

NANO 703/703L
 Lab: TEM Analysis of Carbon Nanotubes
 Due: Following Lab Session

BACKGROUND

Carbon nanotubes (NTs) are among the most versatile and widely explored nanomaterials systems. NTs offer exceptional mechanical strength, high surface area to volume, and variable electrical and thermal conduction properties. NTs have also been used as scaffolding templates for the organization of semiconductor nanoparticles into linear arrays. Carbon NTs are synthesized by methods such as chemical vapor deposition, laser ablation, and electric-arc discharge.

The NT structure is built on graphene sheets rolled into seamless, closed cylinders. An infinite graphene sheet can be described by the pair of basis vectors:

$$\mathbf{a}_1 = a\hat{\mathbf{x}}, \quad \mathbf{a}_2 = -\frac{1}{2}a\hat{\mathbf{x}} + \frac{\sqrt{3}}{2}a\hat{\mathbf{y}}$$

These form a triangular array, with a lattice constant of approximately $a = 0.245 \text{ nm}$. The unit cell contains two C atoms at positions:

$$\mathbf{d}_1 = \mathbf{0}, \quad \mathbf{d}_2 = \frac{2}{3}\mathbf{a}_1 + \frac{1}{3}\mathbf{a}_2,$$

forming a network of open hexagons.

The cylinder curvature of a NT can be described by the perimeter (roll-up) lattice vector $\mathbf{C} = u\mathbf{a}_1 + v\mathbf{a}_2$, denoted by its indices $[u,v]$. The NT circumference is then:

$$C = a\sqrt{u^2 + v^2 - uv}$$

The corresponding diameter is $d = C/\pi$. Single-walled NTs are observed with diameters as small as 0.4 nm.

EXPERIMENT

This lab will be a cursory examination of NTs from different manufacturers. Samples will be prepared by depositing NTs in solution onto C-coated TEM grids. Ultrathin and holey C-coated grids are also available. Colloidal graphite will be examined for comparison with NTs.

\mathbf{C} is related to the helical (chiral) angle α , where:

$$\mathbf{C} \cdot \mathbf{a}_1 = C \cdot a \cdot \cos \alpha$$

NTs with $\alpha = 0$ are referred to as having “zig-zag” helicity; NTs with $\alpha = 30^\circ$ have “armchair” helicity.

A translation vector \mathbf{T} , of indefinite length, extends along the NT axis, normal to \mathbf{C} .

Just as multiple graphene sheets stack to form a graphite crystal, single-walled NTs can assemble into a multiwalled NT. The third axis in the bulk graphite structure has basis vector:

$$\mathbf{a}_3 = c\hat{\mathbf{z}}$$

where the interlayer spacing has a nominal value of $c/2 = 0.34 \text{ nm}$, which is sensitive to the fabrication method and history of the material.

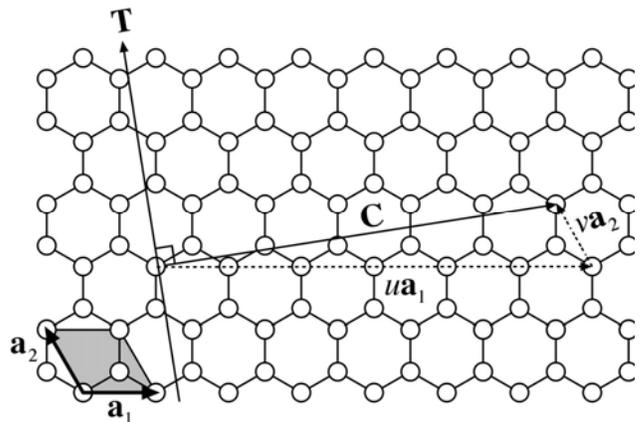


Fig.: Graphene sheet. The basis lattice vectors are labeled. A possible perimeter vector $[6,1]$ and the corresponding components are shown.

- 1) Acquire the following data:
 - a) Bright-field images of graphite and NTs from different sources. If possible, obtain images showing fringes corresponding to the concentric tubes within multiwalled NTs.
 - b) Selected-area diffraction patterns from:
 - i) a high-density ensemble of NTs;
 - ii) colloidal graphite.
- 2) Measure the following quantities:
 - a) The inner diameter $2R_1$ of a multiwalled NT.
 - b) The outer diameter $2R_2$ of the multiwalled NT (not including any encapsulating medium.)
 - c) The number of tubules n within a particular multiwalled NT (if resolvable).
 - d) The d-spacing from an FFT of an image showing a multiwalled NT.
 - e) The d-spacings corresponding to the first few diffraction rings.

ANALYSIS

- 1) If NT walls are resolvable, compute the layer spacing of the multiwalled NT from the image data, using:

$$c/2 = (R_2 - R_1)/n$$

Otherwise, estimate the number of walls using:

$$n = (R_2 - R_1)/(c/2)$$

Clearly state which calculation you have performed.

- 2) Compare $c/2$ to the d-spacing determined from the first diffraction ring.
- 3) Establish any differences or similarities between diffraction from graphite and NTs.

REPORT

Please submit documentation of work including results of measurements, calculations, and representative images.

REFERENCES

- [1] S. Iijima, *Nature* **354** (1991) p. 56.
- [2] Electron Microscopy of Nanotubes, Ed. Z. L. Wang & C. Hui, Kluwer, Boston, (2003).