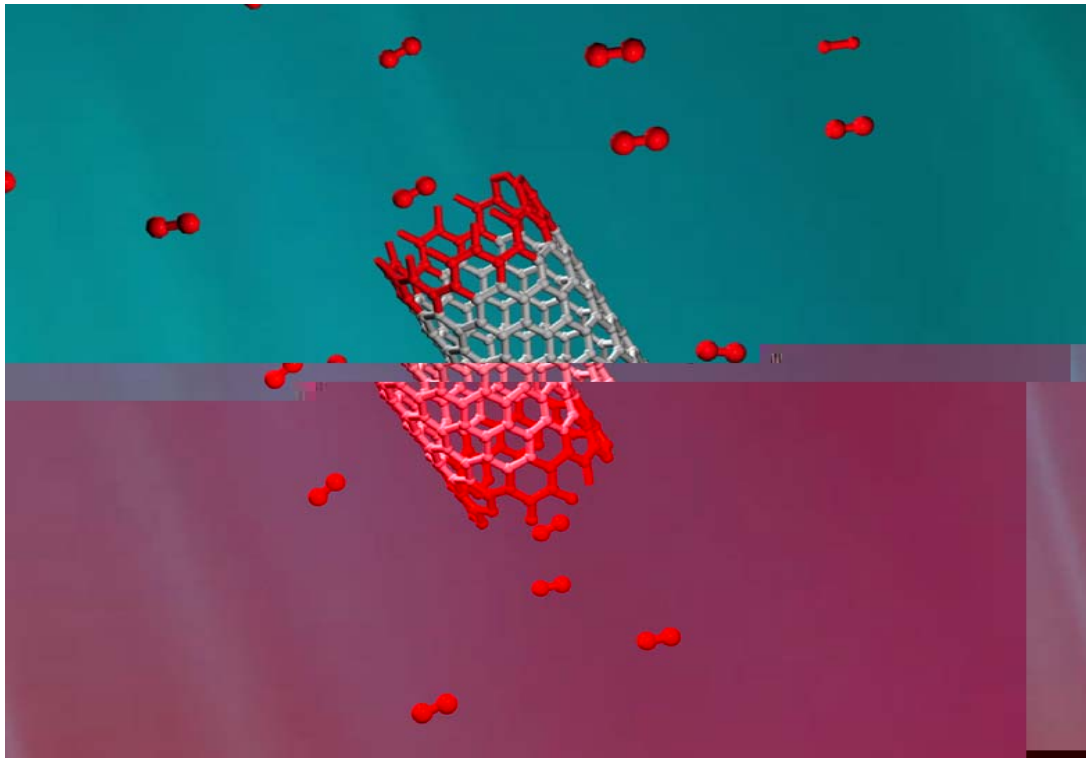
Before growth

After growth



AFM and Raman evidence for the diameter & chirality maintenance

Open-End Growth Mechanism



“VS” growth model ?

featured highlight

Carbon nanotubes: Perfect clones

Published online 29 June 2009

Although methods for producing single-walled carbon nanotubes have advanced considerably in recent years, growing nanotubes with specific structures remains difficult. Nanotubes can be semiconducting or metallic in character

According to the cloning path for carbon nanotubes, hexagons within the structure, and controlling this behavior is crucial for future nanoscale electronic applications.

Now, Jin Zhang and colleagues¹ from Peking University in China have devised a novel method for the growth of single-walled carbon nanotubes that allows precise control of the nanotube properties. Using seeds cut from existing nanotubes, the researchers grew new extensions that retained the original structure — perfect clones of the parent segments (Fig. 1).

Working with ultralong single-walled carbon nanotubes grown by chemical vapor deposition, Zhang's group first removed the nanotubes with

lithography and oxygen plasma ion etching, the researchers prepared short, open-ended segments of the nanotubes as seeds. These small sections were then heated to 700 °C to

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ng, J.^{1*} & Liu, Z.¹ "Cloning" of single-walled carbon nanotubes via open-end et. 9, 1673–1677 (2009). | [article](#) |

y for Molecular Sciences (BNLMS), Key Laboratory for the Physics and Chemistry Laboratory for Structural Chemistry of Unstable and Stable Species, College of Engineering, Peking University, Beijing 100871, China

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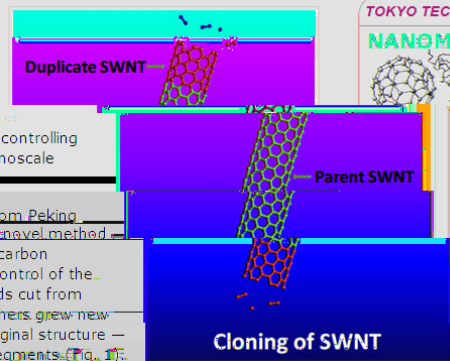


Fig. 1: Using open-end single-walled nanotubes (SWNTs) as seeds for a cloning growth process may solve current structural control issues.

lithography and oxygen plasma ion etching, the researchers prepared short, open-ended segments of the nanotubes as seeds. These small sections were then heated to 700 °C to

over the seed segments. Using atomic force and scanning electron microscopy, the team observed new growth of several micrometers in length extending from the seed segments. Raman spectroscopy confirmed that the newly grown nanotube segment retained the original seed structure.

According to Zhang, growing ultralong nanotubes requires a mixture of acetylene and methane gases. "We used acetylene to facilitate decomposition of methane gas into carbon radicals. If we didn't use acetylene, the growth temperature was below 945 °C, no amplified growth occurred,

Instead of requiring an external additive, the growth mechanism discovered the open ends of the nanotubes themselves as catalysts. The seed segments retained the nanotube structure, and any new growth via carbon radicals retains

The researchers reported that, on a quartz substrate, over 40% of the produced new cloned segments. "We think if the open-ended segments

Zhang and colleagues are now undertaking large-scale growth trials of carbon nanotube clones.

[View the Insight online...](#)

[Reference](#)

[click here](#)

1. Yao, Y.,¹ Feng, C.,¹ Zhang, Z.¹ "Cloning" of single-walled carbon nanotubes via open-end growth mechanism. *Nano*

Author affiliation

1. Beijing National Laboratory of Nanodevices, State Key Laboratory of Chemistry and Molecular Engineering, Peking University, Beijing 100871, China
 *E-mail: jinzhang@pku.edu.cn

www.afm.university.org
 www.afm.university.org

Agilent

Printer-friendly

to desired use the ing

synthesize any

be continual outh mechanism," Jin Zhang tells Nanowerk. "The new 'cloned' SWCNTs and

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 Single-wall carbon nanotubes and Other Derivatives

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Posted: April 3, 2009

'Cloning' might solve the chirality control issue of nanotube production

(Nanowerk Spotlight) Notwithstanding the tremendous amount of research that has gone into the field of carbon nanotubes, the synthesis of single-walled carbon nanotubes (SWCNTs) with controlled chirality is still a challenge. Carbon nanotubes have a wide range of electronic properties, from semiconducting to metallic, depending on their diameter, length, chirality and electronic properties, all packed together in bundles, and often blended with some amount of amorphous carbon. The separation of nanotubes according to their properties remains a technical challenge. Especially SWCNT sorting is a challenge because composition and chemical properties of SWCNTs of different types are very similar, making conventional separation techniques inefficient.

Using the concept of cloning, scientists in China have discovered an effective method to produce special (n, m) indices SWCNTs.

"We found that an open-end single-walled carbon nanotube, which served as seed, could grow via an open-end growth mechanism," Jin Zhang tells Nanowerk. "The new 'cloned' SWCNTs and

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Nanotechnology fabrication of single-walled carbon nanotubes with controlled chirality

Posted: Aug 24th, 2009

Carbon nanotubes: Perfect clones

Posted: Jun 29th, 2009

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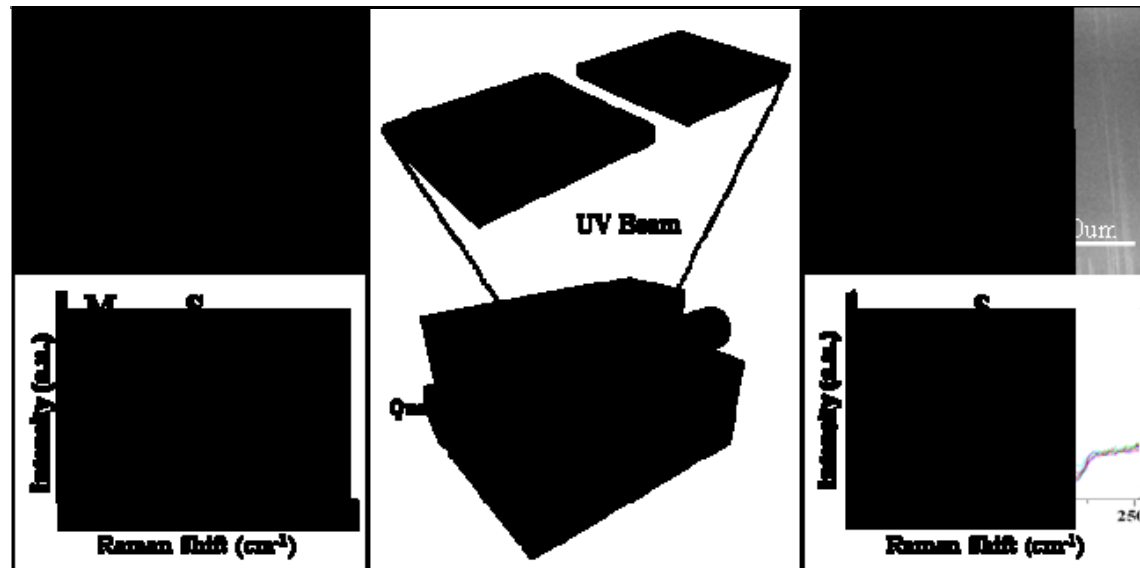
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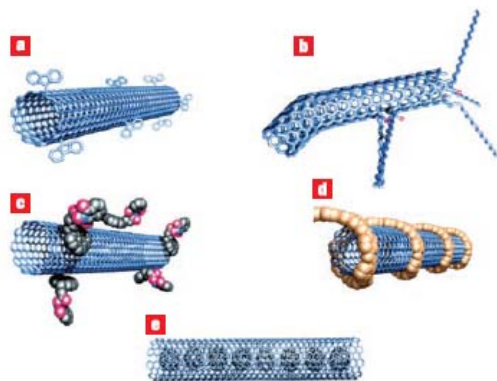


