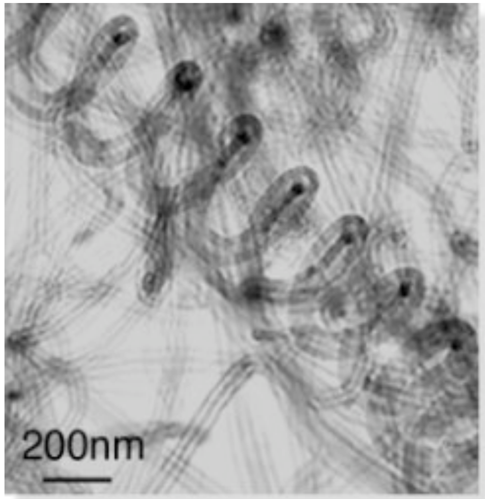
Nanotechnology

4th lecture



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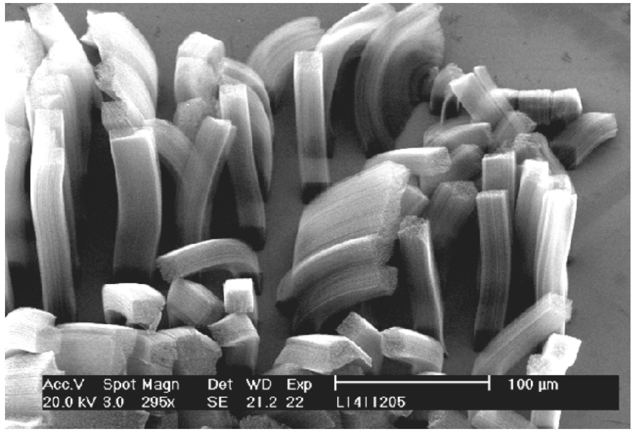

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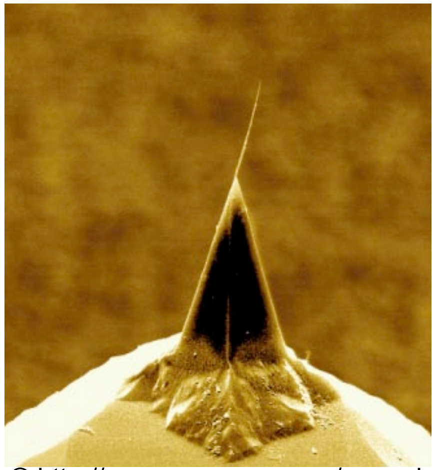

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Last time we had:

2.3. Solid State Devices

Self-Assembled Monolayers

Nanogaps and Nanowires

2.4. Conclusions and Outlook

3. Introduction to Carbon Nanotubes

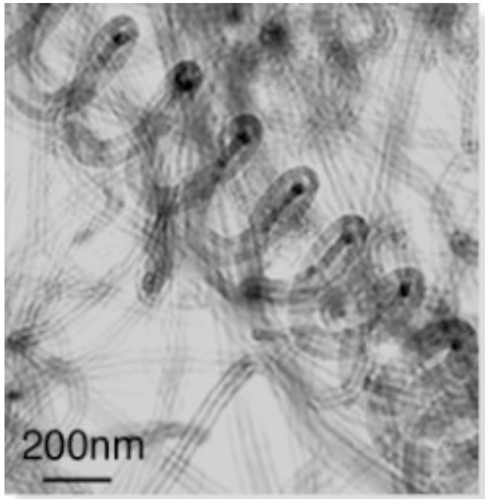
3.1 Structure of Carbon Nanotubes

IAP

Nanotechnology

4th lecture

Technische Universität Wien
Vienna University of Technology



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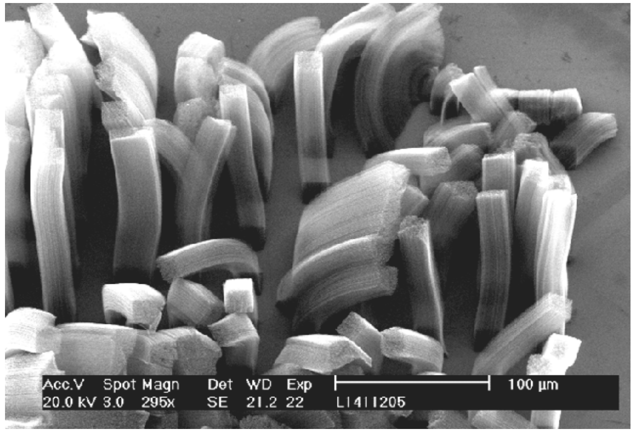

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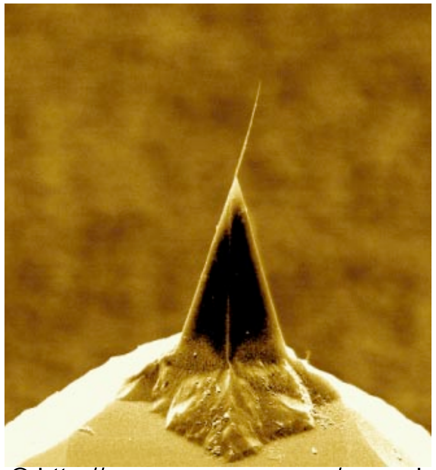
Plan for today:

3. Introduction to Carbon Nanotubes

- ***3.1 Structure of Carbon Nanotubes***
- ***3.2 Synthesis of Carbon Nanotubes***
- ***3.3 Growth mechanisms of Carbon Nanotubes***
- ***3.4 Properties of Carbon Nanotubes***
- ***3.5 Carbon Nanotube based nano-objects***
- ***3.6 Applications of Carbon Nanotubes***



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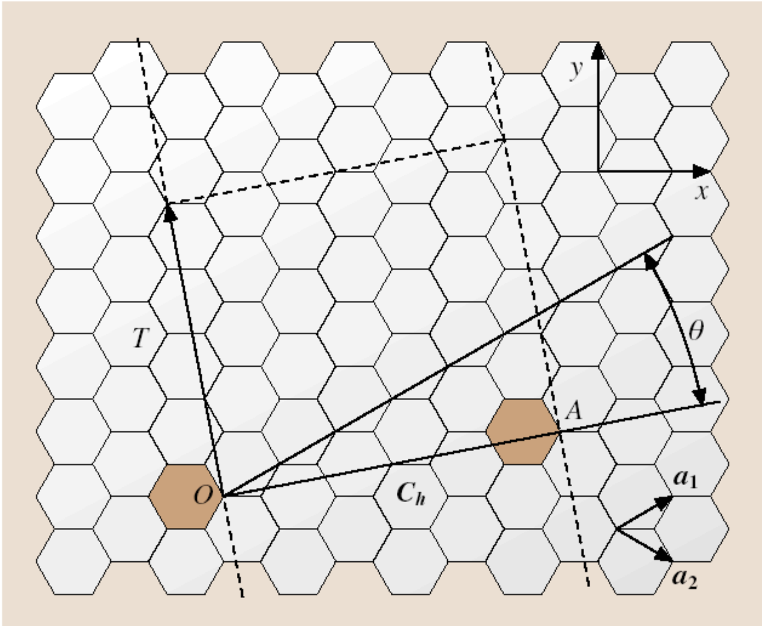
Structure of carbon nanotubes

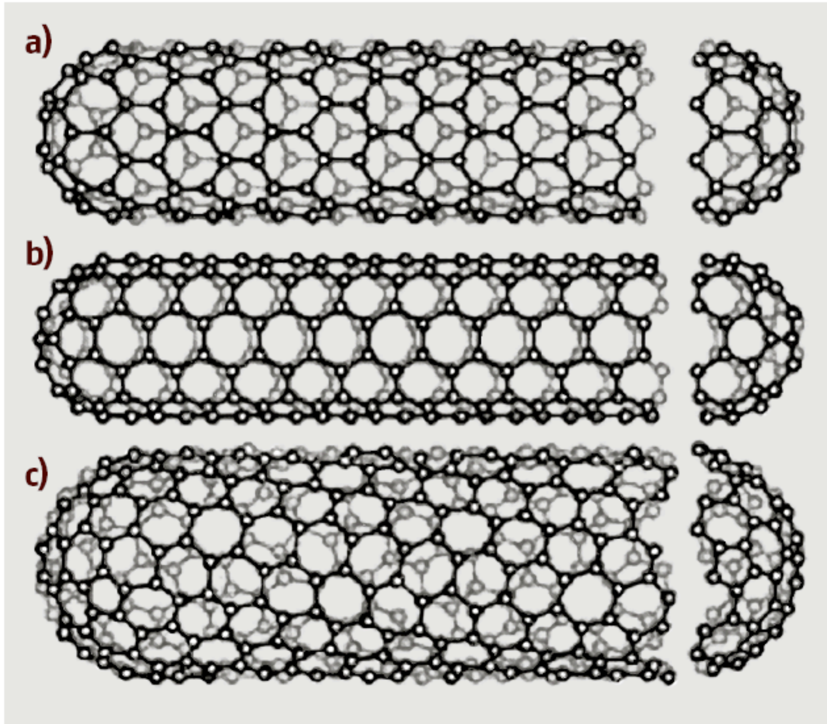
Graphene sheets

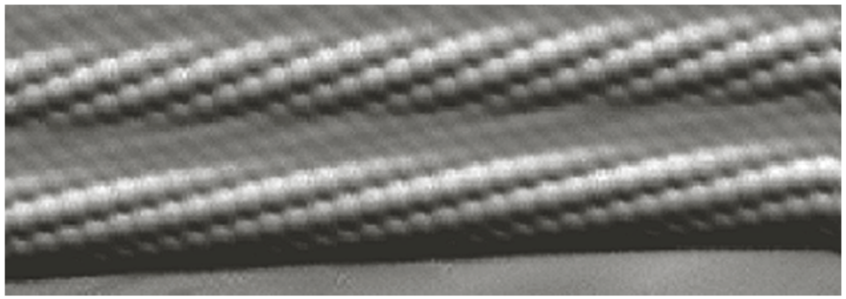


A **graphene sheet** is a polyaromatic mono-atomic layer made of an hexagonal display of **sp^2 hybridized carbon atoms** that genuine graphite is built up with.

