A Detailed TEM Study of Carbon Nano-Onions' Structure



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Introduction

Carbon Nano-Onions (CNOs)

- Polyhedral carbon nanoshells
- Fabrication by graphitization
- Applications include lubrication, catalyst support, conductive filler

Transmission Electron Microscopy (TEM)

- TEM analyses provide crystalline structure and carbon bonding information
- Techniques include:
 - Bright-field & Dark-field imaging
 - Selected area electron diffraction (SAED)
 - Electron energy loss spectroscopy (EELS)
- We expand these using image analysis algorithms to quantify crystallite parameters evident in bright field imaging, and by tomography, for morphology and dimensionality

Bright and Dark Field

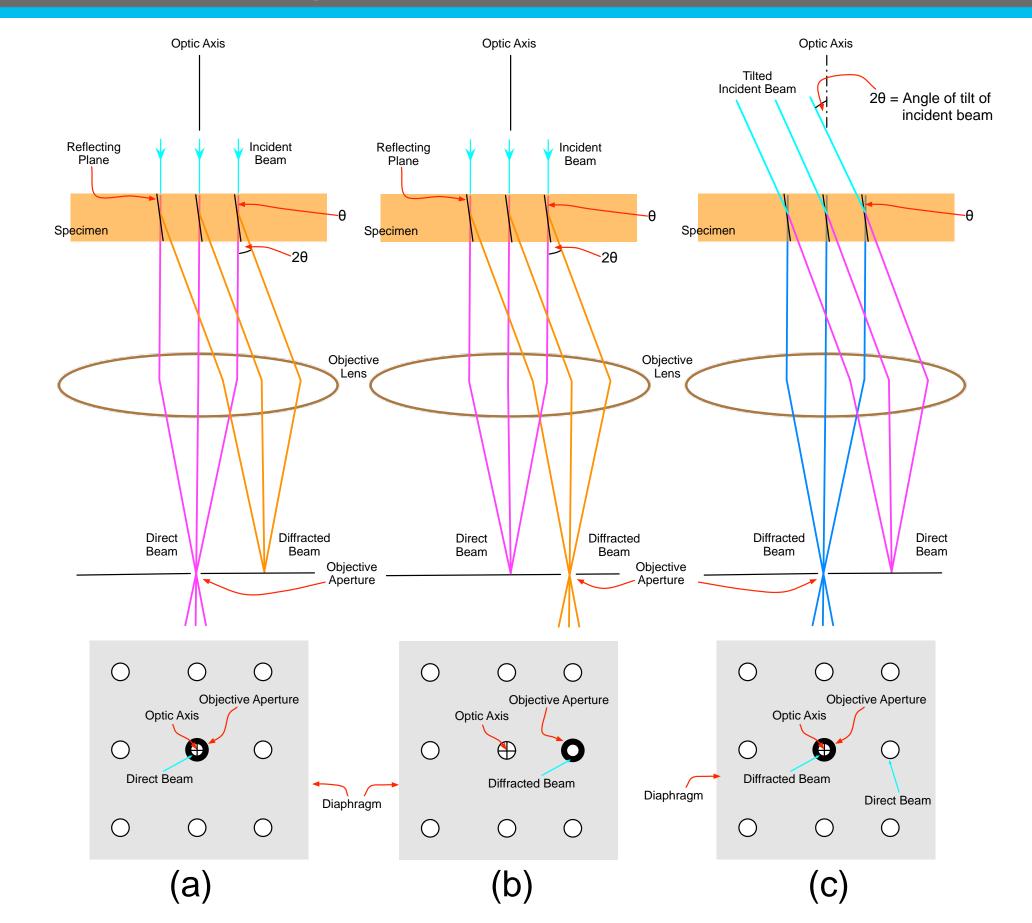


Figure 1. Ray diagrams showing how the objective lens and objective aperture are used in combination to produce (a) a Bright Field imaging, (b) off-axis Dark Field, and (c) on-axis Dark Field