Control of metallic-/semiconducting- of SWNTs

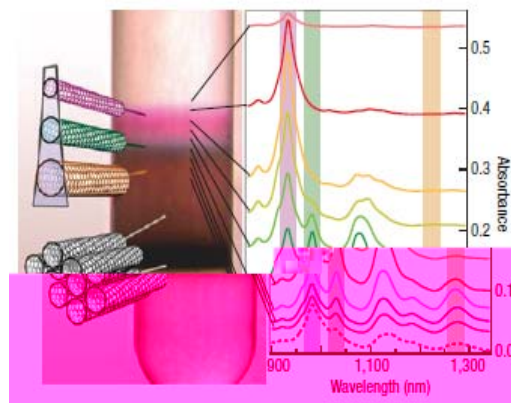
—— UV irradiation assistance CVD growth

Separation of s-SWNTs and m-SWNTs after growth

Functionalization



density gradient ultracentrifugation

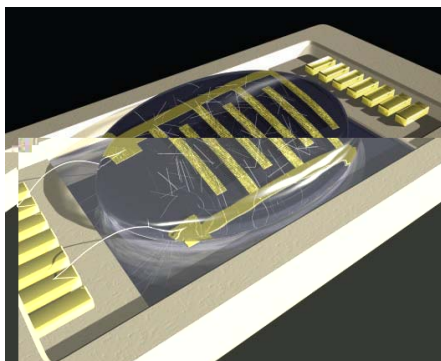


Ion-exchange chromatography



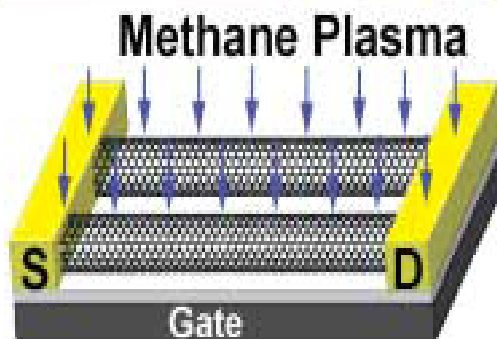
A. Hirsch, Angew. Chem. Int. Ed. , 2002 M. S. Arnold, Nature Nanotech. , 2006 M. Zheng, Nature Mater., 2003

alternating current dielectrophoresis



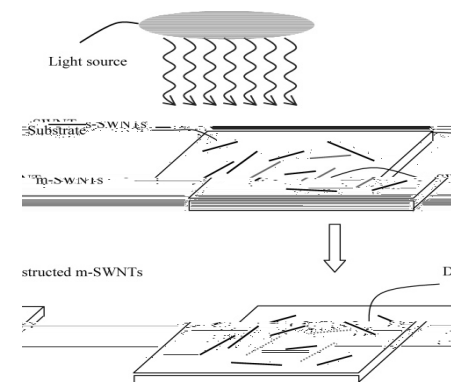
R. Krupke. Science, 2003

gas-phase plasma hydrocarbonation



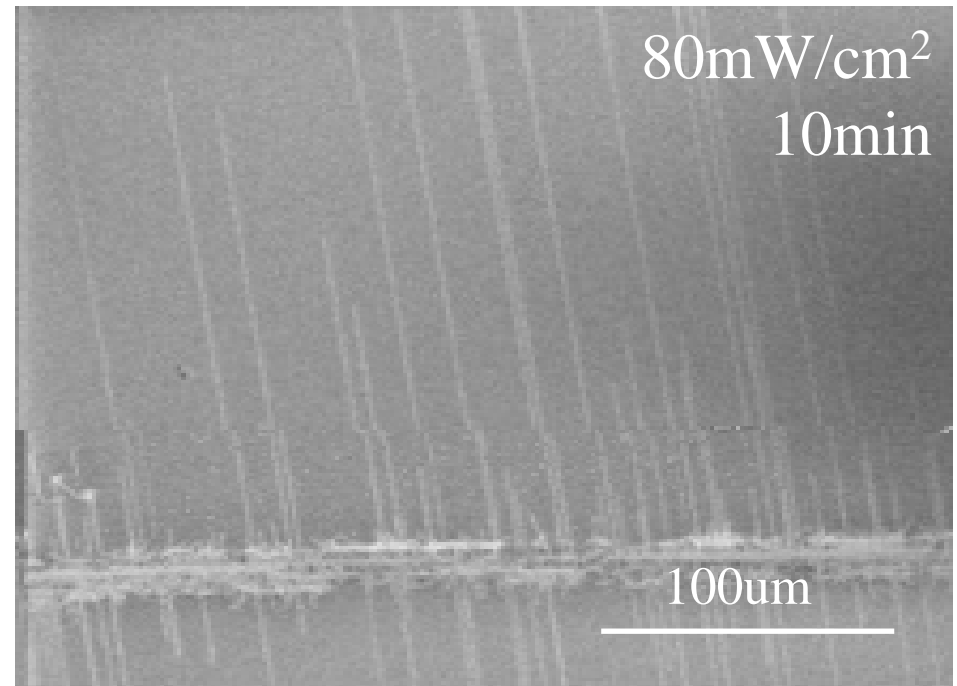
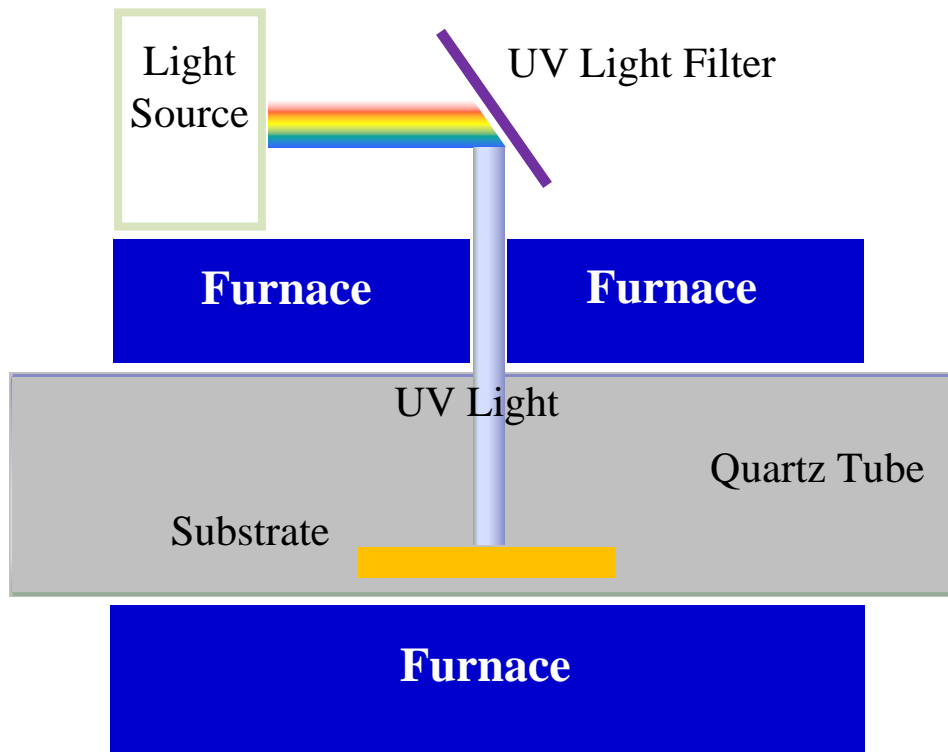
H. J. Dai. Science, 2006

Laser Irradiation

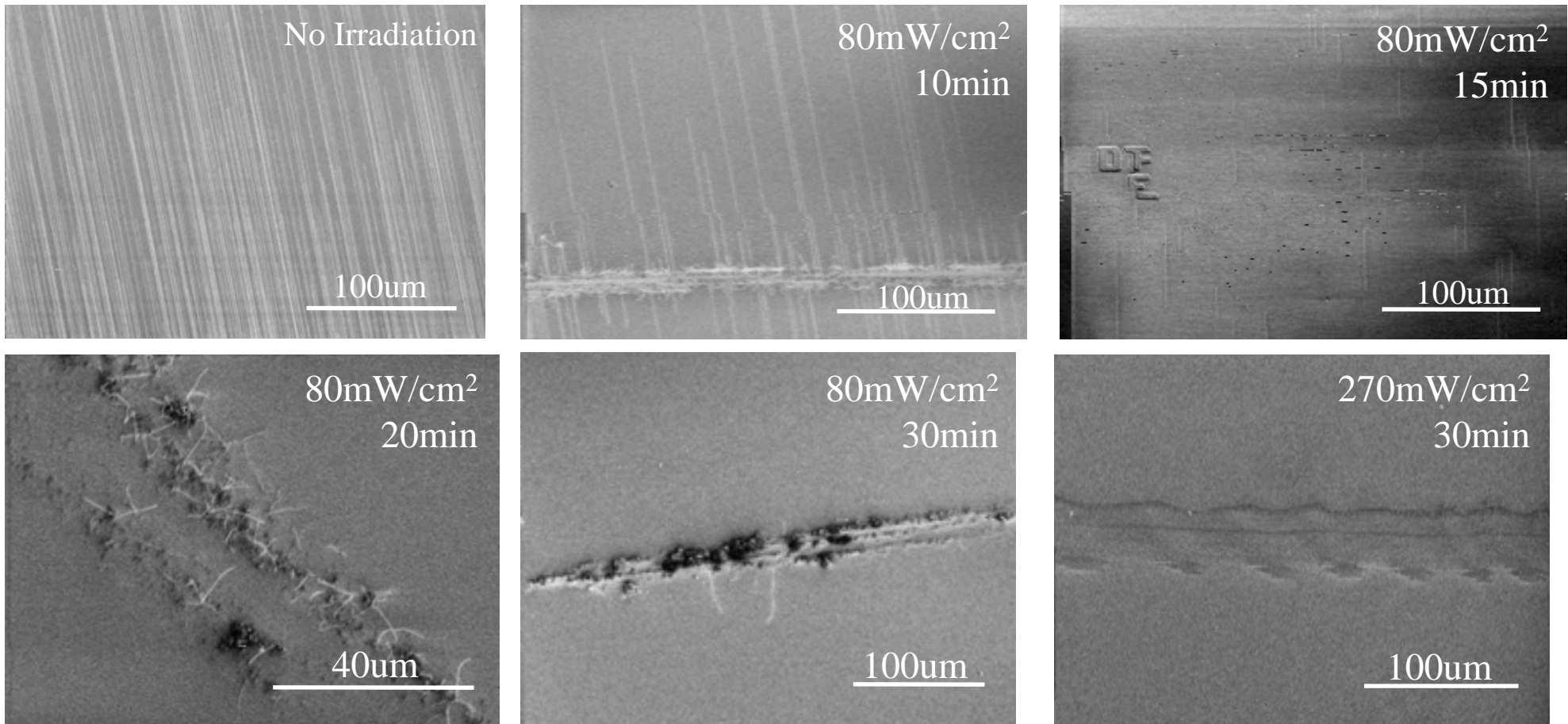


H. J. Huang, J. Phys. Chem. B, 2006

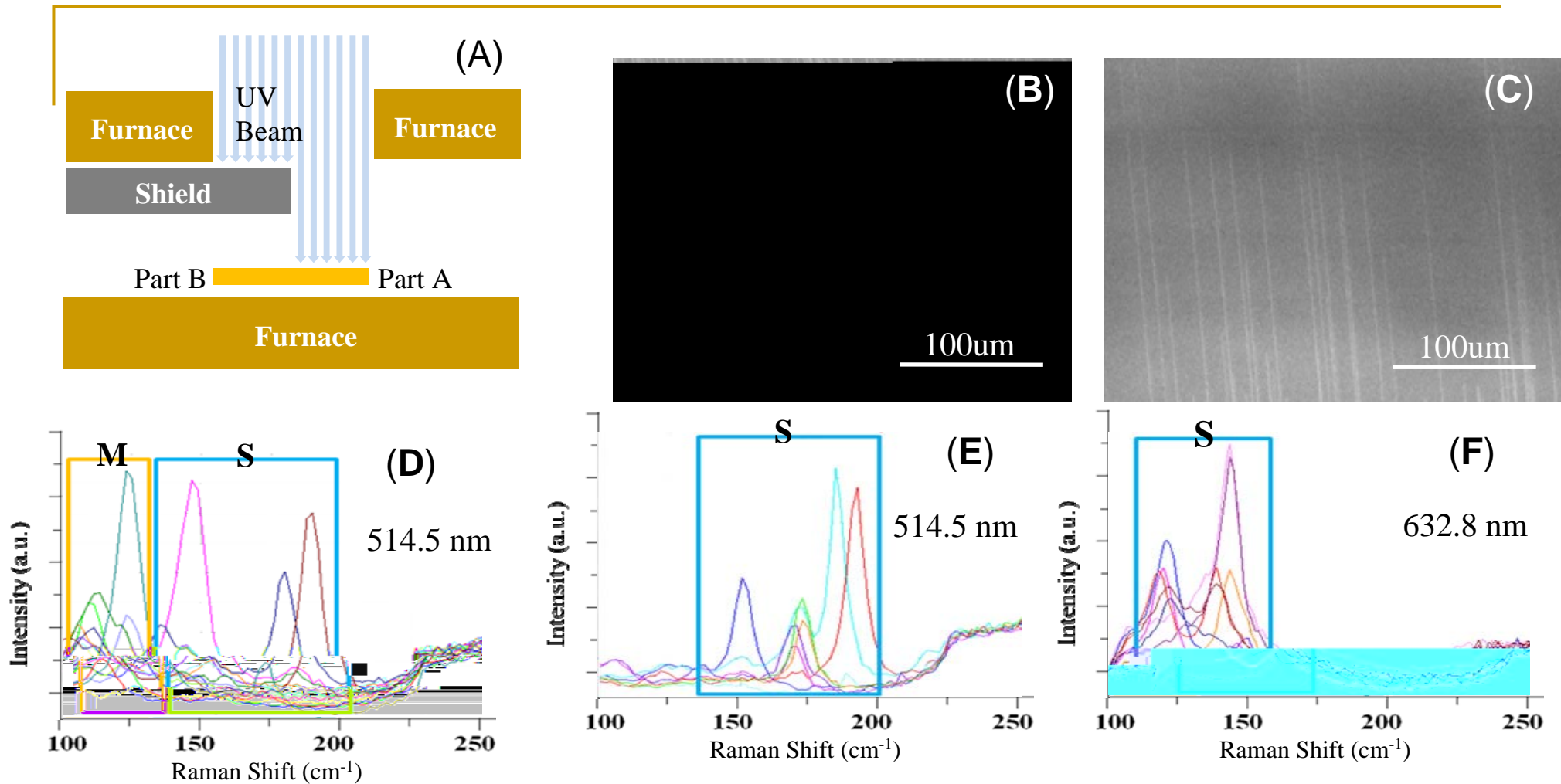
Sketch map of the home-made chemical vapor deposition system



