Tensile Test of Carbon Nanotube using Manipulator in Scanning Electron Microscope

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Outline of talk

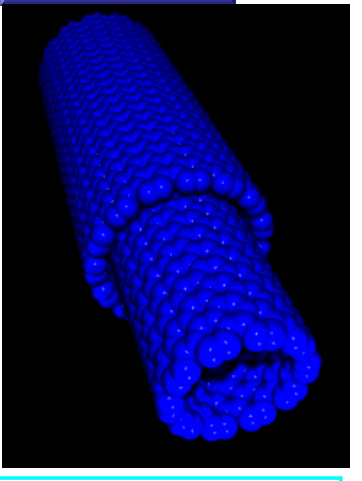
- Science fiction or not
- What is carbon nanotube?
- Set-up for tensile test
- Gripping for tensile test
- Tensile test of MWCNT
  - Fracture surface
- Conclusion

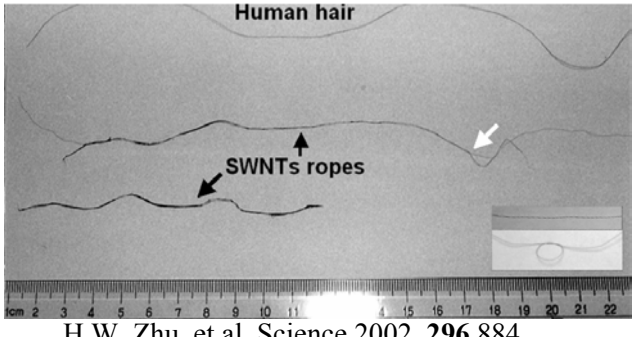
Science Fiction or Not

An elevator to space

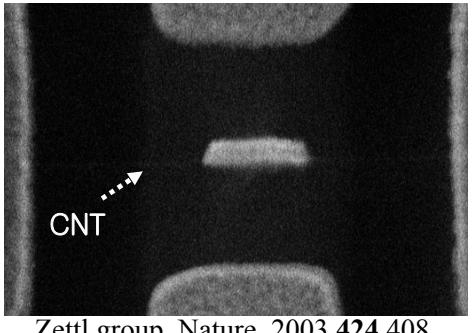
Tether propulsion

Nano-motor





H.W. Zhu, et.al. Science 2002, **296**,884



Zettl group, Nature, 2003,**424**,408

- A possible future material would be carbon nanotubes, which have a theoretical strength of at least 60 GPa.
- Carbon nanotubes are essentially sheets of graphite seamlessly rolled in to long tubes that are mere nanometers in diameter. These are 100 times as strong as steel, but much lighter.