Single-Wall Nanotubes

Roll a perfect graphene **sheet** into a cylinder, and close the tips by two caps, each **cap** being a hemi-fullerene with the appropriate diameter.

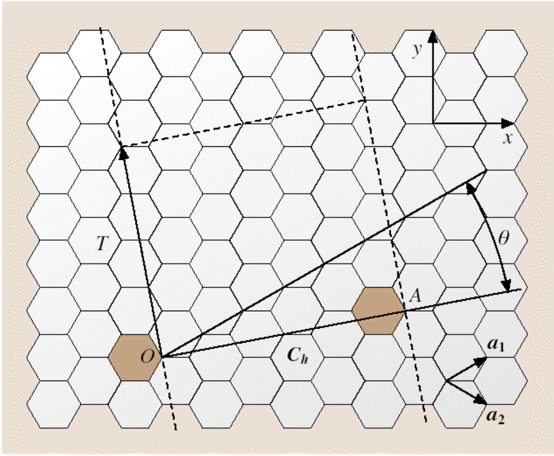


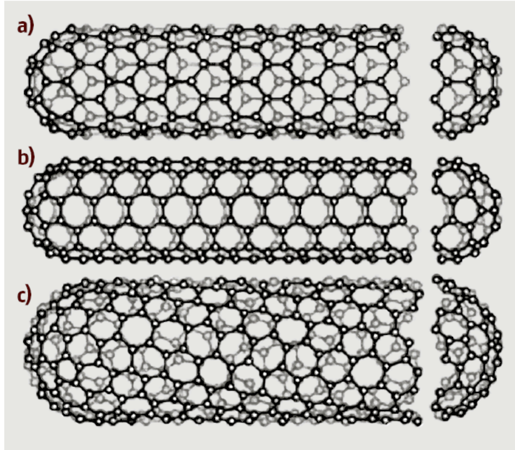


- **Maximum diameter** of SWNT: **2.5nm**

  (below this value, flattened two-layer sheets are energetically more favourable).
- **Minimum diameter**: **0.4nm**
- **Suitable energetic compromise** for diameter: **1.4nm**
- **Length**: up to micrometers or even **millimeters**!
- SWNTs are unique examples of **single molecules with high aspect ratio**.

Consequences from structure



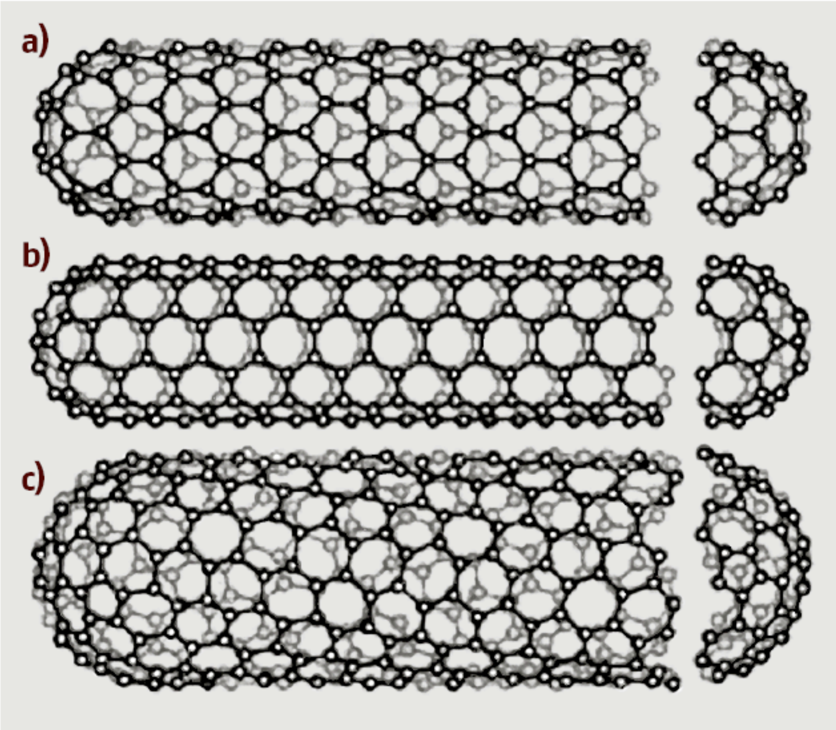


1. All carbon atoms are involved in **hexagonal** aromatic rings only and are therefore in equivalent position, except at the nanotube tips where 6×5 = 30 atoms at each tip are involved in **pentagonal** rings (considering that adjacent pentagons are unlikely).
2. The **chemical reactivity** of SWNTs is **highly favored at the tube tips**, at the very location of the pentagonal rings.
3. **No pure sp^2 hybridization anymore**, since C=C bond angles are no longer planar.

sp^2 vs. sp^3 hybridisation

- The hybridization of carbon atoms are no longer pure sp^2 but get some percentage of the **sp^3 character**, in a proportion that **increases as the tube radius of curvature decreases**.
- **In C_{60} molecules** (highest radius of curvature), the **sp^3 proportion is about 30%**.

SWNT structures



Zig-zag type

Armchair type

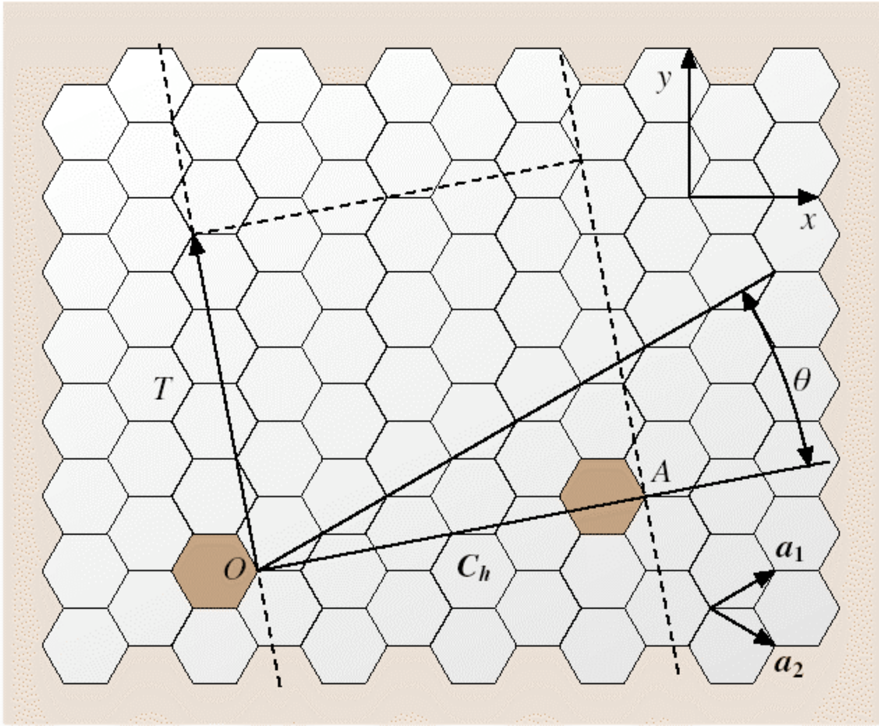
Helical type

$$\vec{C}_h = n\vec{a}_1 + m\vec{a}_2$$

$$\vec{a}_1 = \frac{a\sqrt{3}}{2}\vec{x} + \frac{a}{2}\vec{y}$$

$$\vec{a}_2 = \frac{a\sqrt{3}}{2}\vec{x} - \frac{a}{2}\vec{y}$$

$$a = 0.246nm$$



$$\cos\theta = \frac{2n+m}{2\sqrt{n^2+m^2+nm}}$$

$$D = \frac{\left|\vec{C}_h\right|}{\pi} = \frac{a_{cc}\sqrt{3(n^2+m^2+nm)}}{\pi}$$

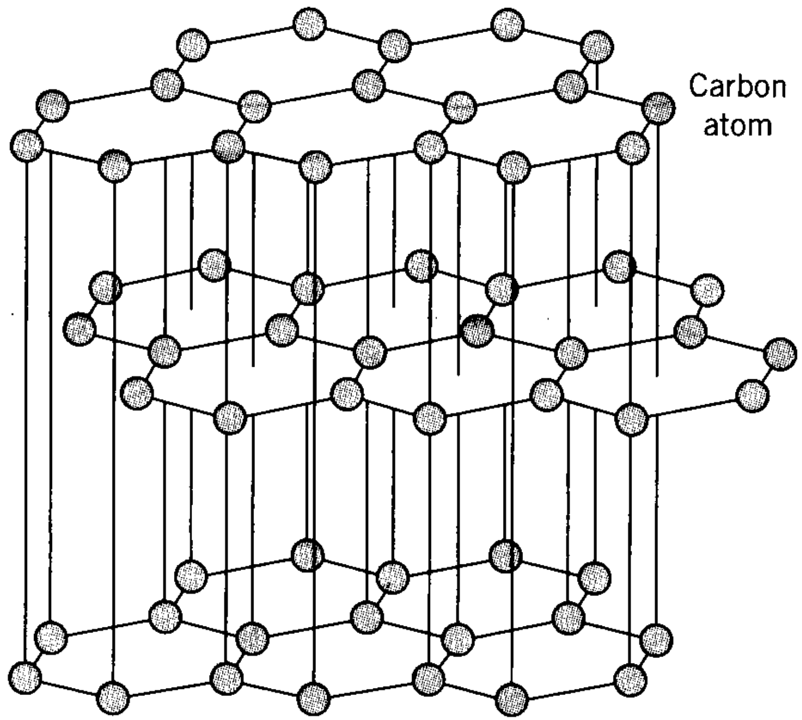
$$0.141nm(graphite) \le a_{cc} = a_{c=c} \le 0.144nm(C_{60})$$