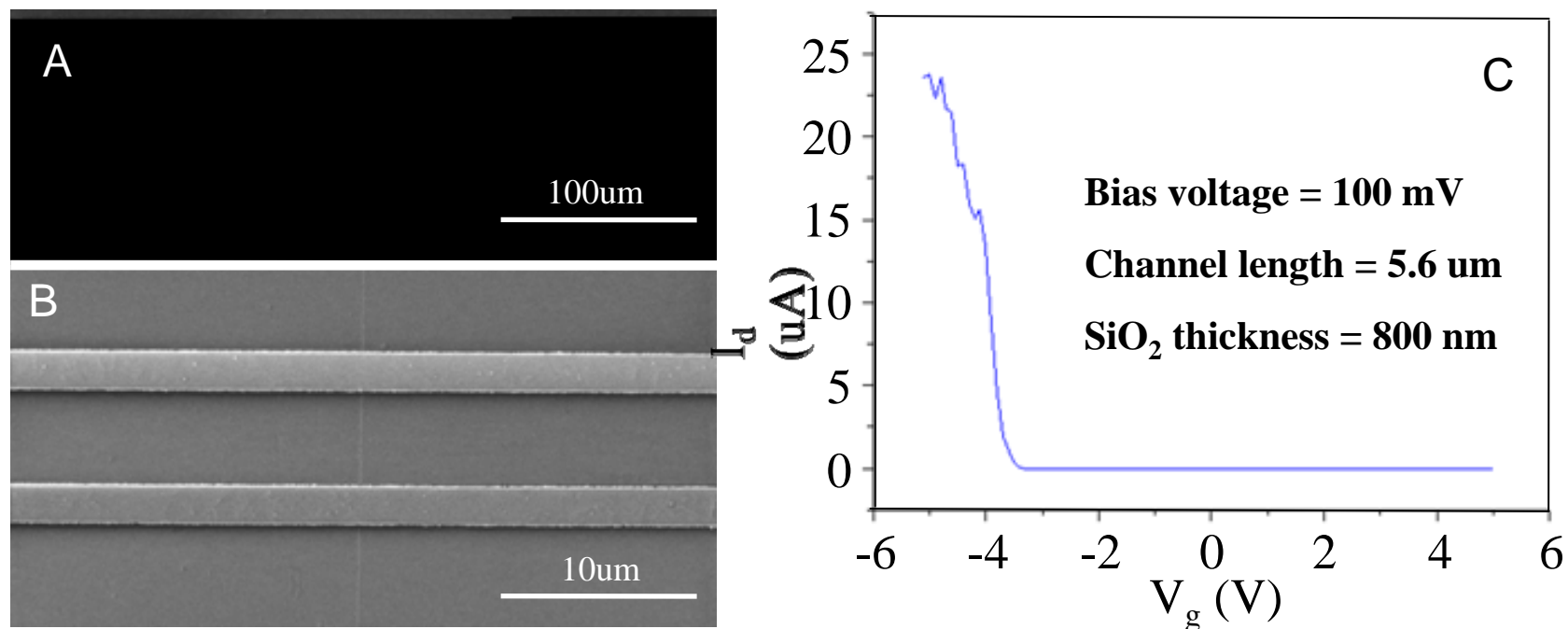
SEM images of SWNTs arrays under different irradiation time



When UV beam acted on the substrate, the density of the SWNT array decreased obviously. From above, the shorter the irradiation time, the longer and denser the SWNTs were. If we continued increasing the irradiation time or the irradiation intensity, SWNTs would become shorter and shorter, and disappeared eventually

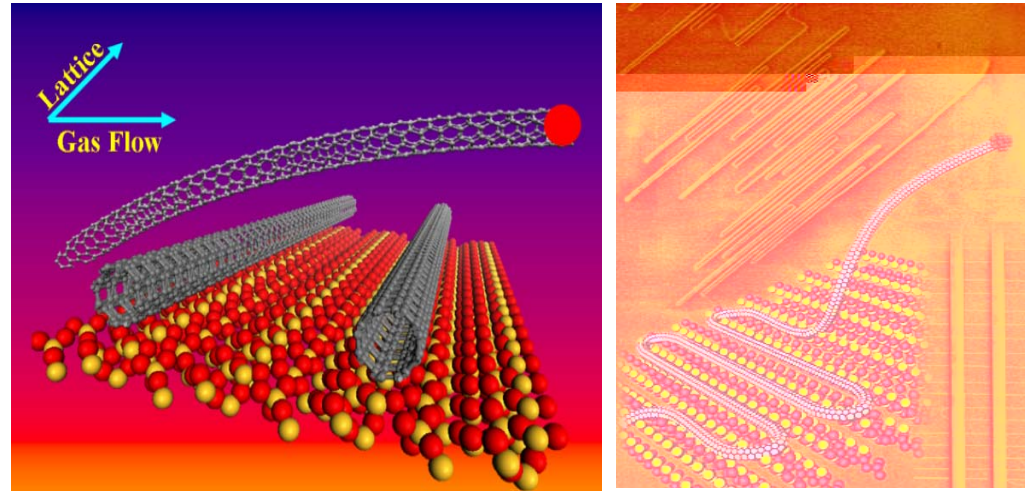


(A) Sketch map of the comparison experiment for SWNT growth with and without UV irradiation. (B)/(C) SEM image of the growth result without/with UV irradiation. (D) Raman spectrum for part B with 514.5 nm excitation. (E)/(F) Raman spectrum for part A with 514.5/632.8 nm excitation. The metallic SWNTs signals were collected in the yellow rectangle while the semiconducting SWNTs signals were collected in the blue rectangle separately for all the three spectra.



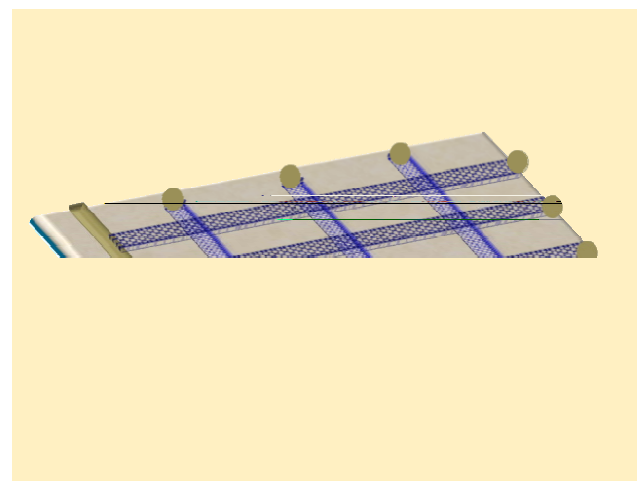
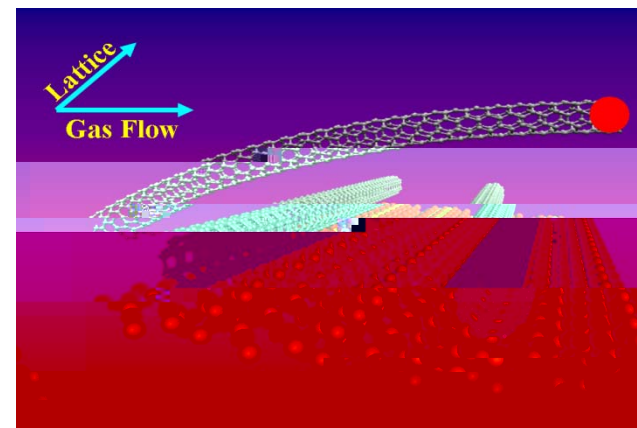
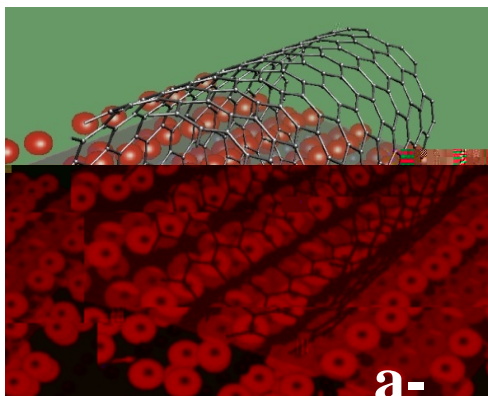
The Raman spectra demonstrated an amazing result that almost 100% SWNTs were semiconducting ones. To further confirm the percentage of the semiconducting SWNTs in this sample, we performed electrical measurement in the single-tube field effect transistors (FET) form. Figure A and B were the low and high magnified SEM images of the single-tube FET structure. Fig. C was the typical I-V curve of a semiconducting SWNT with the on/off ratio over 10^4 . The test parameters of the electrical measurement were inserted in Fig. C. The bias voltage was 100 mV, the channel length was 5.6 μm, and the SiO₂ thickness was 800 nm. The electrical measurement data showed that 21 out of 22 SWNTs were semiconducting ones.