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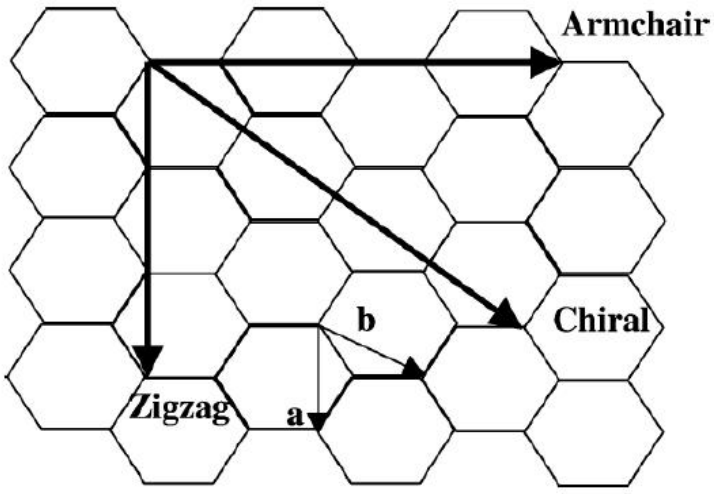
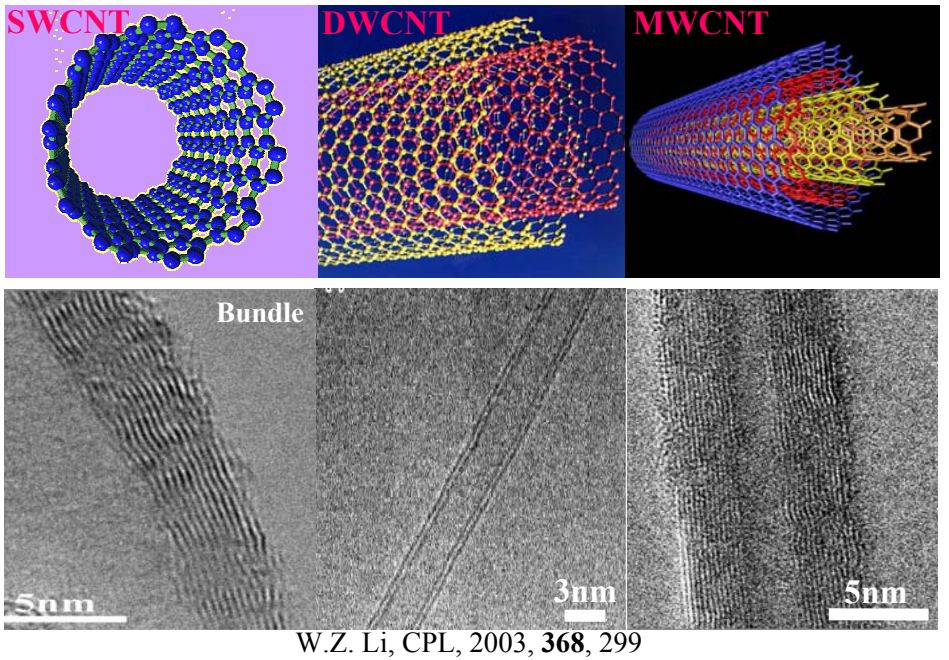
What are Carbon Nanotubes?

- ❖ Discovery 1991 S. Ijima
- ❖ Tubules of closed graphene sheets
- ❖ Like whiskers, are single crystals of high aspect ratio which contain only a few defects
  → excellent mechanical properties to CNT
- ❖ The secret is in the intrinsic strength of the carbon – carbon sp^2 bond
- ❖ For a tube (n,m) there is a rule: If (n-m) = 3 then the tube is metallic, else semiconducting



$C_h = an + bm = (n,m)$



W.Z. Li, CPL, 2003, **368**, 299

Mechanical Properties

- The research teams of Princeton and Illinois in USA measured that the average Young's modulus is 1.8 TPa in 1996.

- In 1997 Goddard showed various Young's Moduluses of nanotubes:
  (10,10) armchair nanotube 640.30 GPa,
  (17,0) zizag nanotube 648.43 GPa and
  (12,6) chiral nanotube 673,93 GPa.
  -These values were calculated from two dimensional differential coefficient of potential and it is shown that they considerably differ from the 1.8 TPa calculated above.

- In 1998, Treaty group reported the elastic modulus is 1.25 TPa and it is comparable to 1.28 TPa which was observed by Wong et. al. in MWNT in 1997.

- Elastic modulus and strength of MWCNT measured by direct tension in a TEM (Demczyk et al., 2002)

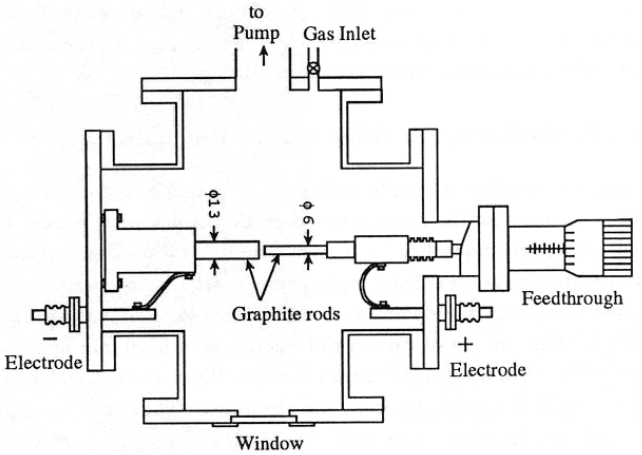
E (TPa)	σ_f (GPa)	F_f (μN)	Tube diameter (nm)
0.91 (±0.18)	150(±45)	18	12.5

- More recent results of MD yield 1.24 to 1.35 TPa for the young modulus (Jin and Yuan, 2003) for SWCNT.