(n,m) SWNTs

- The C-C bond length is elongated by the curvature imposed.
- *n* and *m* are **sufficient** to define any SWNT specifically, by noting it ***(n,m)***.
- **Zig-zag** types are denoted by **(n,0).**
- **Armchair** type are denoted by **(n,n).**
- **Helical** types are denoted by **(n,m).**

Graphite

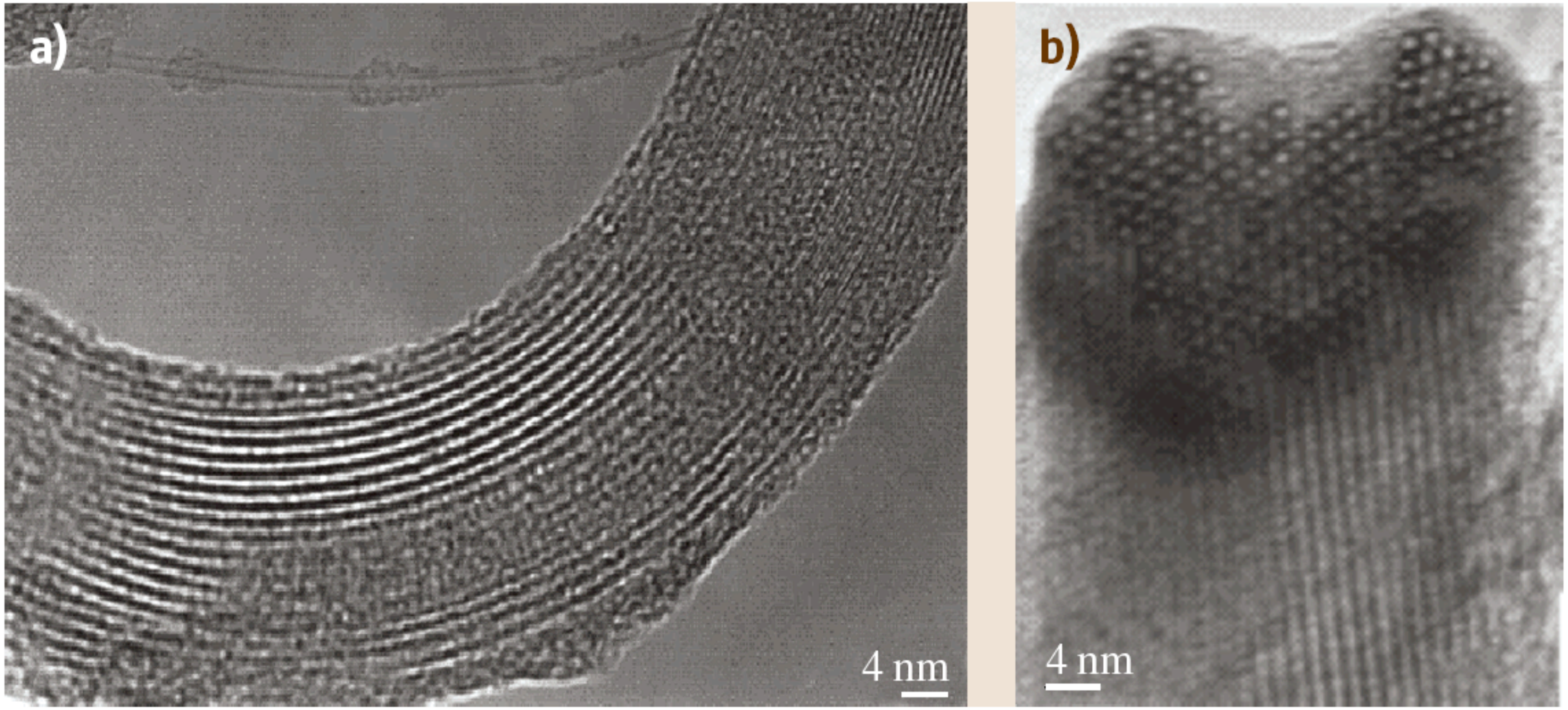
Planar graphenes have π electrons which are satisfied by the **stacking** of graphenes into sheets that allow **van der Waals forces** to develop.



Gathering of SWNTs

- Similar reasons make fullerenes gather and order into **fullerite crystals** and SWNTs into **SWNT ropes**.
- Spontaneously, SWNTs in ropes tend to arrange into an **hexagonal array**, which corresponds to the highest compactness achievable.

Gathering of SWNTs

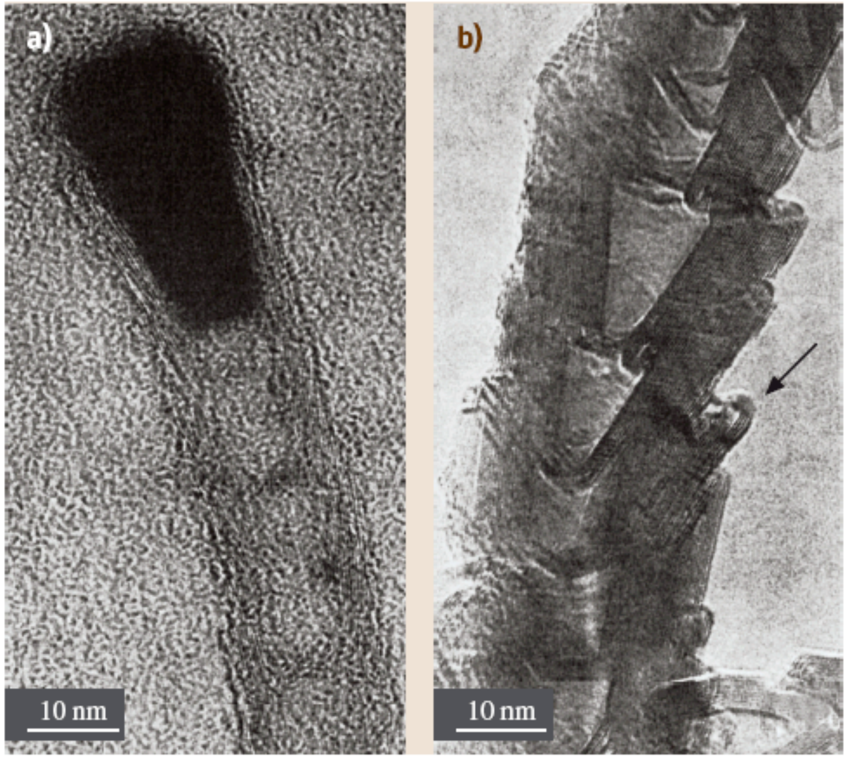


TEM of an SWNT rope in (a) longitudinal view and (b) cross section view.

Multiwall nanotubes: MWNTs

- The image shows a c-MWNTs, i.e. a concentric MWNT.
- c-MWNTs can be fabricated by several techniques, with or without catalyst.
- The number of walls can be from 2 to no upper limit.
- The intertube distance is 0.34nm (as opposed to 0.335nm in genuine graphite).
- Some c-MWNTs exhibit faceted morphologies (see image).
- The facets allow graphenes to go back to a flat arrangement of atoms.

Herringbone MWNT



(a) as-grown
(b) after 2900°C heat-treatment.

Herringbone MWNT

- In h-MWNTs the graphenes make an angle with respect to the nanotube axis.
- If the angle is 0 degrees, we have c-MWNT.
- If the angle is 90 degrees, we have graphenes stacked as piled up plates.
- h-MWNTs are exclusively obtained by processes involving catalysts, generally catalyst-enhanced thermal cracking of hydrocarbons or CO disproportionation.

Unresolved question in h-MWNTs

Does the herringbone texture originate from the scroll-like spiral arrangement of a single graphene ribbon or from the stacking of independent truncated-cone-like graphenes?

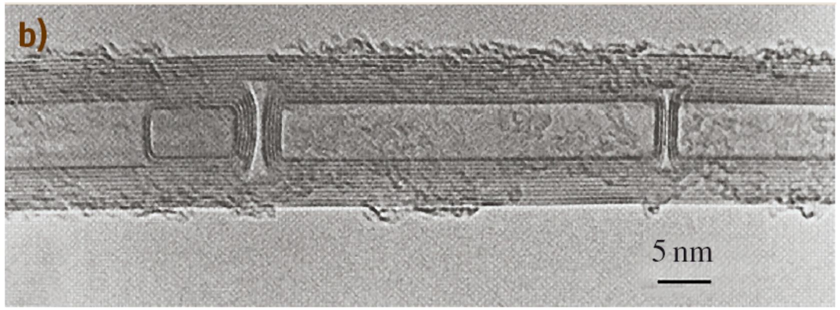
Bamboo texture

- A limited amount of graphenes in the MWNT are oriented perpendicular to the nanotube axis.
- These structures should rather be called nanofibers, because they are no tubes anymore.

bh- and bc-MWNTs

- bh-MWNT (bamboo-herringbone WMNT)

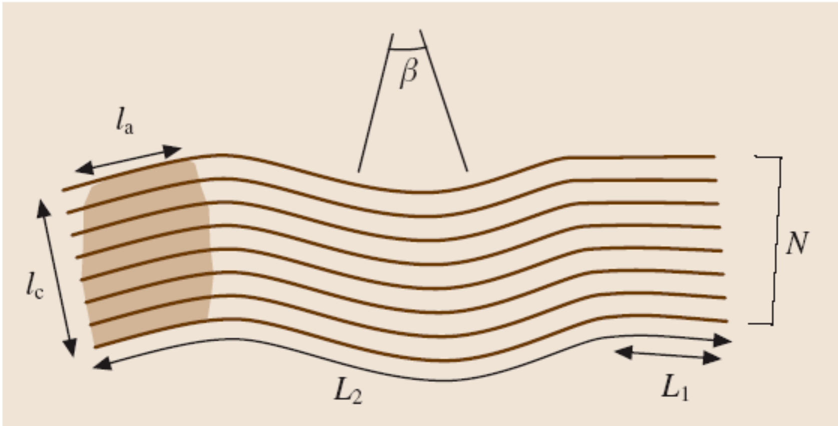
- bc-MWNT (bamboo-concentric MWNT)



Properties of MWNTs (1/2)

- Aspect ratios of MWNTs and nanofibers are usually smaller than that from SWNTs.
- The nanotube tips are frequently found associated with the catalyst crystal from which they have been formed.