J. Zhang et al., J. Am. Chem. Soc., 2009, In press

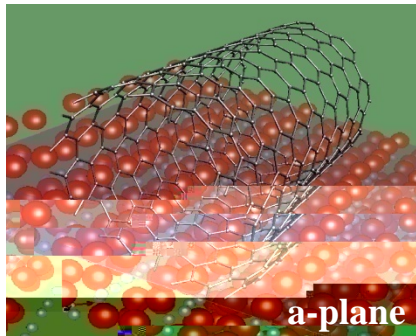


Control of local conformation and architectures

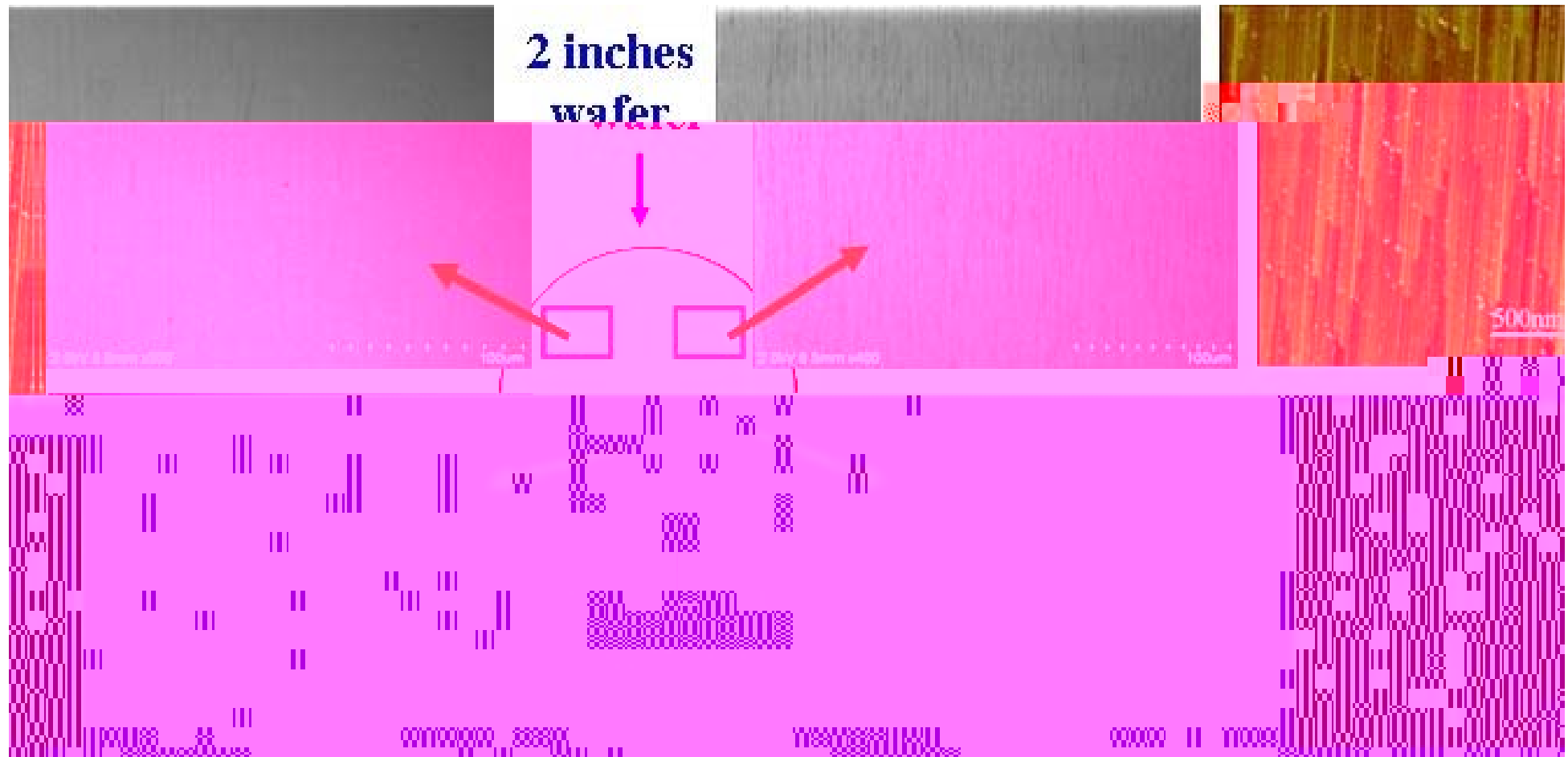
- Cooperative growth of floating and lattice oriented growth modes



High Density SWNTs Array on Surface

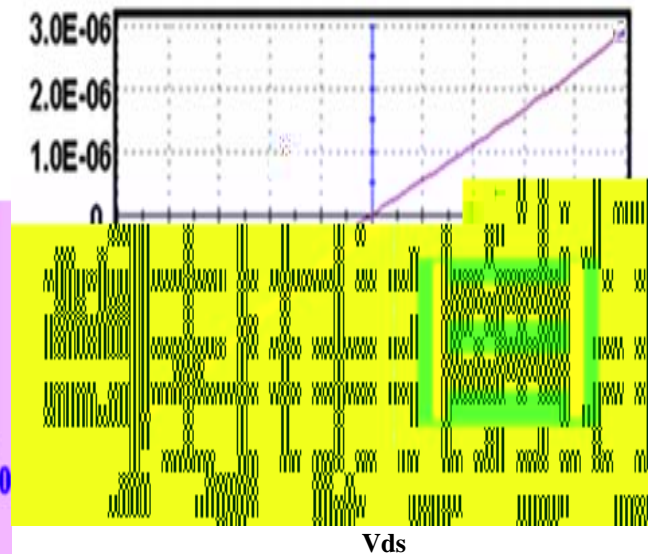
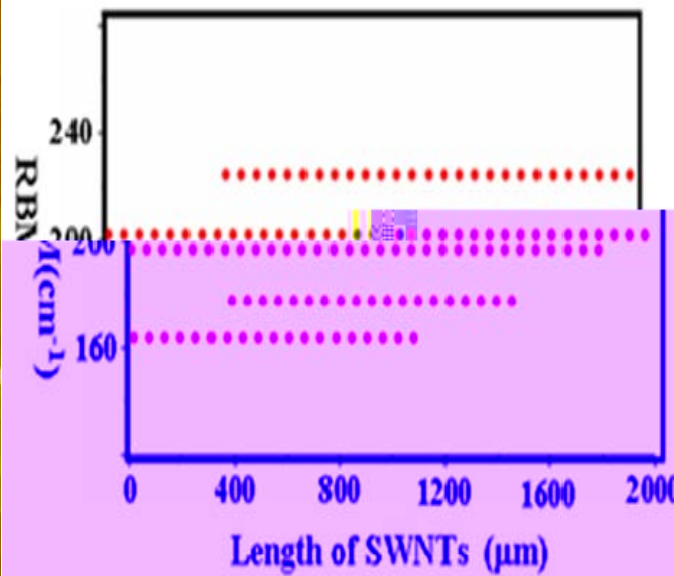
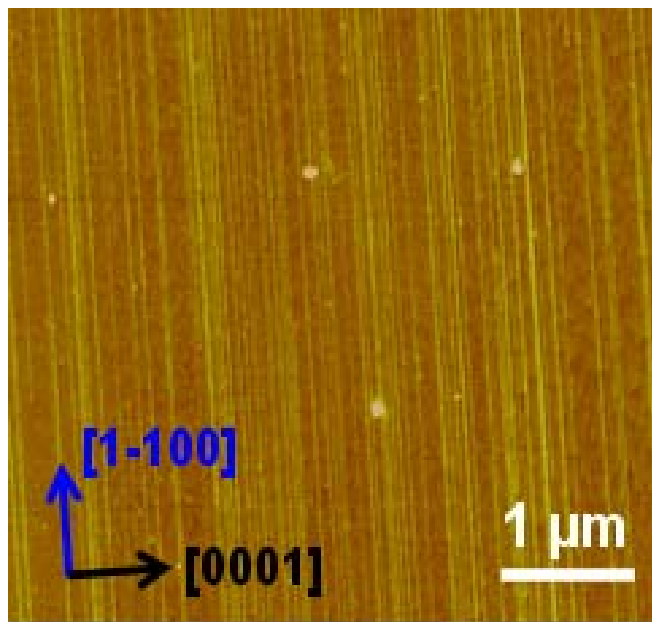
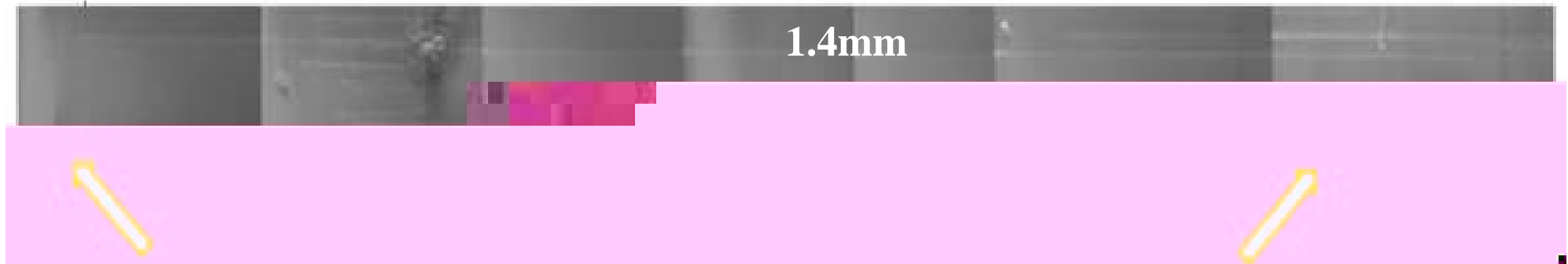


High Density SWNTs Arrays on 2 Inches Wafer

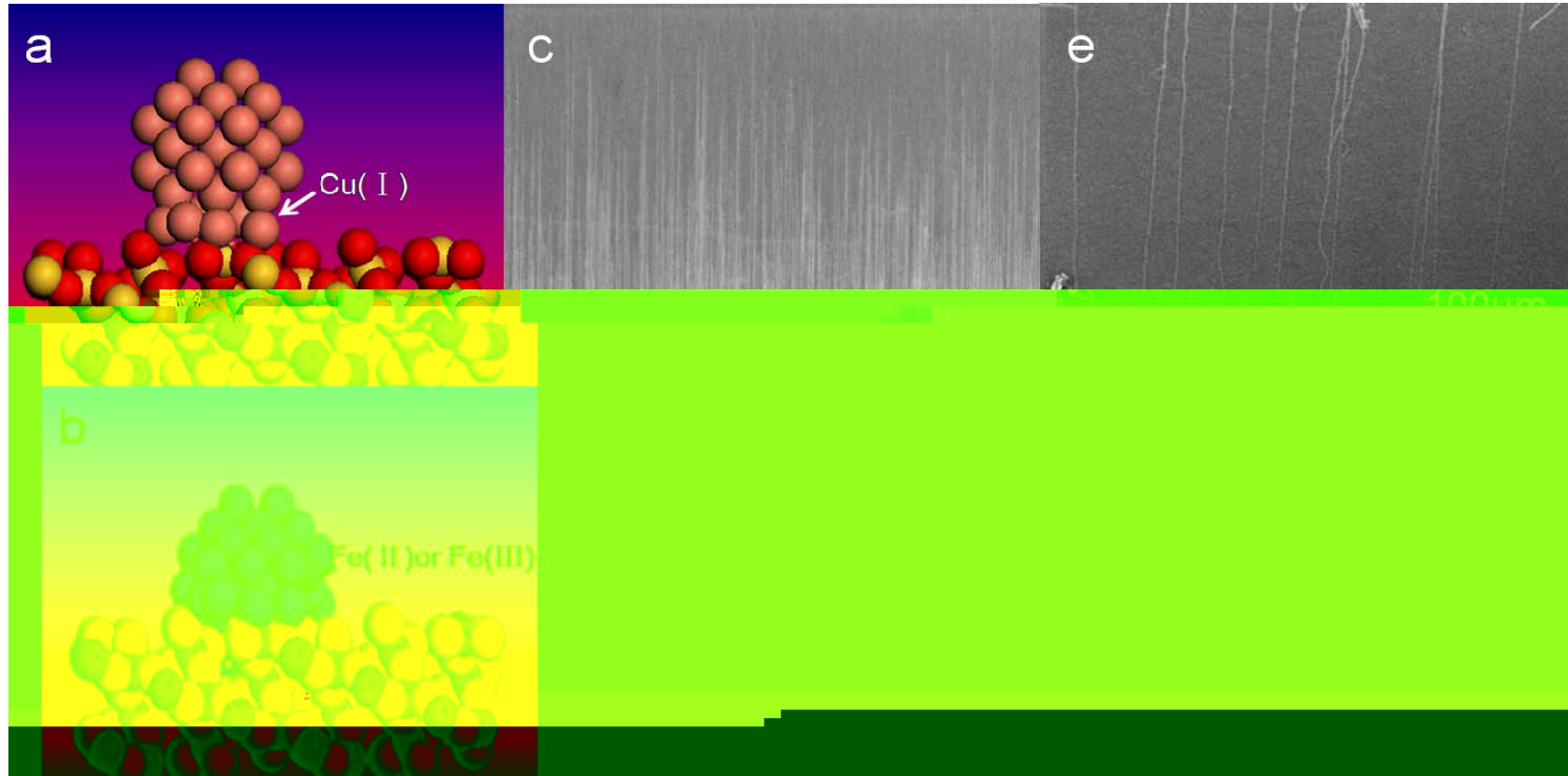


The Density is about 35- 40 tubes per micrometer

The length of the High Density SWNTs



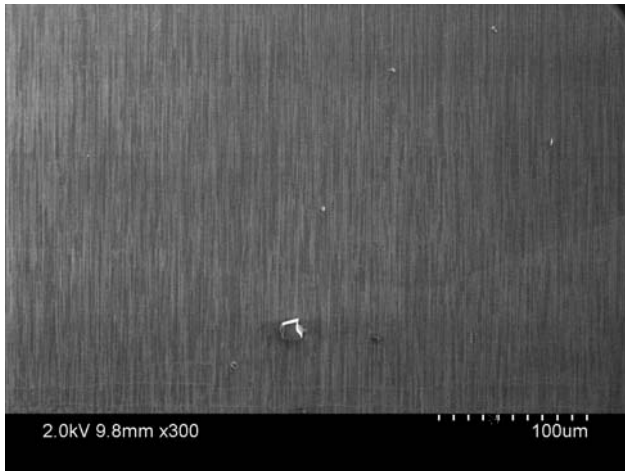
Interaction between Cu, Fe Catalysts and Quartz Surface