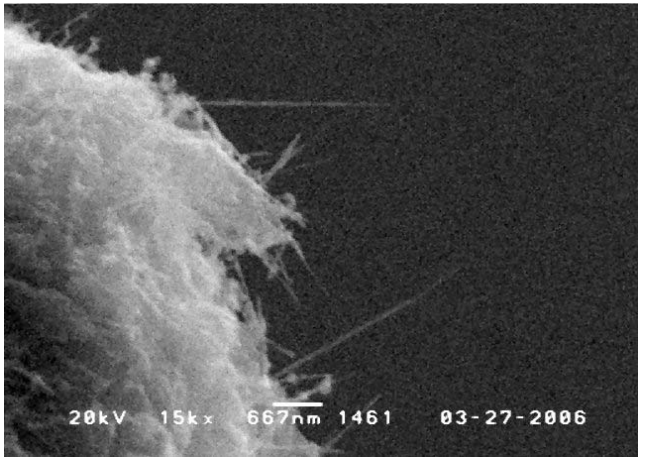
Multi Walled Carbon Nanotube

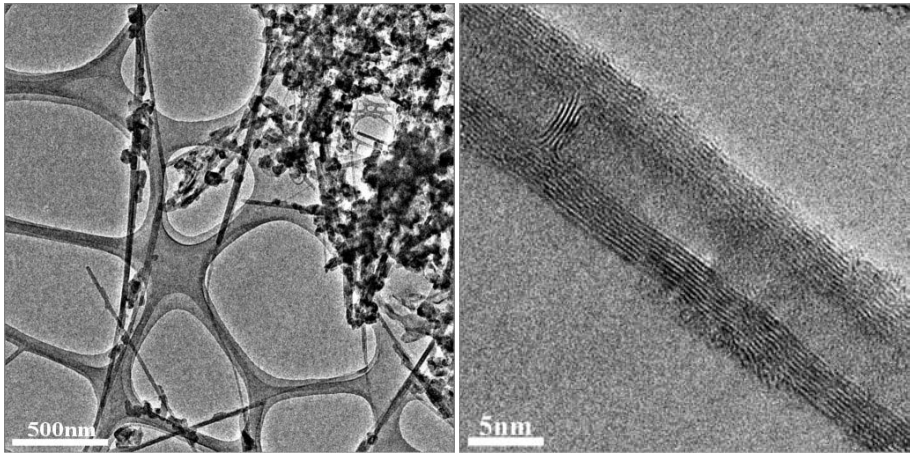
Arc-discharged MWCNT

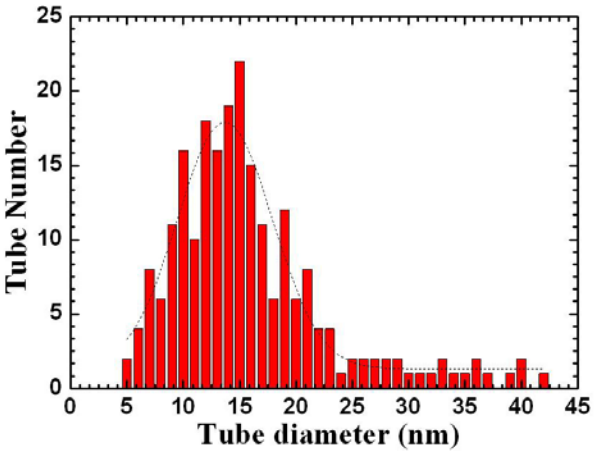
❖ The multi walled carbon nanotubes(MWCNT) were produced by arc-discharged method.



Saito et. al, J. Appli. Phys. 80, 3062(1196)