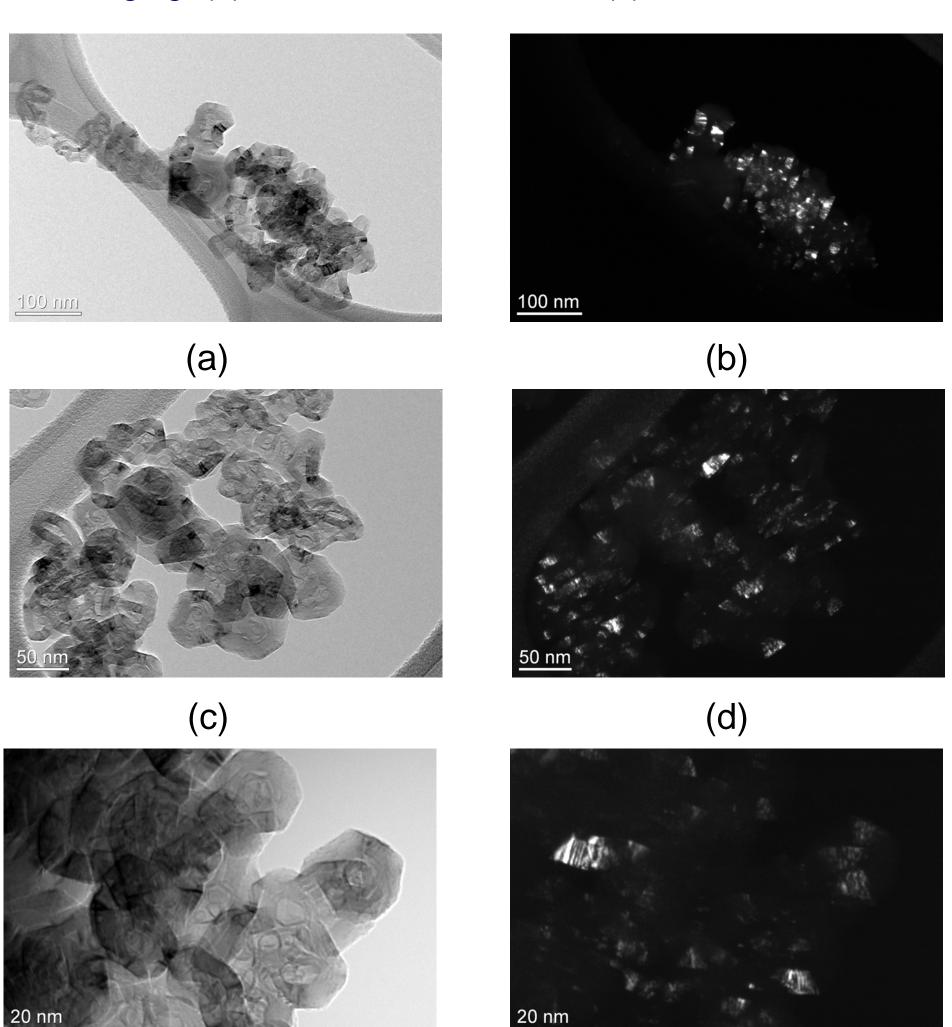


Figure 2. Bright and Dark Field TEM images of CNOs at different magnifications (a)–(b) 40,000x, (c)–(d) 60,000x, and (e)–(f) 100,000x. Dark field images were acquired using the (002) diffraction

(e)

Incident Angle Angle Atomic Layer Planes

 $n\lambda = 2d \sin\theta$ Figure 3. Illustration of Bragg diffraction from atomic layer planes