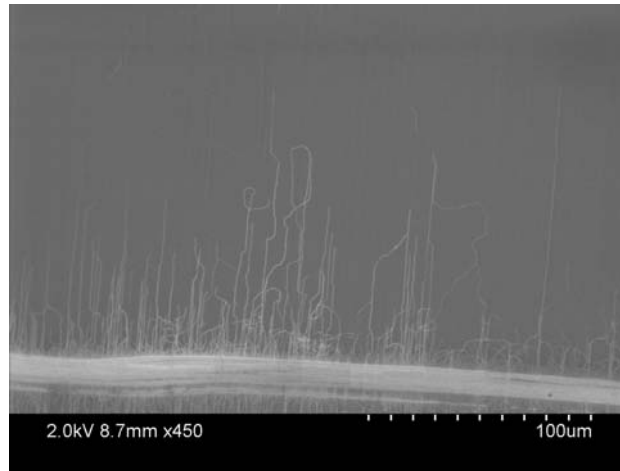
a) and b) illustrate the interaction between Cu, Fe nanoparticles and surface of quartz, the red balls represent oxygen atoms. c) and d) High-magnification SEM image of the lattice assisted SWNTs catalyzed by Cu and Fe. e) and f) Results of gas flow directed growth of carbon nanotubes where Cu and Fe are used as catalysts respectively.

The Effect of Temperature on the Growth of SWNTs on Quartz Surface

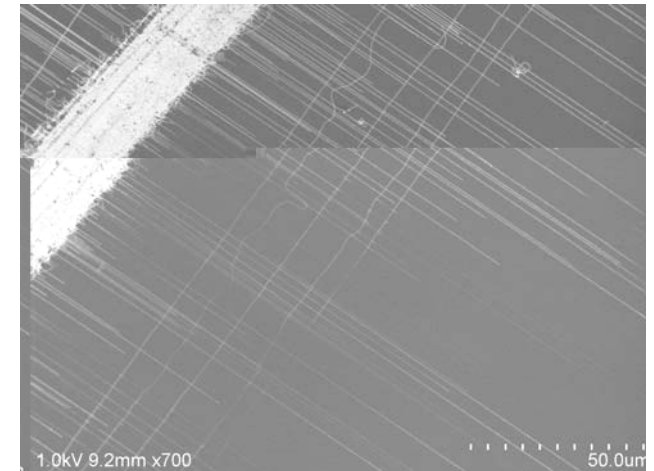
900°C



970°C



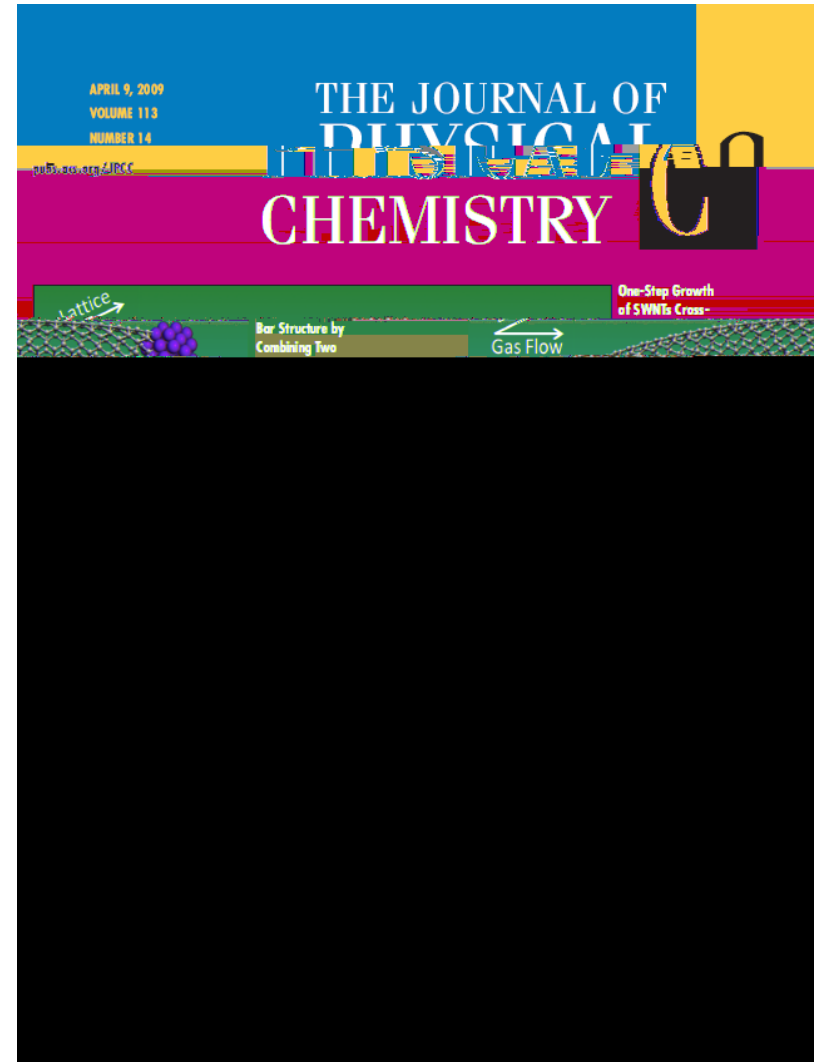
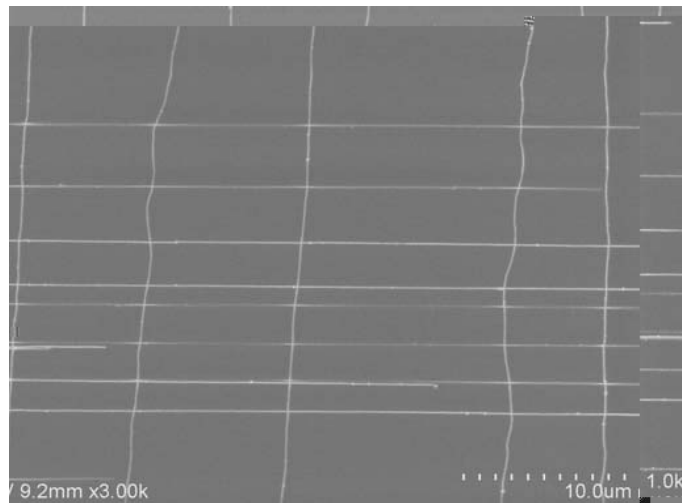
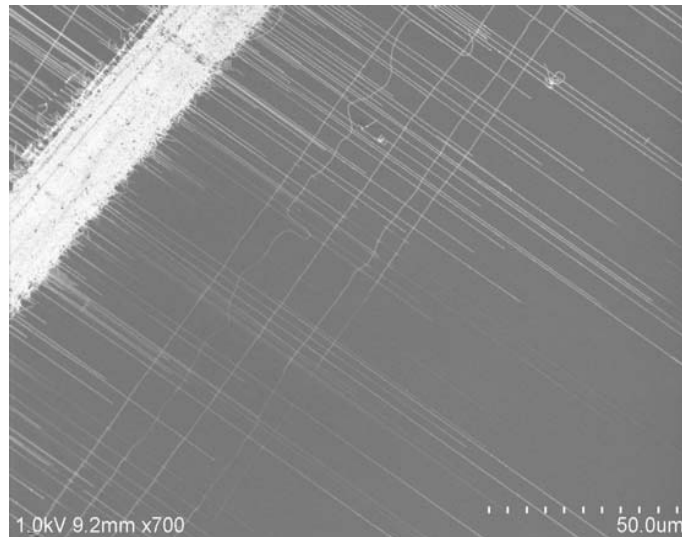
930°C



1. low temperature(900): high density of lattice directed carbon nanotubes, few gas flow directed carbon nanotubes
2. optimized temperature(930-950): the density of lattice directed carbon nanotubes is acceptable and it is possible for the growth of gas flow directed carbon nanotubes
3. high temperature(970): lattice will be damaged by such high temperature

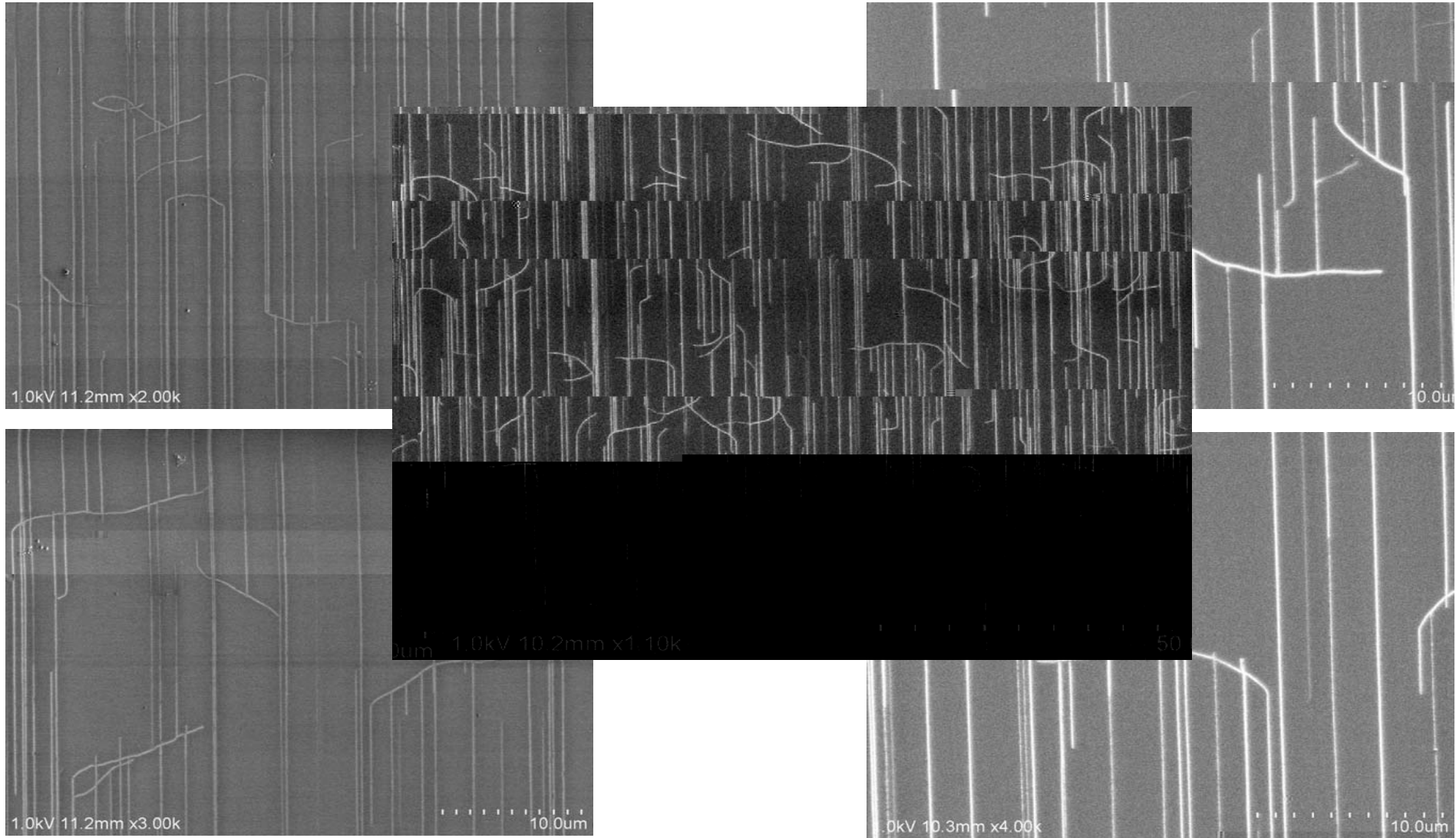
SWNTs Crossbar and Its Potential Application