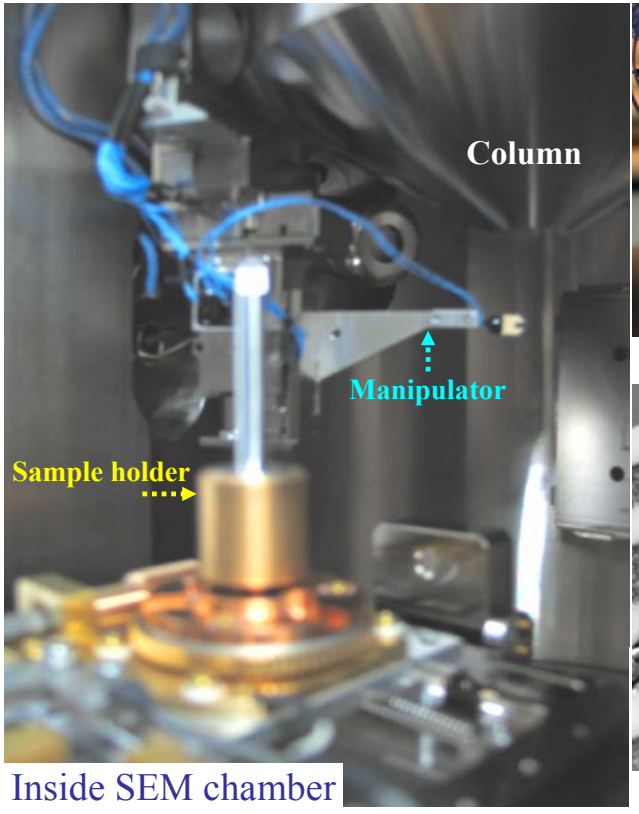




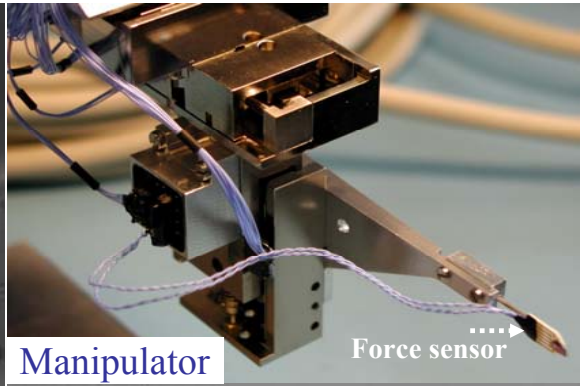


▪ The average of tube diameter is about 15nm

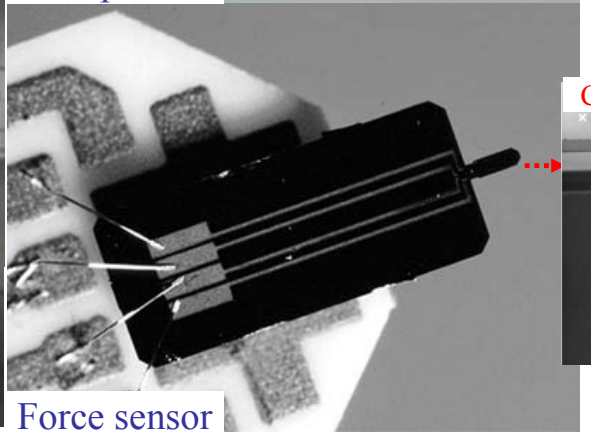
Set-up for Tensile test



Inside SEM chamber



Manipulator

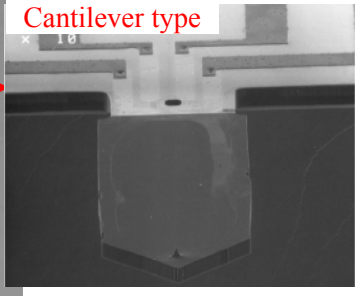

Force sensor

- **Nano-Manipulator**

Resolution : 1~2 nm
Operating range :120mm(x,y,x)