Properties of MWNTs (2/2)



- Graphene perfection is quantified by several parameters preferably obtained from high resolution transmission electron microscopy.
- nanotexture can be improved by subsequent thermal treatments at high temperatures (e.g., > 2,000 ◦C).

3.2. Synthesis of Carbon Nanotubes

Today, we **cannot specifically control** chirality, purity and structural quality of SWNTs, due to the lack of knowledge regarding several **parameters** controlling the synthesis conditions.

Solid Carbon Source-Based Production Techniques for Carbon Nanotubes

1. Laser ablation.
2. Solar energy furnace.
3. Electric arc.

Common features of these techniques:

- high temepratrure (1000K<T<6000K)
- the carbon source originates from the erosion of solid graphite

Drawback: SWNTs never come pure.

Solid Carbon Source-Based Production Techniques for Carbon Nanotubes

- Before being utilized for carbon nanotube synthesis, the laser ablation, solar energy furnace and electric arc methods were used for the production of fullerenes.
- A common feature of these techniques is the formation of a plasma, i. e. an electrically neutral ionized gas, composed of neutral atoms, charged particles (molecules and ionized species), and electrons.
- Ionisation degree:

$$i = \frac{n_e}{n_e + n_0}$$

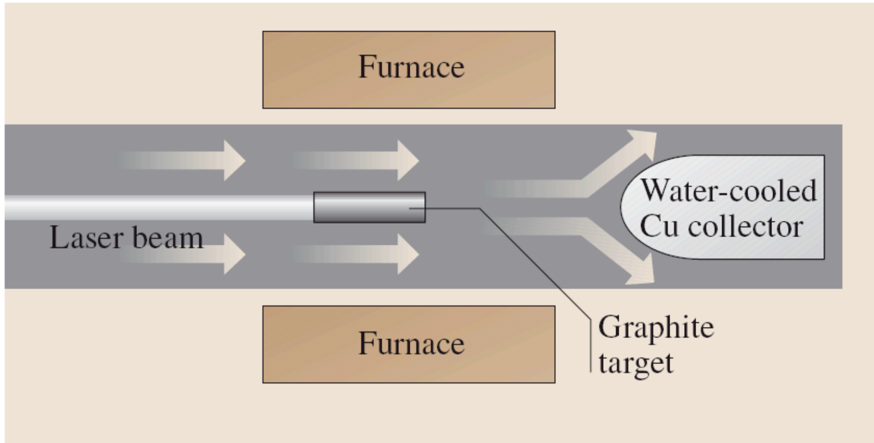
n_e - density of electrons

n_0 - density of neutral particles

1. Laser ablation

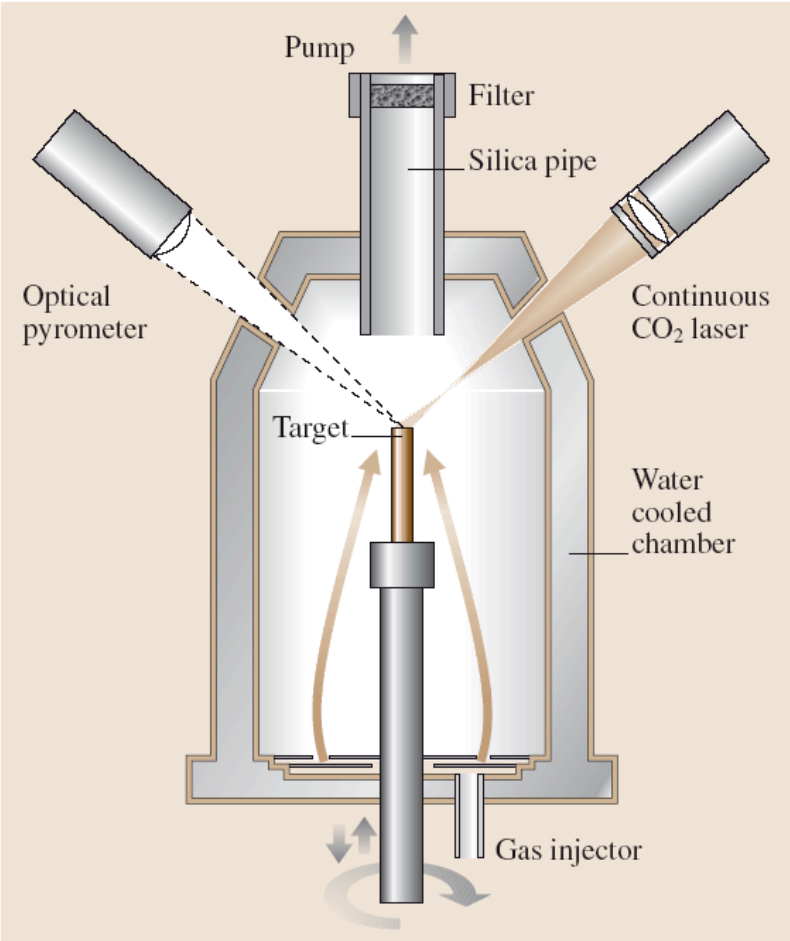
- The first lasers were built in 1960.
- Advantage: concentration of a large quantity of energy inside a very small volume within a relatively short time.

Laser ablation – experimental devices



- laser working in pulsed mode or continuous mode
- graphite pellet and catalyst pellet in inert gas-filled quartz tube placed in an oven
- 1200 ○C
- laser beam focused on the pellet
- graphite vaporizes and sublimes
- carbon species swept away by a flow of neutral gas
- carbon deposited in different regions.

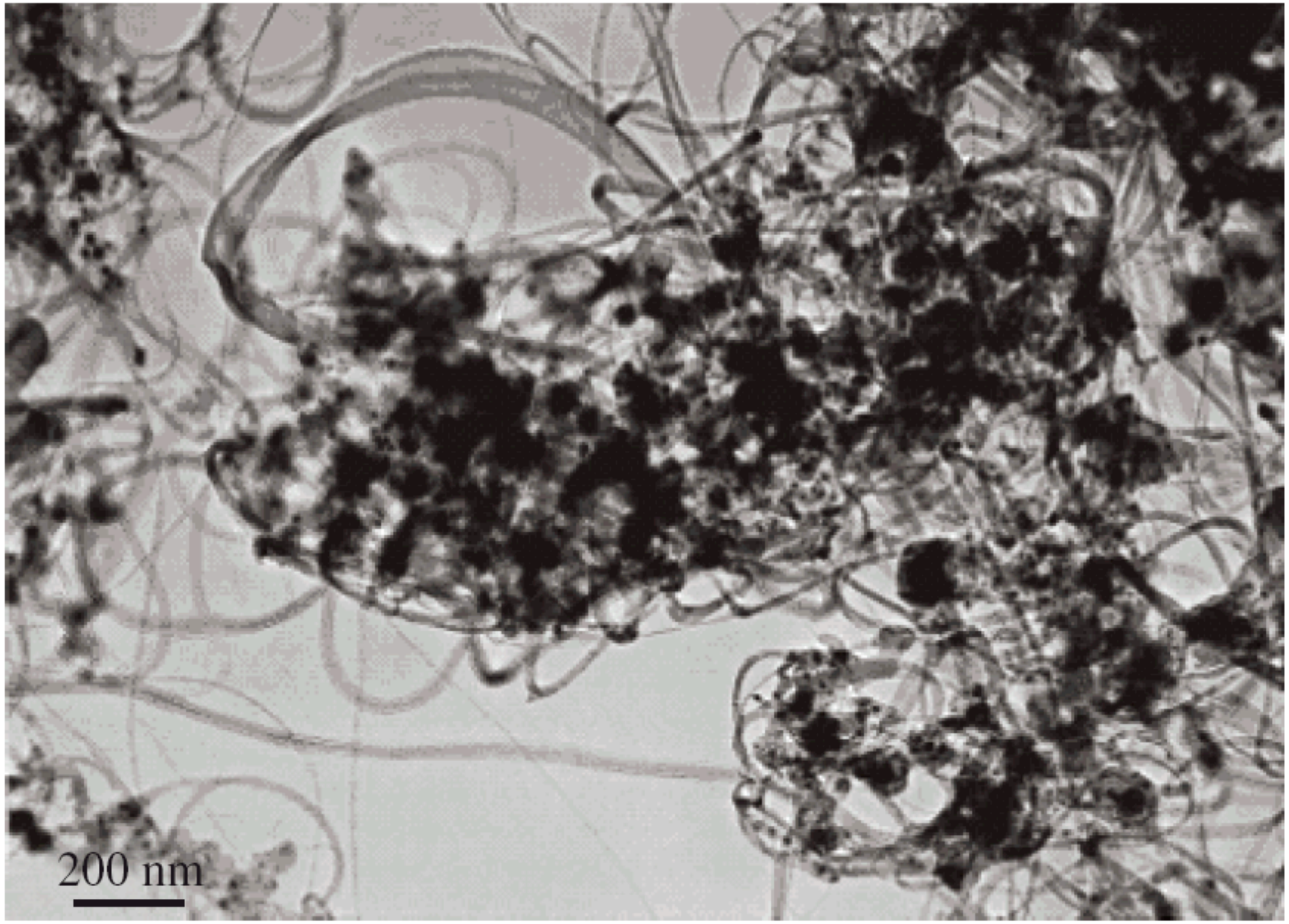
Laser ablation with CO_2 laser



- Operated with a continuous CO_2 laser, wavelength 10.6 micrometers (mid-IR).
- 100W to 1600W
- Optical pyrometer used to regulate the vaporization temperature.
- The solid products are collected on the filter.