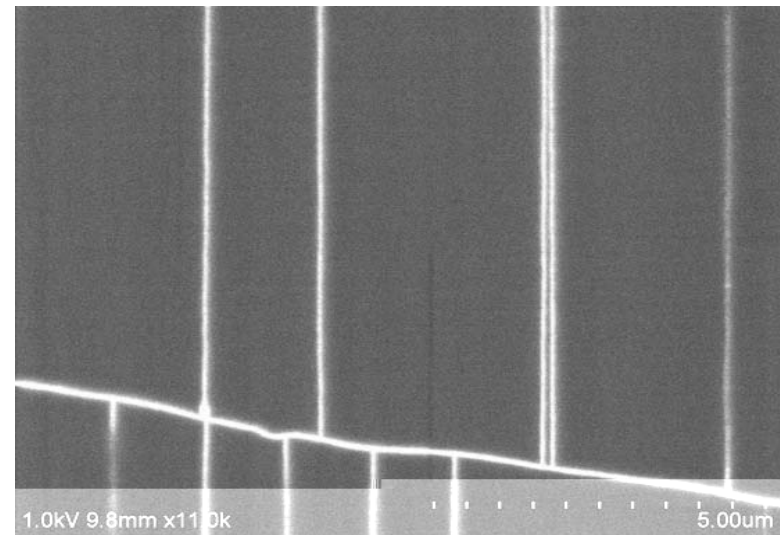
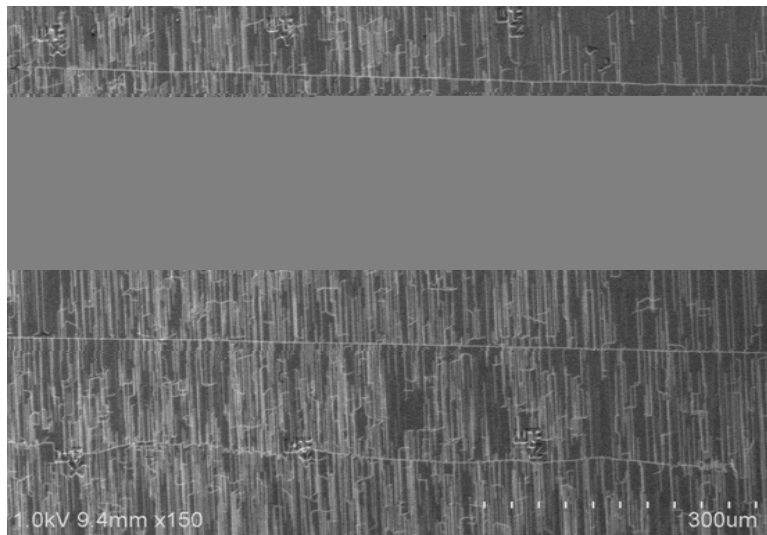
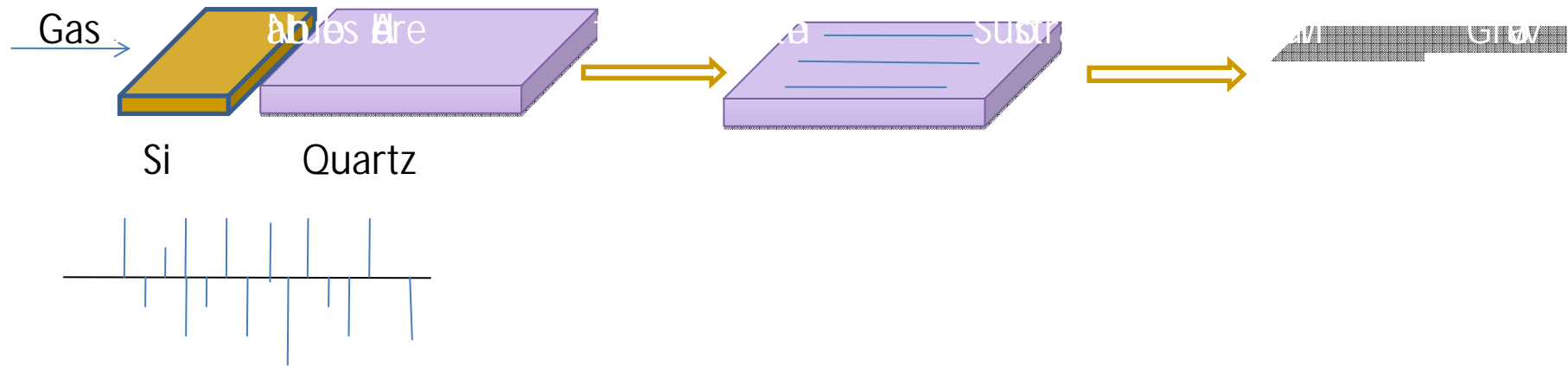
Low temperature favors for lattice oriented growth mode and high T for gas flow directed growth mode. With a moderate 930-950°C, crossbar can be grown.



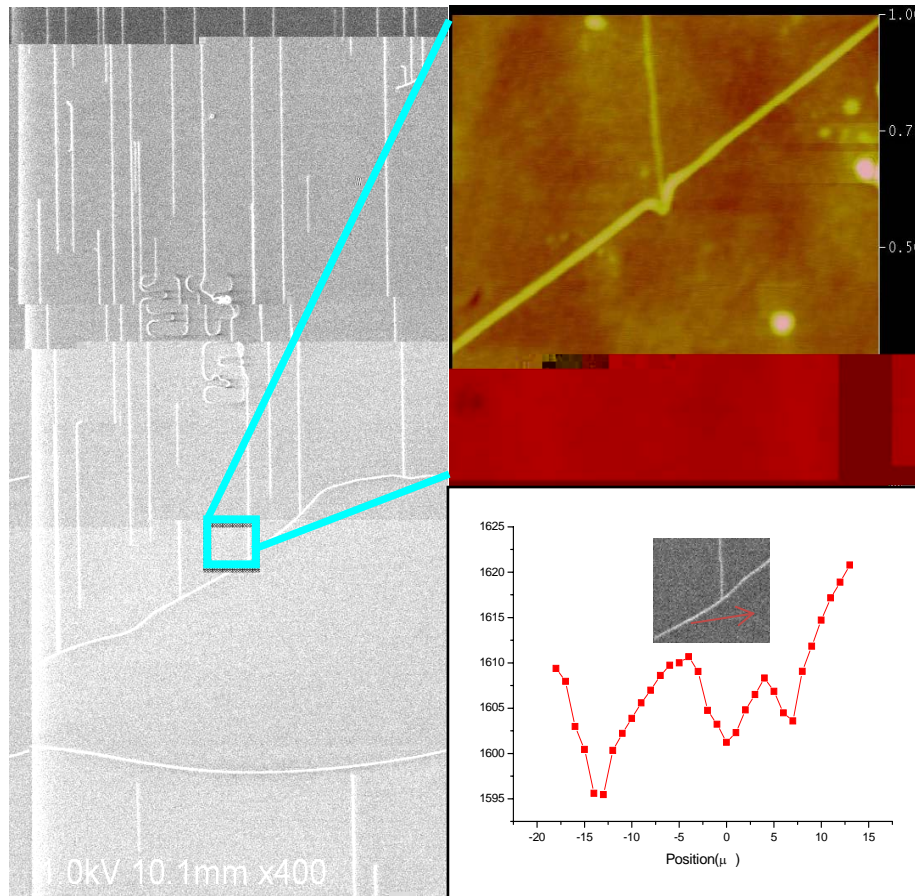
J. Zhang et. al., J. Phys. Chem. C. 2009, 113, 5341-5344 (cover)