- **Force sensor**

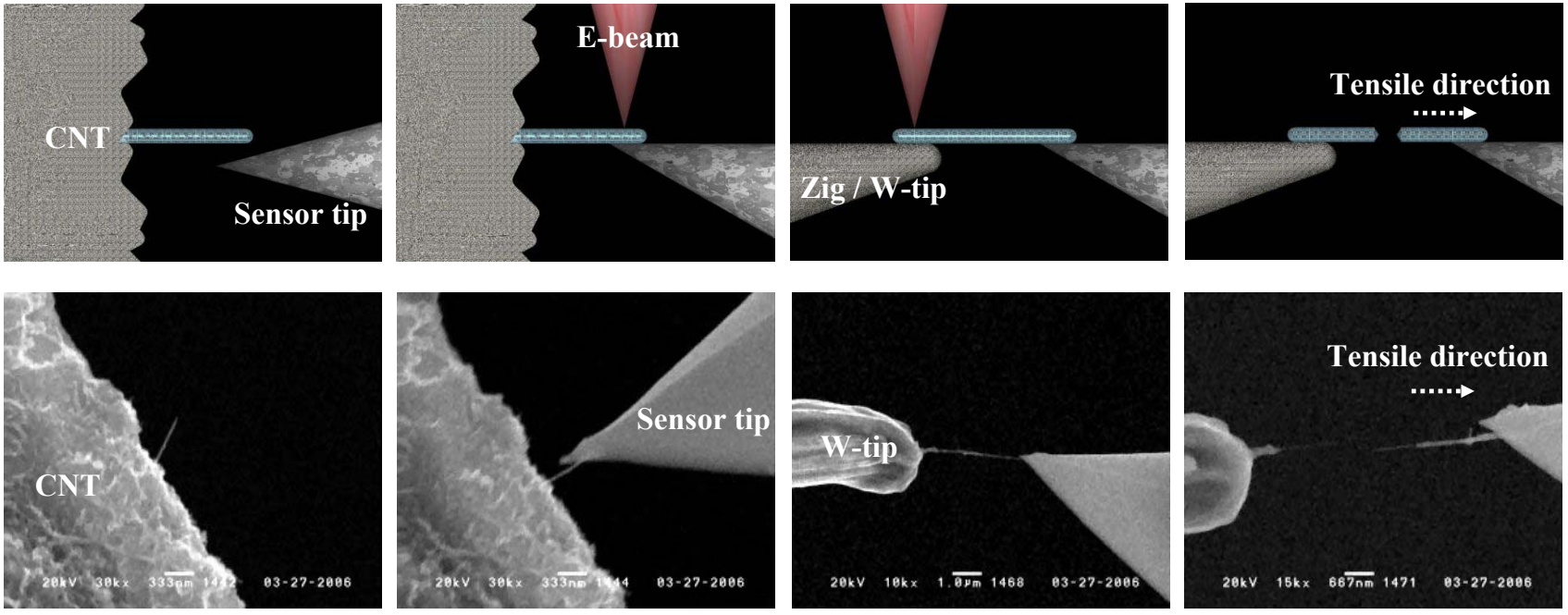
Type : cantilever
Resolution : sub nano-Newton
Force range : up to 1 mN


Cantilever type

- ❖ The force sensor tip is piezo-resistive cantilever mounted on a PCB support.
- ❖ The force sensor is inserted into the front of the nano-manipulator.
- ❖ The force sensor is controlled by nano-manipulator and personal computer.