Laser ablation - Results

- In the absence of catalysts in the target, mainly c-MWNTs with a length up to 300nm are produced.
- The best quality is obtained for an oven temperature of 1,200°C.
- When catalysts (less than 1% of transition metals like Co or Ni) are added, SWNTs are formed.
- These SWNTs have remarkably uniform diameter and they self-organize into rope-like crystallites 5–20 nm in diameter and tens to hundreds of micrometers in length.
- The ends of all SWNTs appear to be perfectly closed with hemispherical end caps.
- Finding the two tips of a SWNT is rather challenging, considering the huge aspect ratio of the nanotube and their entanglement.



TEM image of raw SWNT material obtained from the laser vaporization technique.
Fibrous morphologies are SWNT bundles, and dark particles are catalyst remnants.

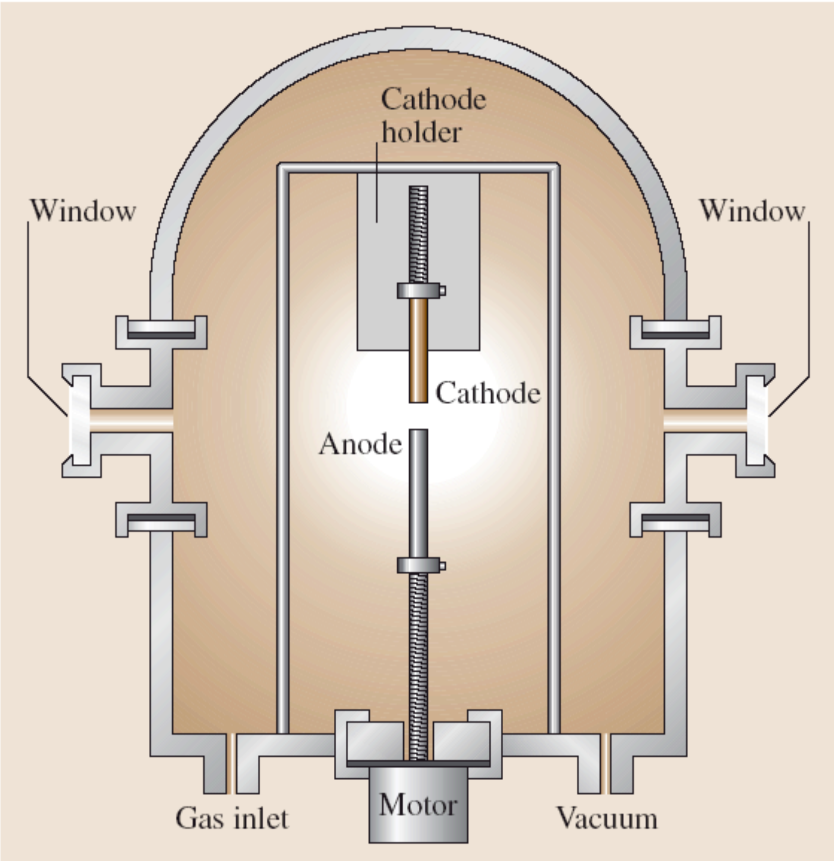
Laser ablation - Summary

- The laser ablation method is one of the three methods currently used to prepare SWNTs as commercial products.
- Market situation:
  - December 2002: 1000$/g raw material.
  - Today (2007): 200Euro/g (www.nanobest.co.kr)
  - Prediction: 0.03$/g

2. Electric arc method

- Electric arcs between carbon electrodes have been utilized it to obtain fullerenes in macroscopic quantities.
- *Iijima* discovered the catalyst-free formation of perfect c-MWNT-type carbon nanotubes using this method.
- Catalyst-promoted formation of SWNTs was incidentally discovered after some amounts of transition metals were introduced into the anode in an attempt to fill the c-MWNTs with metals while they grow.

Electric arc method – experimental devices



- The principle of this technique is to vaporize carbon in the presence of catalysts (iron, nickel, cobalt, yttrium, boron, gadolinium, and so forth) under reduced atmosphere of inert gas (argon or helium).
- The sketched reactor is 30cm in diameter and 1 meter in hight.
- The graphite electrodes are a few millimeters in diameter.

Electric arc method – Results

- Both the nanotube morphology and the nanotube production efficiency strongly depend on the experimental conditions and, in particular, on the nature of the catalysts.
- Many parameters – high dimensional system → see table.

Catalyst (atom%) **Arc conditions**	**0.6Ni + 0.6Co (homogeneous anode)** $P \sim 60\,\text{kPa}$ $I \sim 80\,\text{A}$	**0.6Ni + 0.6Co (homogeneous anode)** $P \sim 40\,\text{kPa}$ $I \sim 80\,\text{A}$	**0.5Ni + 0.5Co** $P \sim 60\,\text{kPa}$ $I \sim 80\,\text{A}$	**4.2Ni + 1Y** $P \sim 60\,\text{kPa}$ $I \sim 80\,\text{A}$
Soot	• **MWNT** + **MWS** + **POPAC** or **Cn** ± catalysts $\phi \sim 3$–$35\,\text{nm}$ • NANF + catalysts • AC particles + catalysts • [*DWNT*], [*SWNT*], ropes or isolated, + POPAC	• **POPAC** and AC particles + **catalysts** $\phi \sim 2$–$20\,\text{nm}$ • NANF + catalysts $\phi \sim 5$–$20\,\text{nm}$ + MWS • [*SWNT*] $\phi \sim 1$–$1.4\,\text{nm}$, distorted or damaged, isolated or ropes + Cn	• **AC** and POPAC particles + catalysts $\phi \sim 3$–$35\,\text{nm}$ • **NANF** + catalysts $\phi \sim 4$–$15\,\text{nm}$ • [*SWNT*] $\phi \sim 1.2\,\text{nm}$, isolated or ropes	• **POPAC** and **AC** + particles + catalysts $\phi \leq 30\,\text{nm}$ • SWNT $\phi \sim 1.4\,\text{nm}$, clean + Cn, short with tips, [*damaged*], isolated or ropes $\phi \leq 25\,\text{nm}$ • [*SWNC*] particles
Web	• [*MWNT*], **DWNT**, $\phi\, 2.7 - \mathbf{4} - 5.7\,\text{nm}$ SWNT $\phi\, 1.2$–$1.8\,\text{nm}$, isolated or **ropes** $\phi < 15\,\text{nm}$, + POPAC ± **Cn** • AC particles + catalysts $\phi \sim 3$–$40\,\text{nm}$ + MWS • [*NANF*]	None	None	• **SWNT**, $\phi \sim 1.4\,\text{nm}$, isolated or **ropes** $\phi \leq 20\,\text{nm}$, + **AC** • POPAC and AC particles + catalysts $\phi \sim 3 - \mathbf{10} - 40\,\text{nm}$ + MWS
Collaret	• **POPAC** and **SWNC** particles • Catalysts $\phi \sim 3$–$250\,\text{nm}$, $< \mathbf{50}\,\text{nm}$ + MWS • SWNT $\phi\, 1$–$1.2\,\text{nm}$, [*opened*], **distorted**, isolated or **ropes** $\phi < 15\,\text{nm}$, + **Cn** • [*AC*] particles	• AC and **POPAC** particles + catalysts $\phi \sim 3$–$25\,\text{nm}$ • SWNT $\phi \sim 1$–$1.4\,\text{nm}$ clean + Cn, [*isolated*] or ropes $\phi < 25\,\text{nm}$ • Catalysts $\phi \sim 5$–$50\,\text{nm}$ + MWS, • [SWNC]	• **Catalysts** $\phi \sim 3$–$170\,\text{nm}$ + MWS • AC or POPAC particles + **catalysts** $\phi \sim 3$–$50\,\text{nm}$ • SWNT $\phi \sim 1.4\,\text{nm}$ clean + Cn isolated or ropes $\phi < 20\,\text{nm}$	• **SWNT** $\phi \sim 1.4$–$2.5\,\text{nm}$, clean + Cn, [*damaged*], isolated or **ropes** $\phi < 30\,\text{nm}$ • POPAC or AC particles + catalysts $\phi \sim 3$–$30\,\text{nm}$ • [*MWS*] + catalysts or catalyst-free.
Cathode deposit	• **POPAC** and SWNC particles • **Catalysts** $\phi \sim 5$–$300\,\text{nm}$ MWS • MWNT $\phi < 50\,\text{nm}$ • [*SWNT*] $\phi \sim 1.6\,\text{nm}$ clean + Cn, isolated or ropes	• **POPAC** and **SWNC** particles + Cn • Catalysts $\phi \sim 20$–$100\,\text{nm}$ + MWS	• **MWS**, catalyst-free • **MWNT** $\phi < 35\,\text{nm}$ • POPAC and PSWNC particles • [*SWNT*], isolated or ropes • [*Catalysts*] $\phi \sim 3$–$30\,\text{nm}$	• **SWNT** $\phi \sim 1.4$–$4.1\,\text{nm}$, clean + **Cn**, short with tips, isolated or ropes $\phi \leq 20\,\text{nm}$. • **POPAC** or **AC** particles + catalysts $\phi \sim 3$–$30\,\text{nm}$ • MWS + catalysts $\phi < 40\,\text{nm}$ or catalyst-free • [*MWNT*]

Abundant – Present – [*Rare*]

Glossary: AC: amorphous carbon; POPAC: poorly organised polyaromatic carbon; Cn: fullerene-like structure, including C_{60}; NANF: nearly amorphous nanofiber; MWS: multiwall shell; SWNT: single-wall nanotube; DWNT: double-wall nanotube, MWNT: multiwall nanotube; SWNC: single-wall nanocapsule.