The Phenomenon of the Experiment

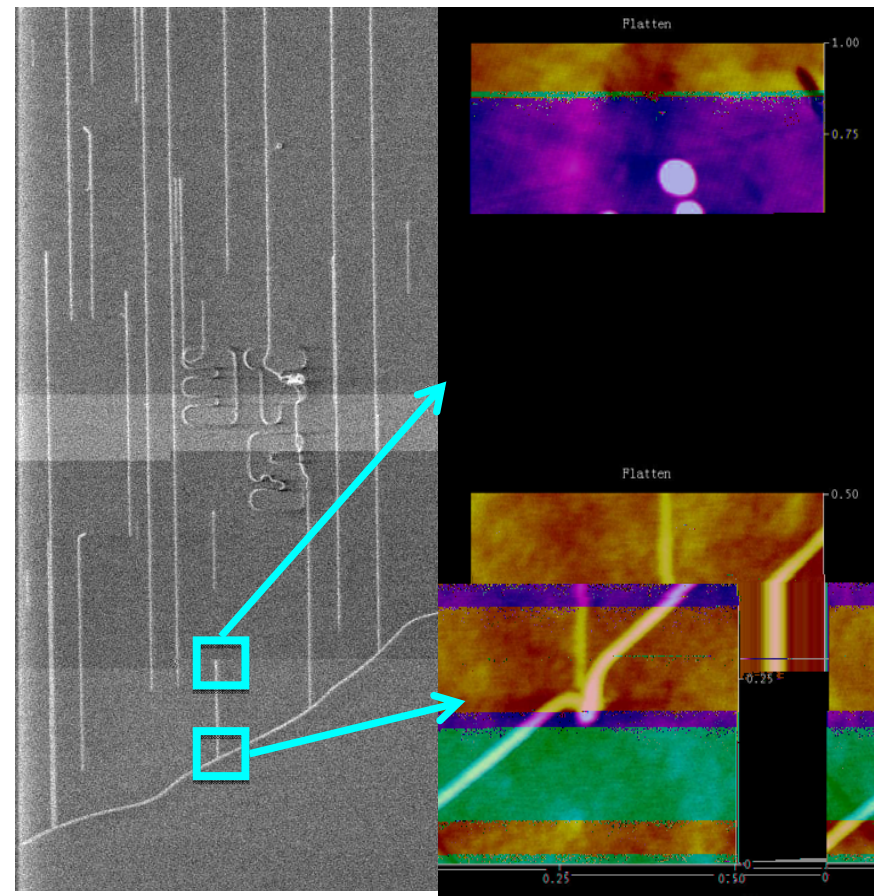




SEM AFM & Raman Results

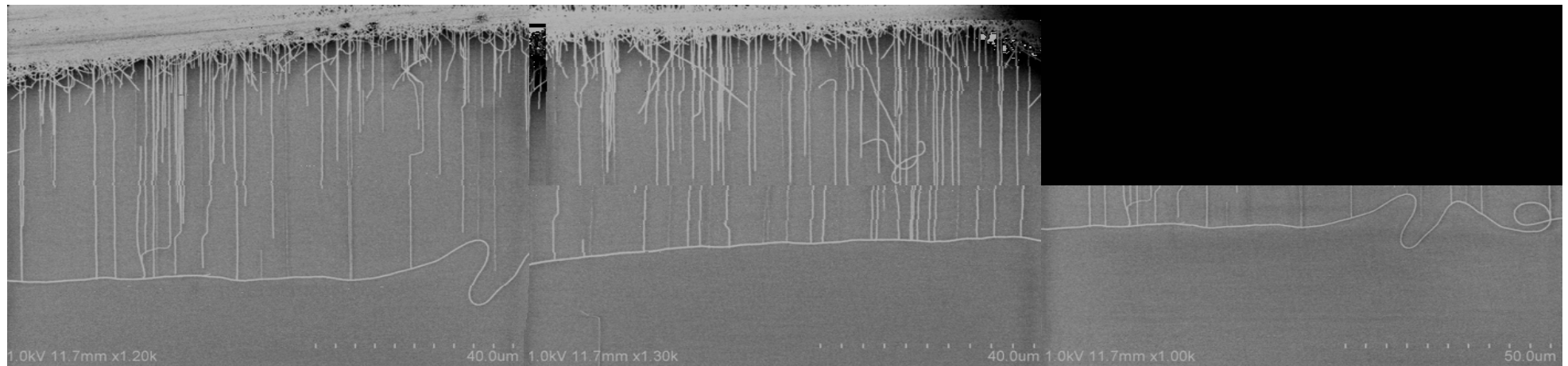
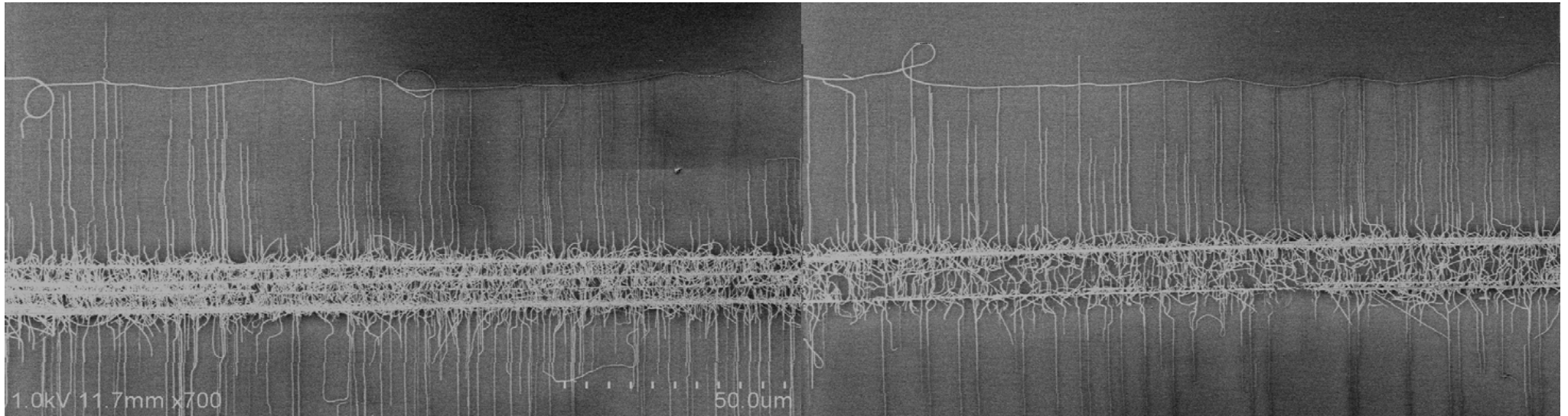


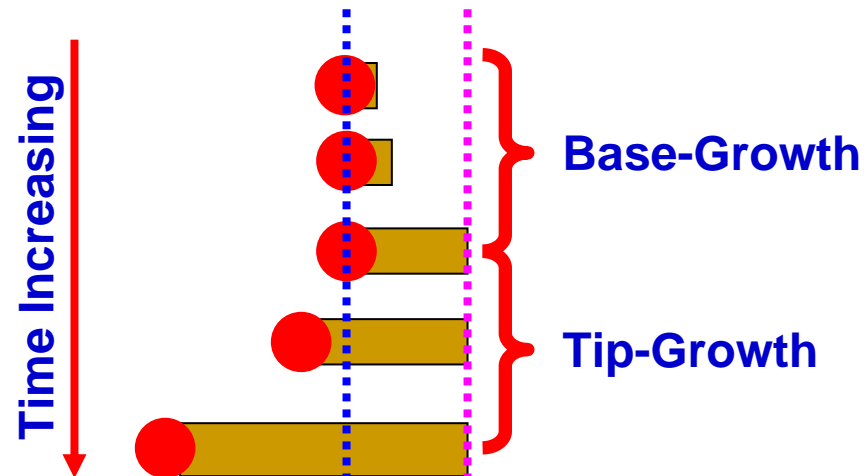
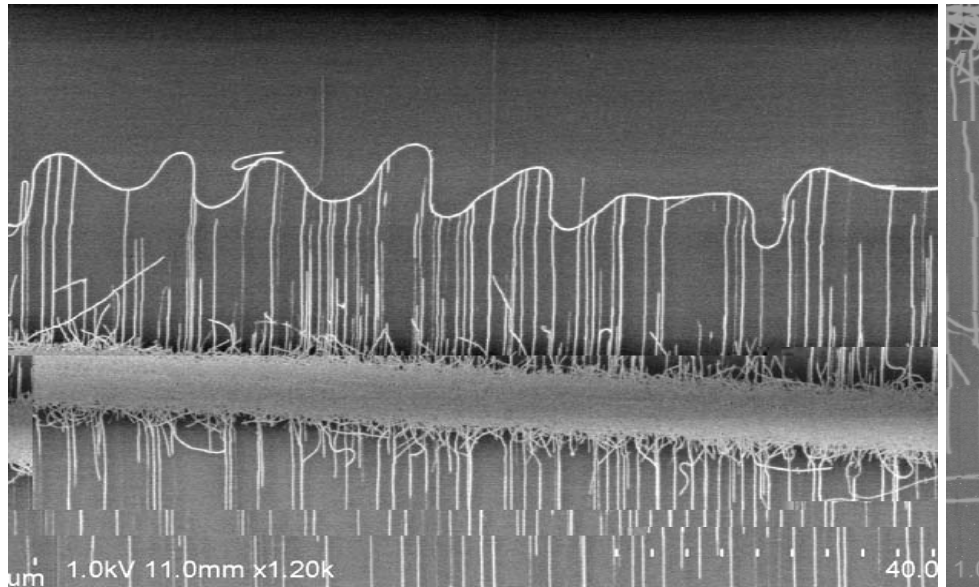
One carbon nanotube hit against another one.



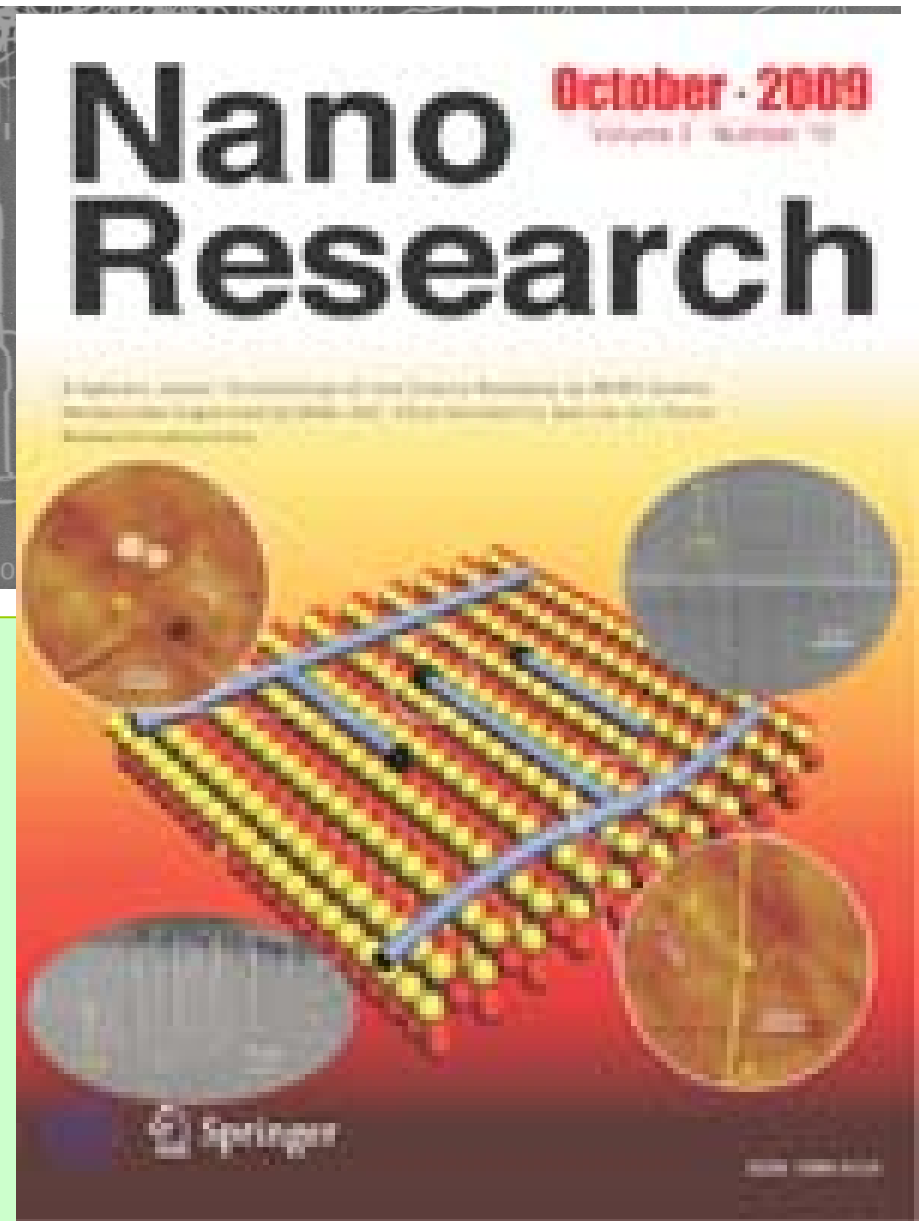
Base-growth Mechanism

Length-controlled Growth of SWCNTs



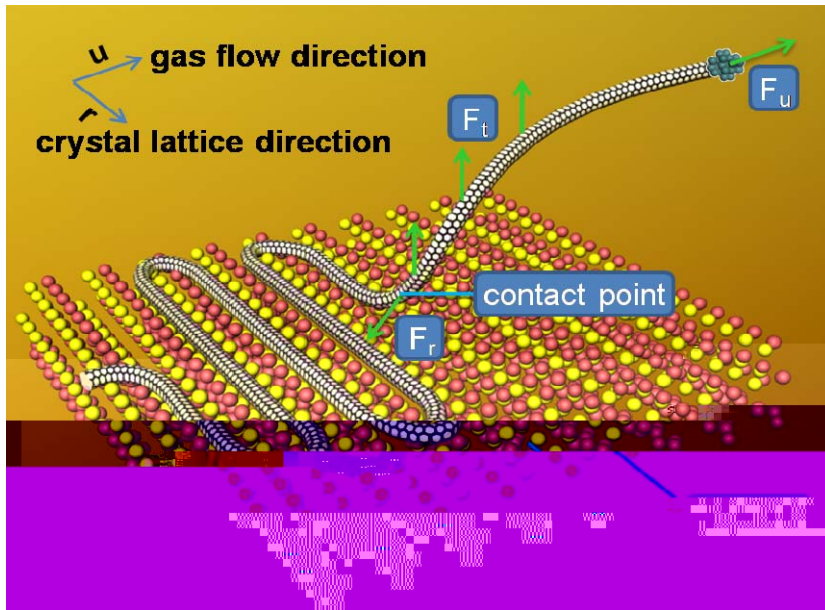


Growth Mode of SWNTs on Quartz



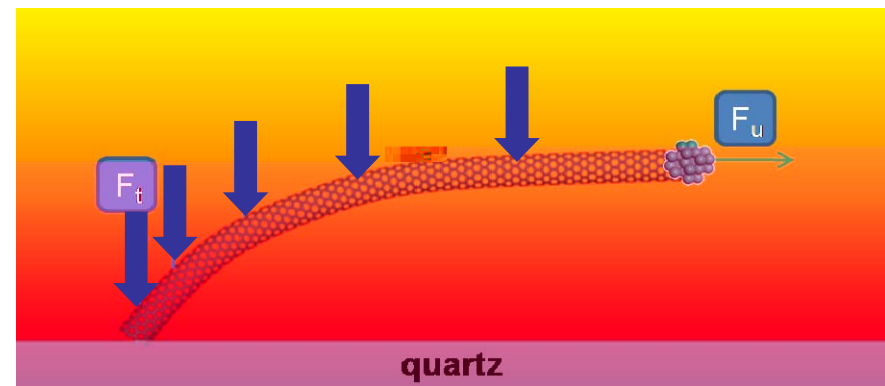
J. Zhang et al., Nano Research., 2009, 2(10),768-773.(Cover paper)