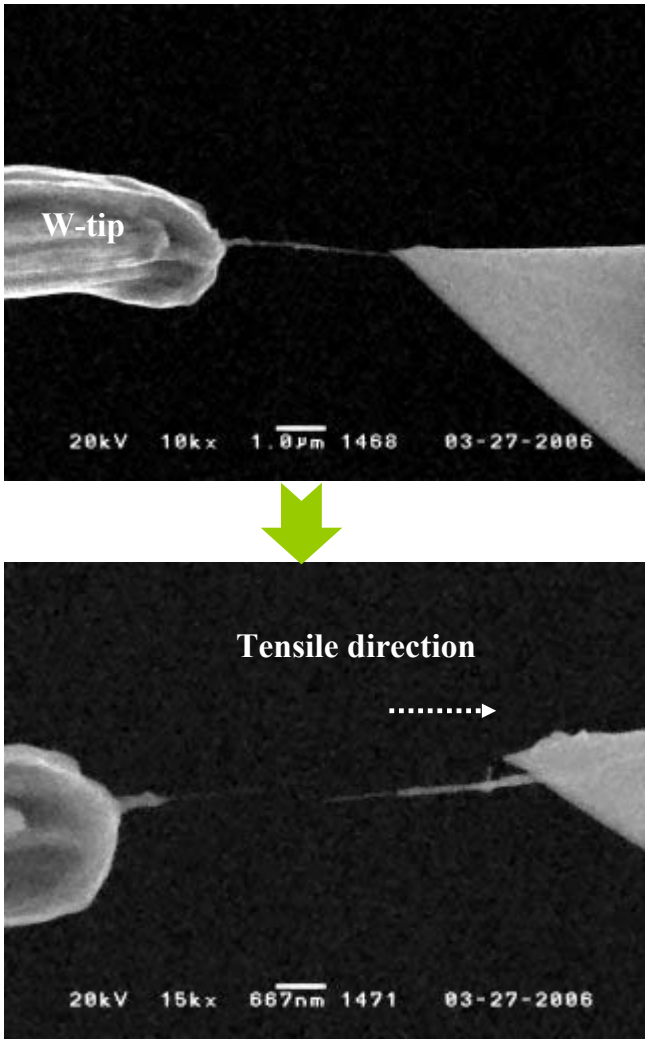
Gripping for Tensile Test

Griping and tensile test procedure

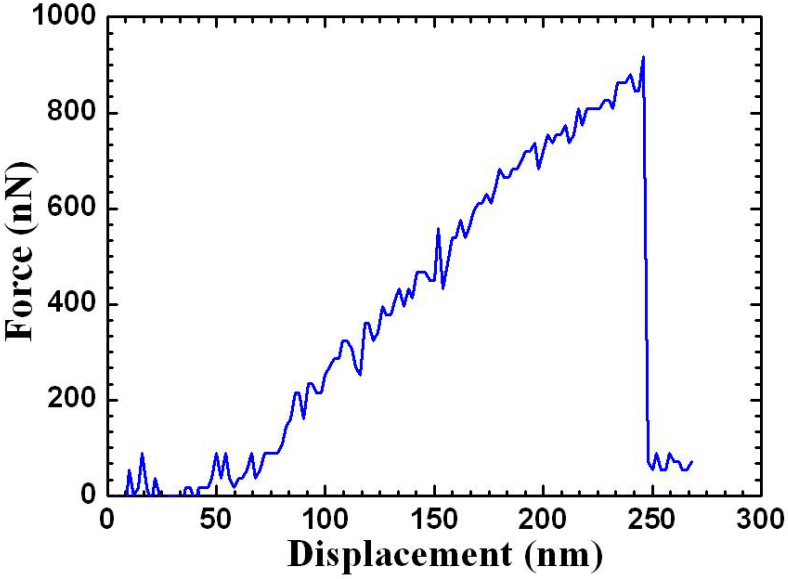


- The sensor tip and/or W-tip gripped an MWNT by electron beam (E-beam) induced deposition.
- The focused E-beam in a SEM can be used to deposit a small amount of hydrocarbon contamination so as to attach the MWCNT on an sensor tip and W-tip
- This nano-welding technique has been used to manipulate CNTs inside a SEM and to carry out mechanical testing. (MinFeng Yu, et.al. Nanotechnology, 1999, **10**, 244)

Tensile Test of Carbon Nanotube



Force-Displacement curve



- Tensile load : 916 nN
- Displacement of MWCNT : 197.7 nm

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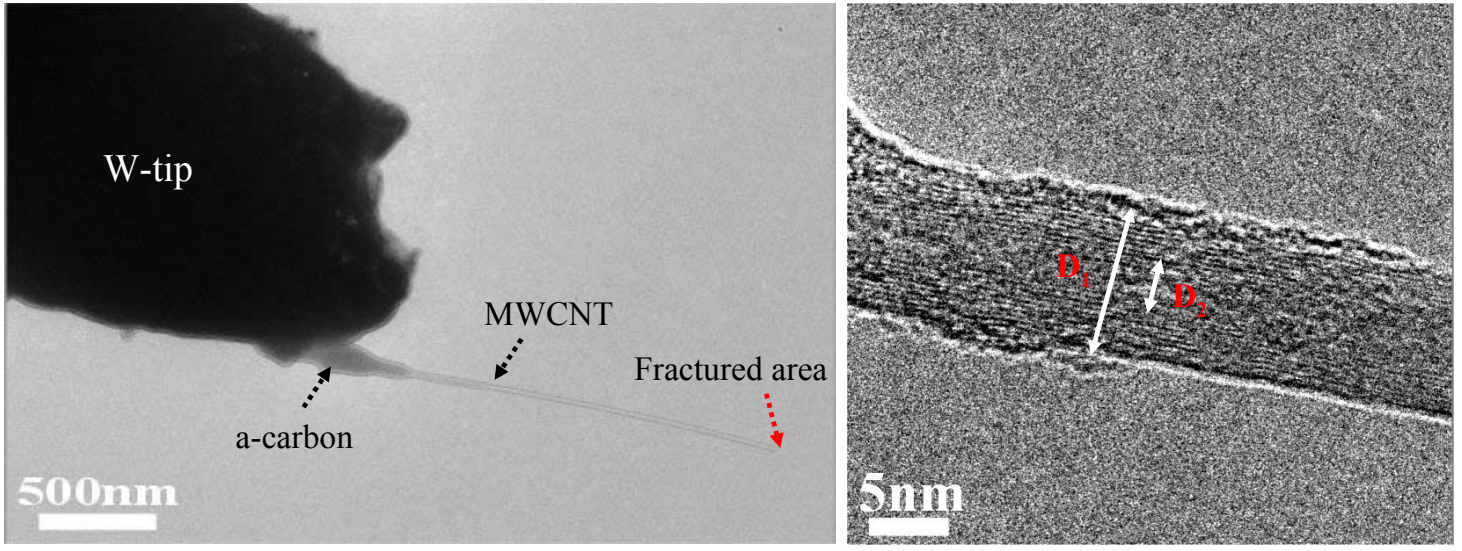
Fractured Surface of Carbon Nanotube