Electric arc method – increase SWNT yield

- A significant increase (up to +230%) in the SWNT yield is obtained, if graphite powder (*sp*^{2} hybridized carbon) is replaced by diamond powder (*sp*^{3} hybridized carbon)
- This strongly points toward the fact that physical phenomena (charge and heat transfers) that occurr in the anode during the arc are of utmost importance.

Electric arc method – increase SWNT yield

- Utilizing a rare earth element (such as Y) associating eith a transition metal (such as Ni) leads to the highest SWNT yields.
- Utilizing a single rare earth element leads to telescope-like and open c-MWNTs able to contain nested Gd crystals.

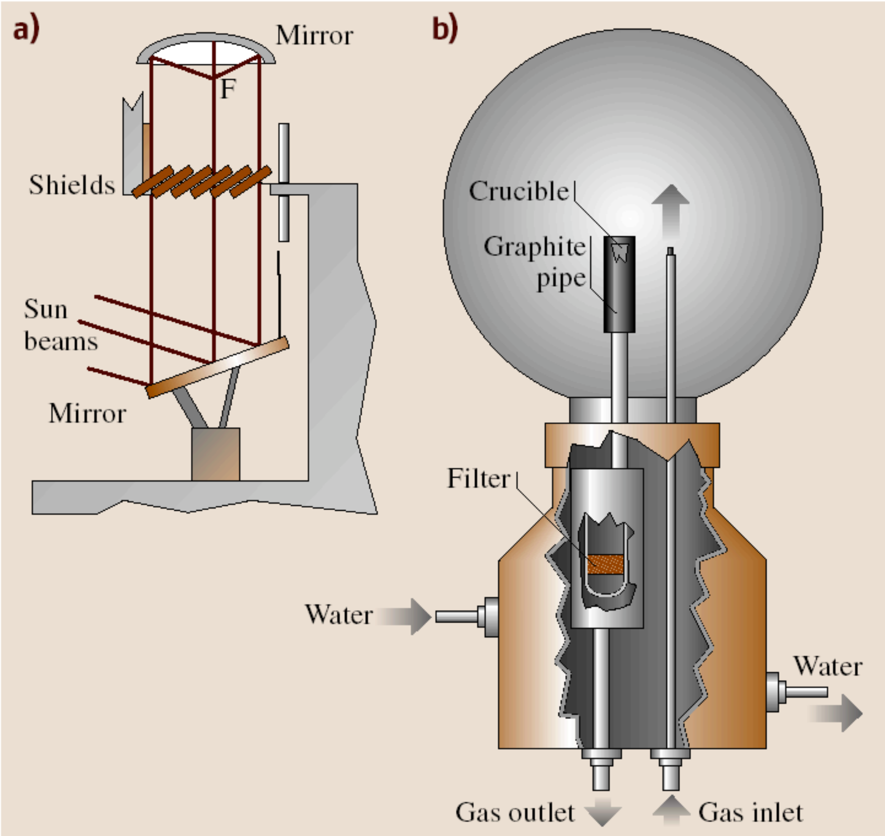
Electric arc method - Summary

- The electric arc method is one of the three methods currently used to prepare SWNTs as commercial products.
- Market situation:
  - 2003: 20kg/year, 90Euros/g raw material.
  - Prediction: 5tons/year, 2-5Euros/g before 2007.

3. Solar furnace

- Solar furnesses were originally utilized to obtain fullerenes.
- Using more powerful ovens allows for production of nanotubes.

Solar furnace – Experimental devices



Principle: sublimation of a mixture of graphite powder and catalysts placed in a crucible in an inert gas.

Solar rays are focussed onto the graphite pellet → 4000K.

Carbon and catalysts vaposize, vapors are dragged by the gas and condense.

Solar furnace – results

- Crucible filled with powered graphite for production of fullerenes.
- Crucible filled with mixture of graphite and catalysts for nanotube synthesis.
- MWNTs, SWNTs, fullerenes.
- With Co as catalyst: SWNTs with 1-2nm diameter.

- The laser ablation, the electric arc method and the solar furnace were all solid carbon based methods.
- Now we come to some gaseous carbon based production techniques for carbon nanotubes.

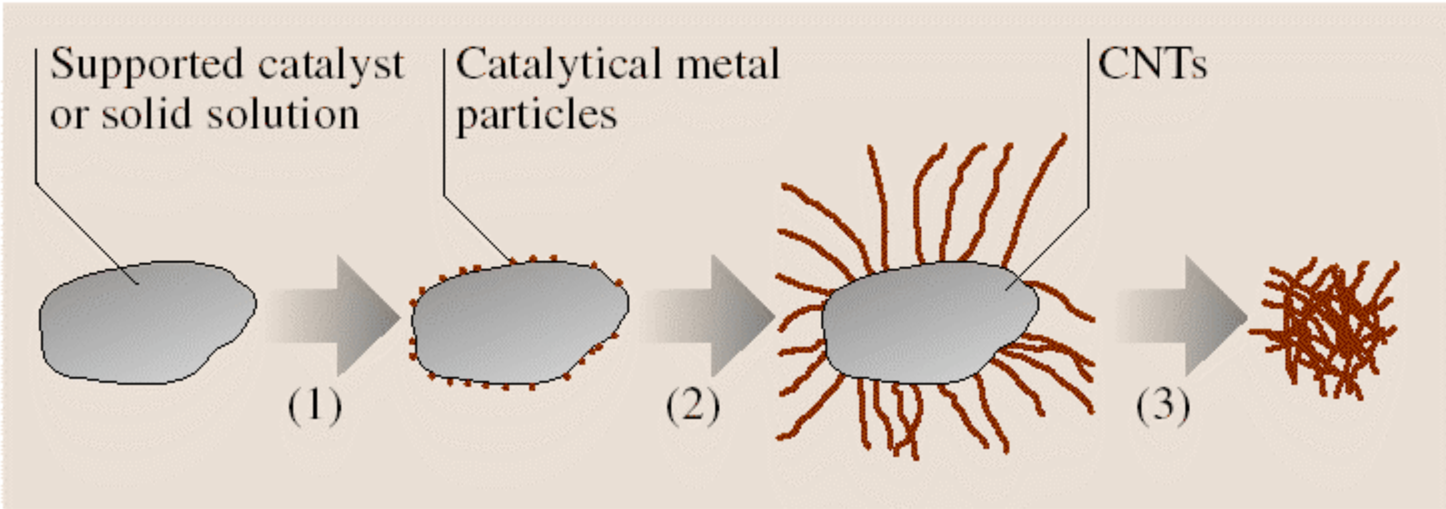
Gaseous carbon based production techniques for carbon nanotubes

- Catalytic chemical vapor deposition (CCVD) denotes catalysis-enhanced thermal cracking of gaseous carbon sources (hydrocarbons, CO).
- The tubes grow on small metallic particles or clusters.
- Low temperature process: 600-1000°C.

Gaseous carbon based production techniques for carbon nanotubes

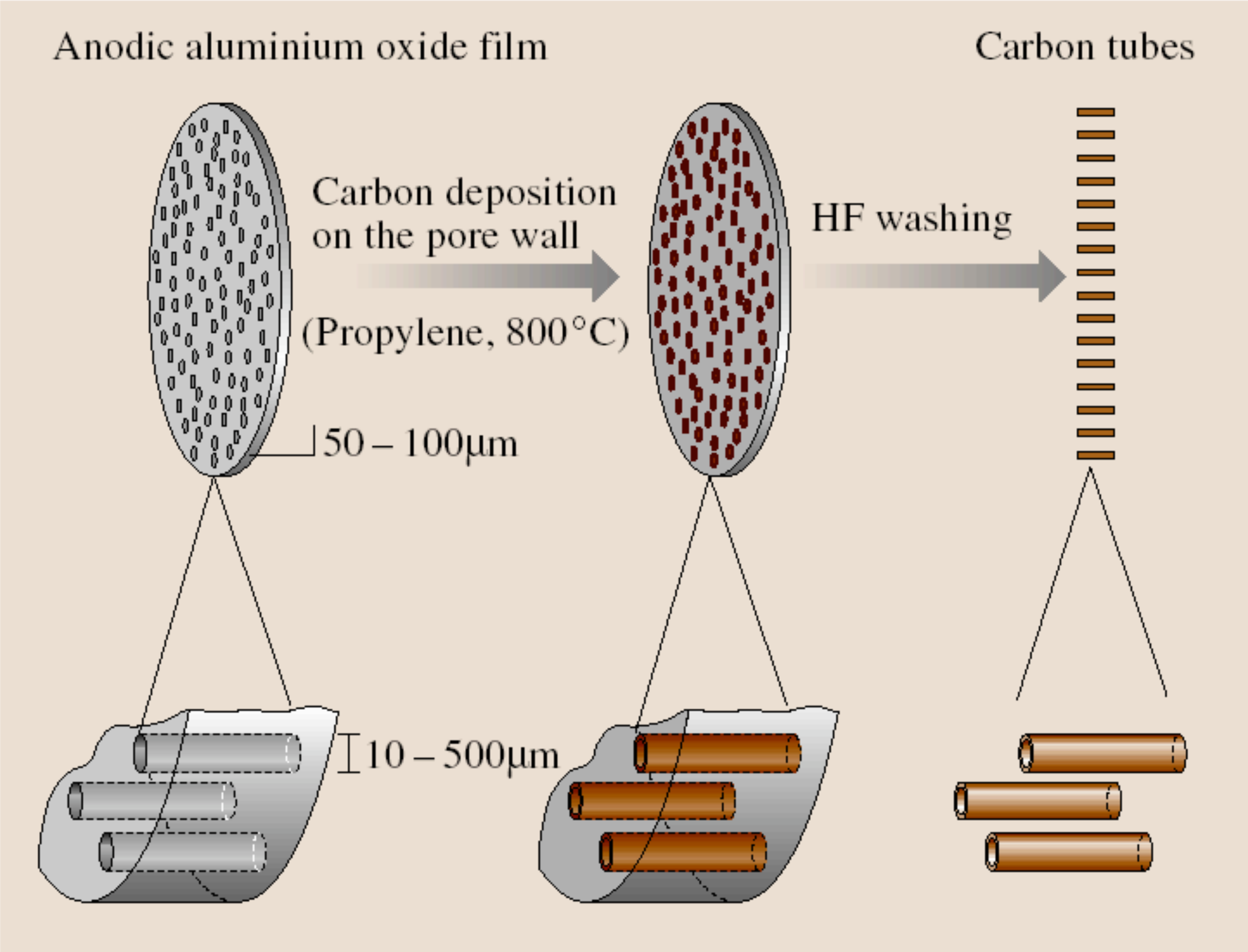
- Catalysts are very important.
- Nanotubes obtained with this method are longer and have more structural defects.
- Defects can be removed by susequent heat treatment.
- Large scale, low cost process.