Control of Local Conformation



- Shear friction force (F_u)
- Thermal buoyancy force (F_t)
- Lattice-alignment force (F_r)

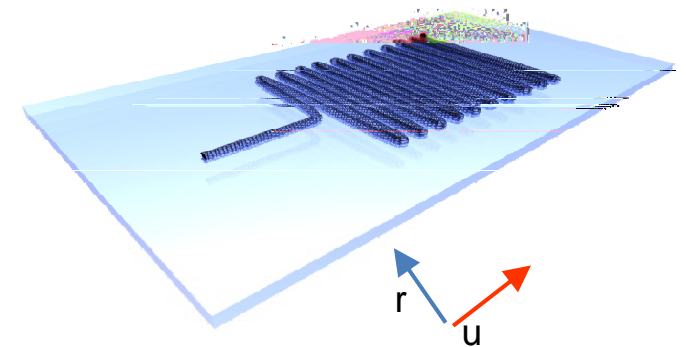
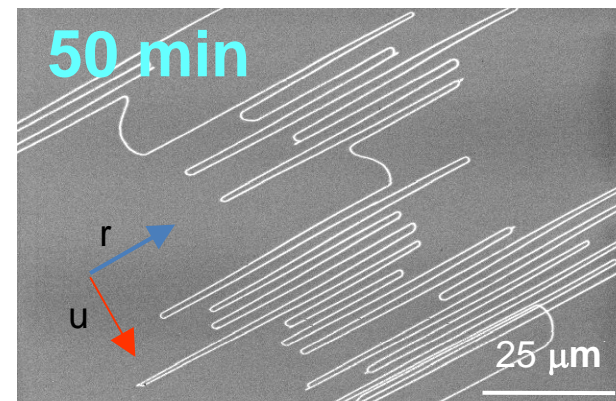
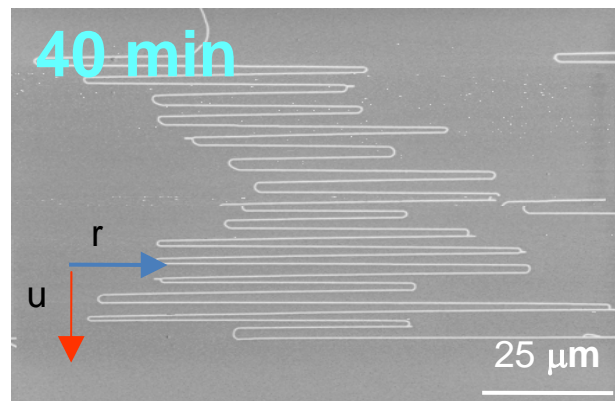
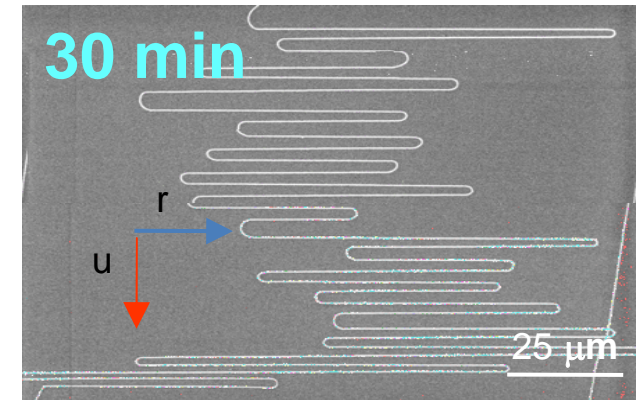
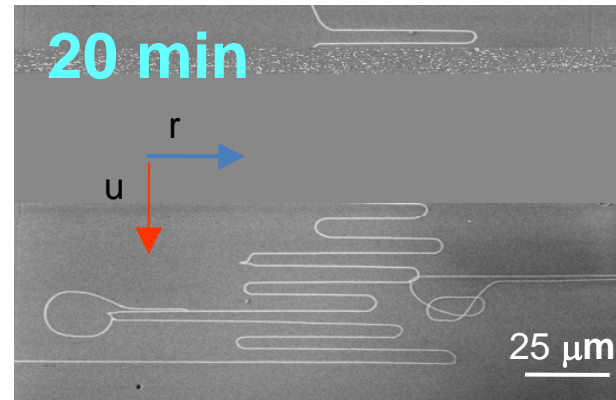
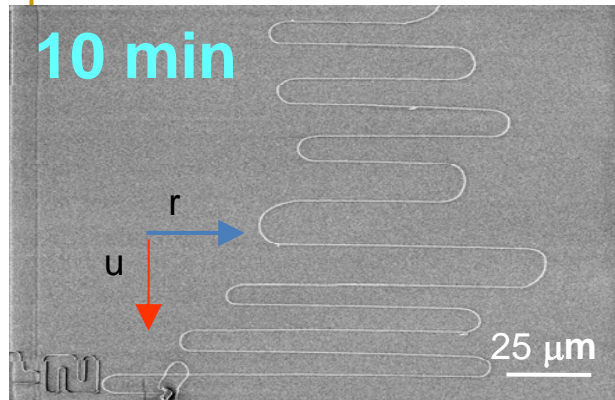
Cooling down process



Cooperation of floating and lattice-oriented modes

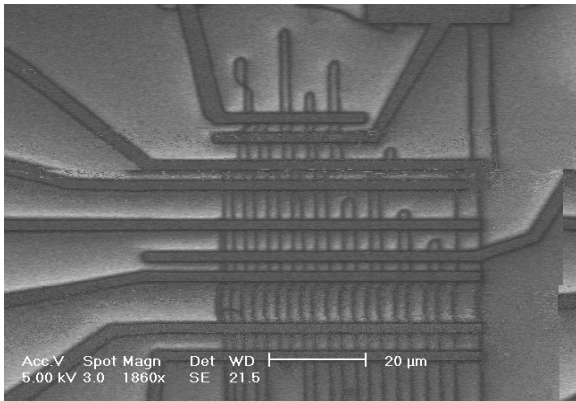
- (1) Activate the floating mode by growing at high temperature (975°C);
- (2) Stop the growth and switch to cooling process;
- (3) Activate the lattice-oriented mode by slowing down the falling speed.

J. Zhang et al., Adv. Mater., 2009, in press (Inside Front Cover)

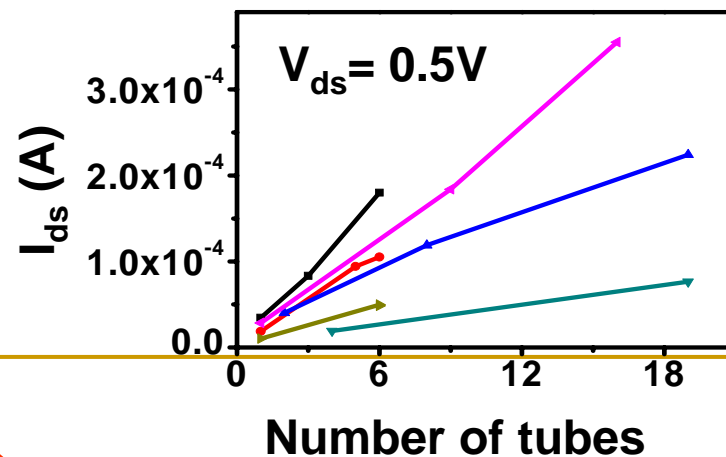
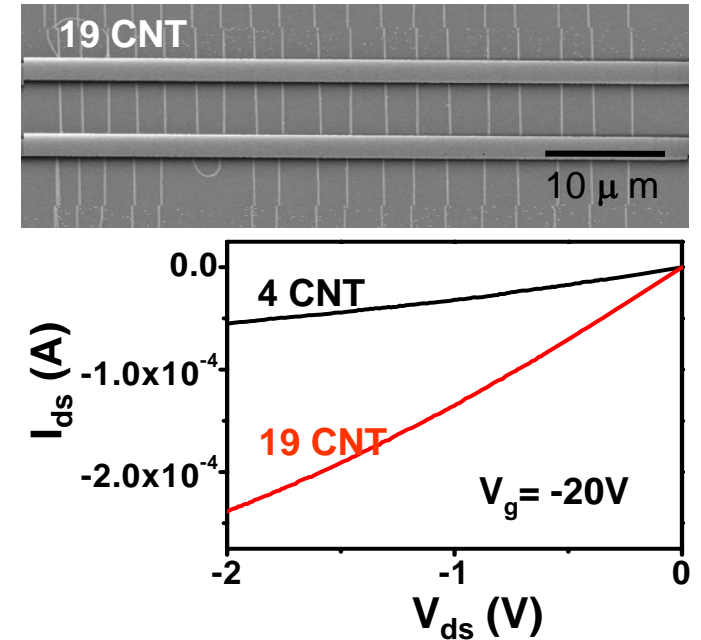
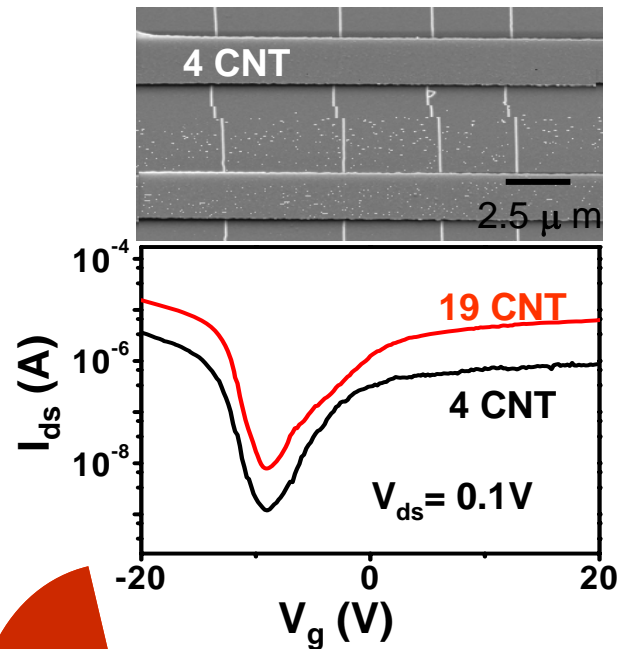


When setting gas flow direction perpendicular to the lattice orienting direction and cooling down the system from 975°C to 775°C at low speed (4-20°C/min), we found that, the slower the cooling speed, the higher the tube packing density.

High Performance SWNT-FET with Identical Chirality



Pd electrode, 800nm SiO_2
gate dielectric



Summary

Single-Walled Carbon Nanotubes



Application the SWNTs in Future Devices



Growing SWNTs on Surface
with Controlled Structures

Although it is still difficult to make a precise control of the diameter, chirality and local band structure of single-walled carbon nanotubes, there exists a big space for further efforts.

Thank Yo