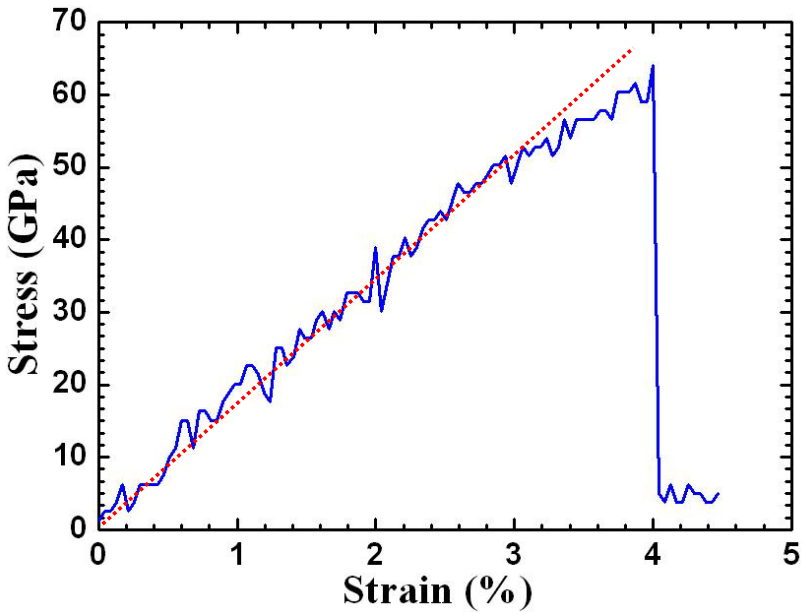
- The fractured surface of MWCNT was observed by transmission electron microscope (TEM).



- The MWCNT tip could not be imaged in high resolution mode, probably due to vibration.
- The vibration of the tube end was occurred owing to the influence of the electron beam.
- The fractured area of MWCNT is considered to be in the D_1-D_2 region
- Outer diameter of MWCNT is 8.6 nm and inner diameter is 2.4 nm. The layers are 9.
- The cross-section area of D_1-D_2 region was calculated by 14.29 nm^2.

Elastic Modulus of Carbon Nanotube

Stress-Strain curve



- Tensile strength of the MWCNT : 64.13 GPa
- The tensile elongation of MWCNT : 4.2 %

❖ In order to calculate of elastic modulus, the deflection of sensor cantilever must be considered, but the deflection of cantilever can be ignored because of infinitesimality. (the value of deflection is 5.5 pm)

❖ The elastic modulus of MWCNT : 1.53 TPa

❖ MWCNT used in this study is assumed to be an excellent elastic material.

Conclusion

- Tensile test of an individual MWCNT was carried out using force sensor inside SEM.
- Fracture surface of MWCNT was observed by TEM. The calculated fractured area was about 14.29nm^2 from the result of TEM analysis.
- The elastic modulus of MWCNT was 1.53 TPa.

Thank you for listening !