CCVD



1. Formation of the catalytic metal particles by reduction of a precursor
2. Catalytic decomposition of a carbon-containing gas, leading to the growth of carbon nanotubes (CNTs)
3. Removal of the catalyst to recover the CNTs

Templating technique

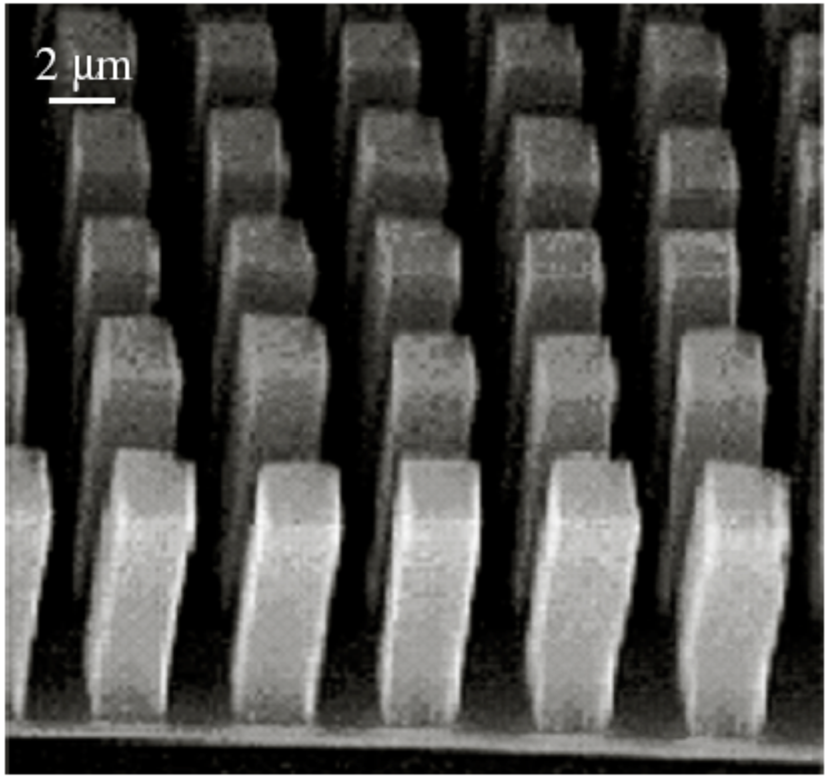


Templating technique

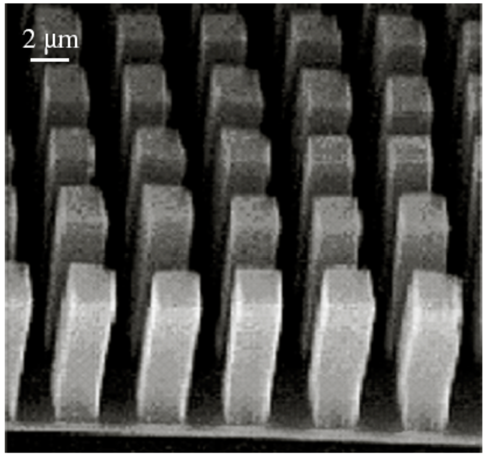
- Not suitable for mass production.
- CNT synthesis without catalyst.
- For production of c-MWNTs and SWNTs.
- Smallest SWNTs ever (0.4nm diameter) obtained with this technique.

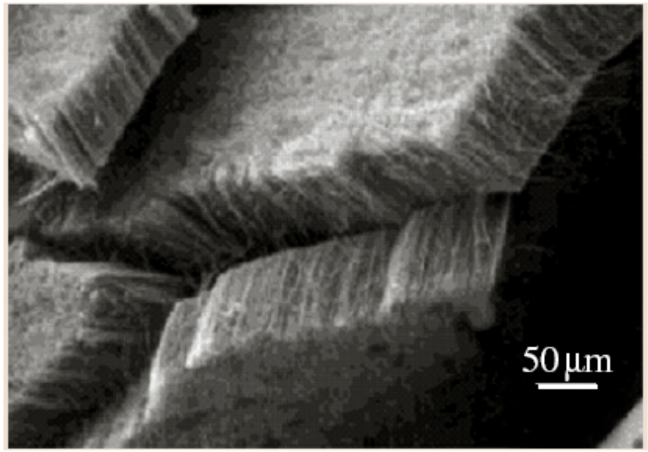
Synthesis of aligned carbon nanotubes

Several applications (such as field-emission-based display) require that carbon nanotubes grow as highly aligned bunches, in highly ordered arrays, or located at specific positions.



Production of aligned nanotubes





- CNTs self-assemle on patterned substrates, e.g. mesoporous silica (left).
- With CCVD densely packed coatings of vertically alligned MWNTs can be obtained (right).

3.3. Growth mechanisms of carbon nanotubes

- Growth mechanisms of carbon nanotubes are still debated.
- The phenomena are quite fast and difficult to observe in situ.
- It is generally agreed, however, that growth should occur so that the number of dangling bonds is limited, for energetic reasons.

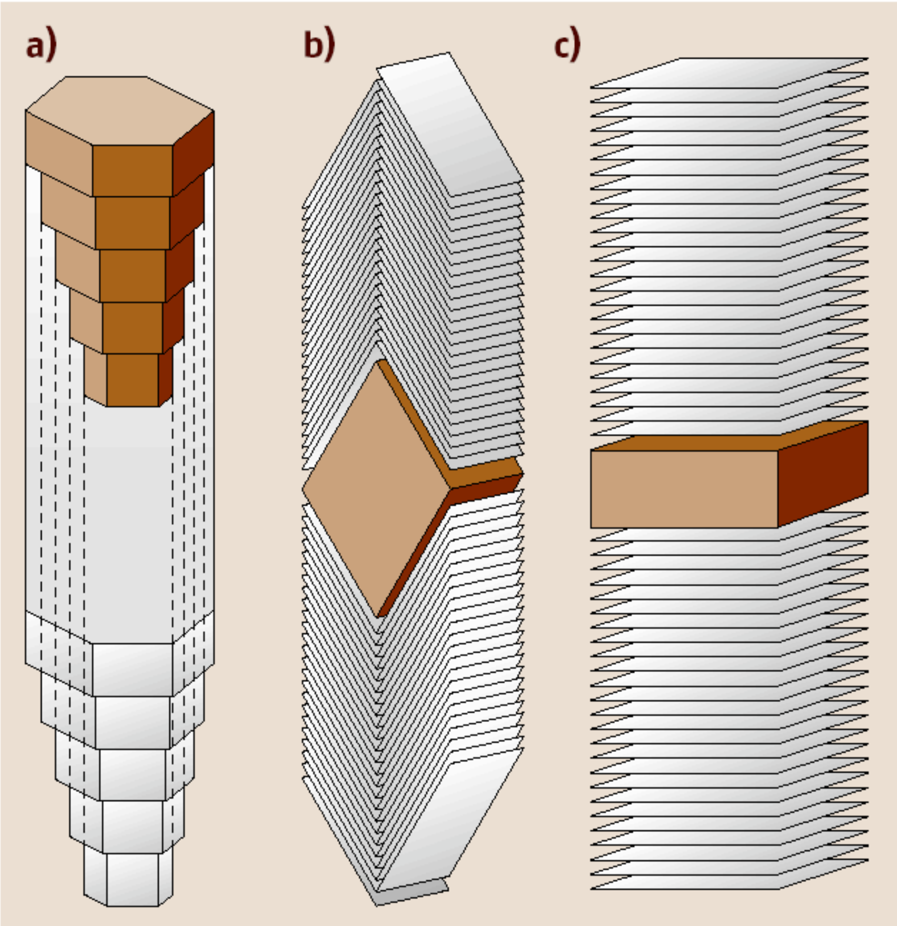
3.3.1. Catalyst-Free Growth

The growth of c-MWNT as a deposit onto the cathode in the electric-arc method is a rare example of catalyst-free carbon nanofilament growth.

3.3.2. Catalytically activated growth

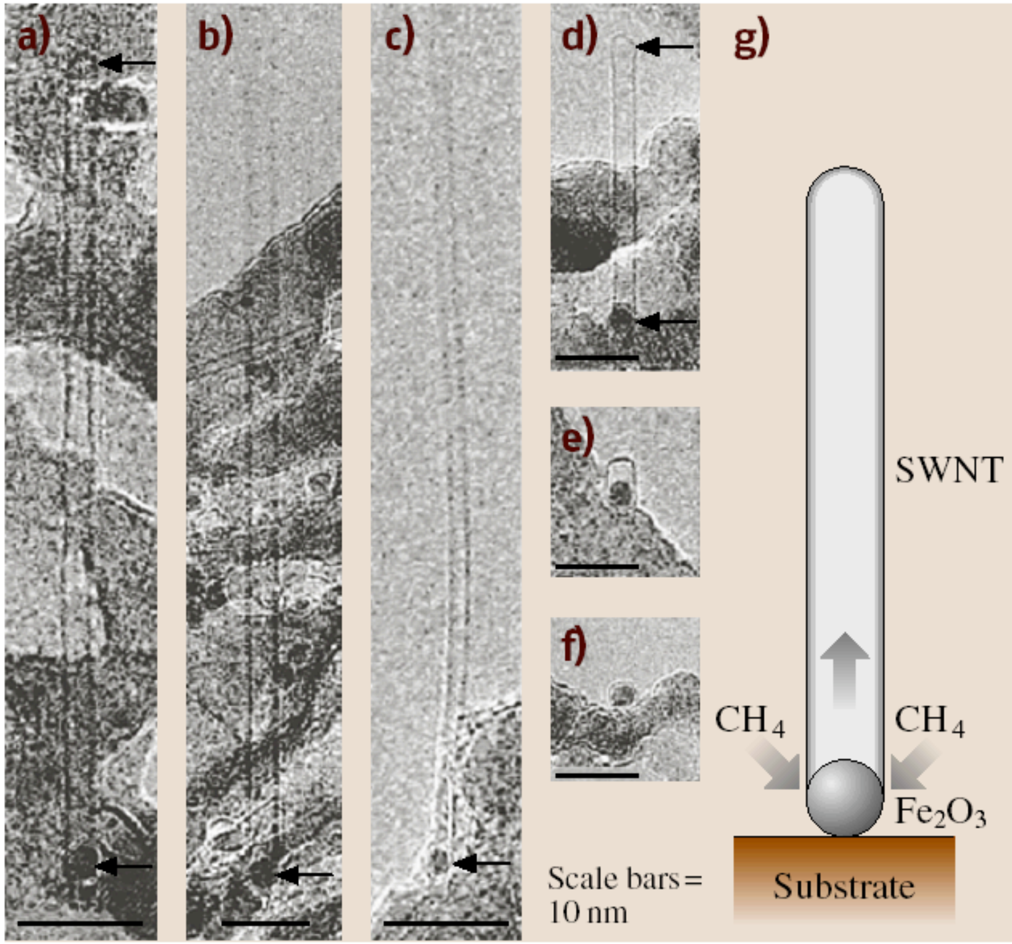
- The most important parameters are probably the thermodynamic conditions, the catalyst particle size, and the presence of a substrate.
- Two different techniques:
  - Low temperature conditions (below 1000°C)
  - High temperature conditions (several thousands of °C)

Low temperature conditions



- CCVD method
- 1 catalyst particle = 1 nanofilament
- Control of the catalyst particle size is critical.
- SMNTs: catalyst particles need to be smaller than 2nm.

Low temperature conditions

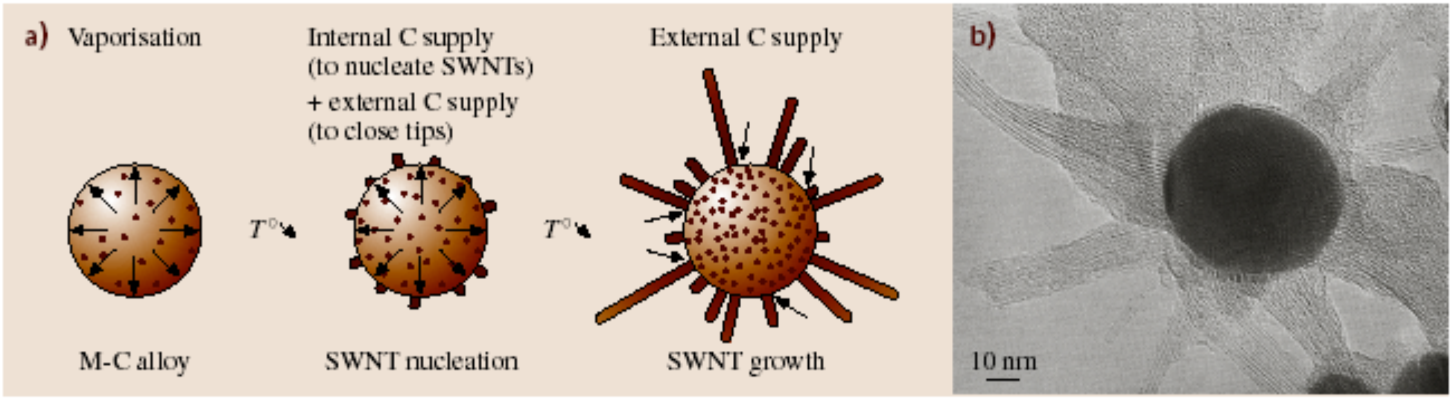


The particle size determines the SWNT diameter.

Low temperature conditions

		Increasing temperature . . . → . . . and physical state of catalyst			Substrate		Thermal gradient	
		Solid (crystallized)	**Liquid from melting**	**Liquid from clusters**	**Yes**	**No**	**Low**	**High**
Catalyst particle size	$<\sim$ **3** nm	SWNT	SWNT	?	base-growth	tip-growth	long length	short length
	$>\sim$ **3** nm	MWNT (c,h,b) platelet nanofiber	c-MWNT	SWNT	tip-growth			
Nanotube diameter		(heterogeneous related to catalyst particle size)		homogeneous (independent) from particle size)				
Nanotube/particle		one nanotube/particle		several SWNTs/particle				

High temperature conditions



- Electric arc, laser vaporization, solar furnace.
- 1 catalyst particle = many nanofilaments
- catalyst particle size 10-50nm.
- SMNTs: catalyst particles need to be smaller than 2nm.
- The nanotubes are shorter than from the CCVD technique, but the SWNT diameters are much more homogenous.
- Most frequent SWNT diameter: 1.4nm (seems to be the best energy compromise).

3.4. Properties of carbon nanotubes

- Carbon is a unique light atom that can form one, two-, or threefold strong chemical bonds.
- Properties of the carbon nanotubes may change drastically depending on whether SWNTs or MWNTs are considered.
- MWNTs are generally not much different from that of regular polyaromatic solids (polyaromatic solids are phases built with stacked graphenes instead of single graphenes).

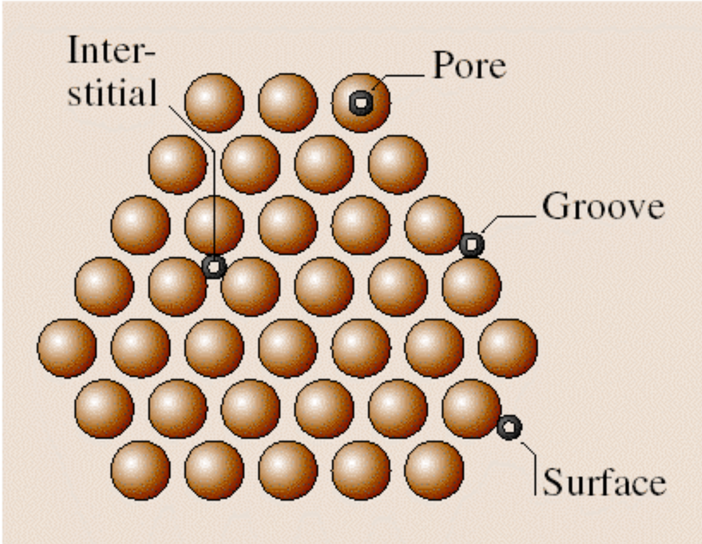
MWNT properties

- In stacked graphenes, the bond strength is quite different depending on whether the in-plane direction or the direction perpendicular to it is considered:
  - in-plane: very strong covalent bonds, only 0.142nm bond length
  - perpendicular: very weak van der Waals bonds, ~0.34 nm

SWNT properties

- SWNTs properties may change dramatically, depending on whether they are ropes or single tubes.
- SWNTs are narrower in diameter than the thinnest line able to be obtained by electron beam lithography.
- Stable up to 750°C in air, up to 1800°C in inert atmosphere (above: polyaromatic solids).
- In stacked graphenes, the bond strength is quite different depending on whether the in-plane direction or the direction perpendicular to it is considered:
  – in-plane: very strong covalent bonds, only 0.142nm bond length
  – perpendicular: very weak van der Waals bonds, ~0.34 nm

SWNT adhesion properties



- SWNTs have a very high surface area (the highest ever).
- Isolated SWNTs, with one end open, have a surface area of 2700m^2/g (accounting for both sides).
- 4 different binding sites.
- Binding energy 75% higher than on single graphene.

SWNT transport properties

- SWNTs are ideal quantum wires.
- Metallic SWNTs can transport huge current densities (max. 10^9 A/cm^2) without being damaged, i. e. about three orders of magnitude higher than in Cu.
- SWNTs are model systems to study one-dimensional charge transport phenomena.
- Visible light is strongly absorbed. It has been observed that flash illumination with a broadband light can lead to spontaneous burning of a macroscopic sample of agglomerated (i. e., ropes) carbon nanotubes in air and room temperature.
- SWNT thermal conductivitiy is comparable to that of high purity diamond.

SWNT mechanical properties

- The particularly strong threefolded bonding (*sp*2 hybridization of the atomic orbitals) of the curved graphene sheet is stronger than in diamond (*sp*3 hybridization) as revealed by their difference in C-C bond length (0.142 vs. 0.154 nm for graphene and diamond respectively).
- The tensile strength of SWNT is 20 times that of steel. It is even higher for MWNT.
- Defect-free carbon nanotubes could revolutionize the current panel of high performance fibrous materials.