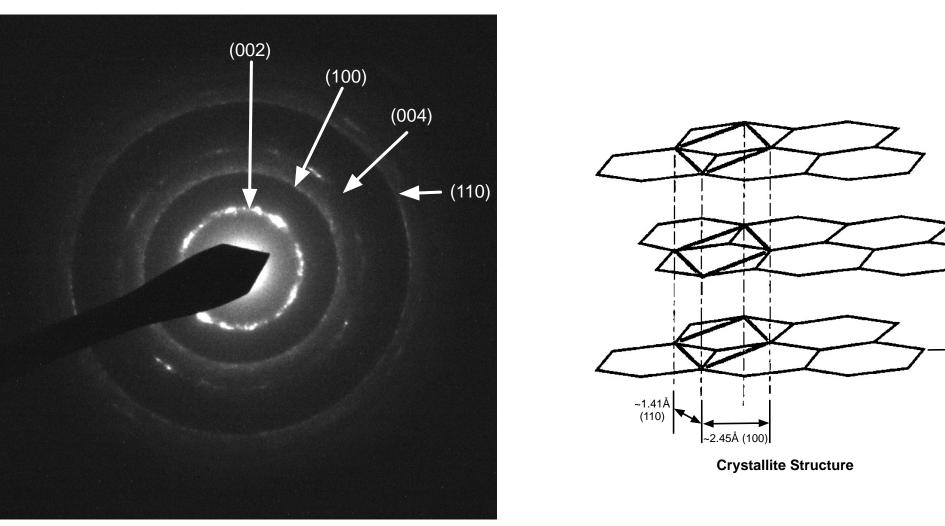
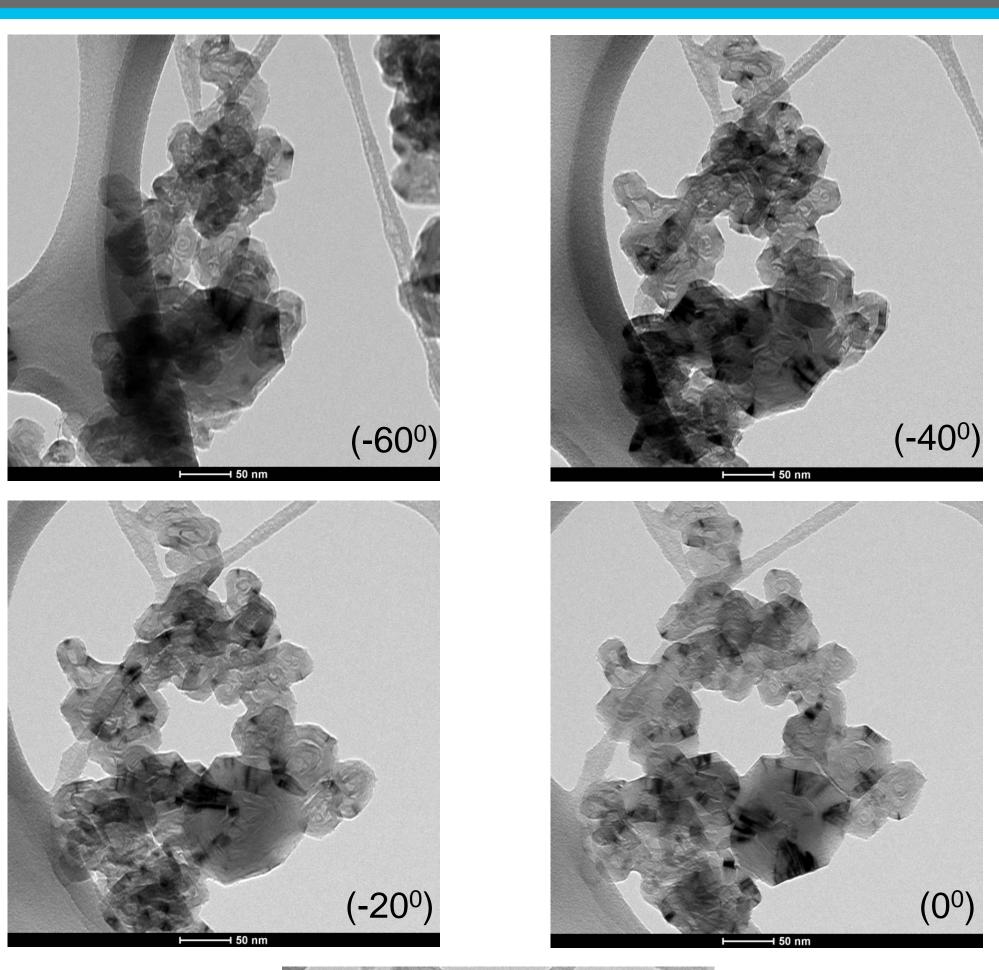
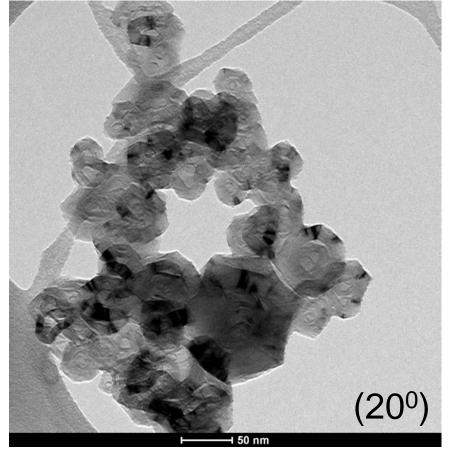


Figure 4. SAED pattern of CNOs. the pattern shows diffraction rings corresponding to graphite (002), (100), (004), and (110) layer planes, respectively.

Tomography





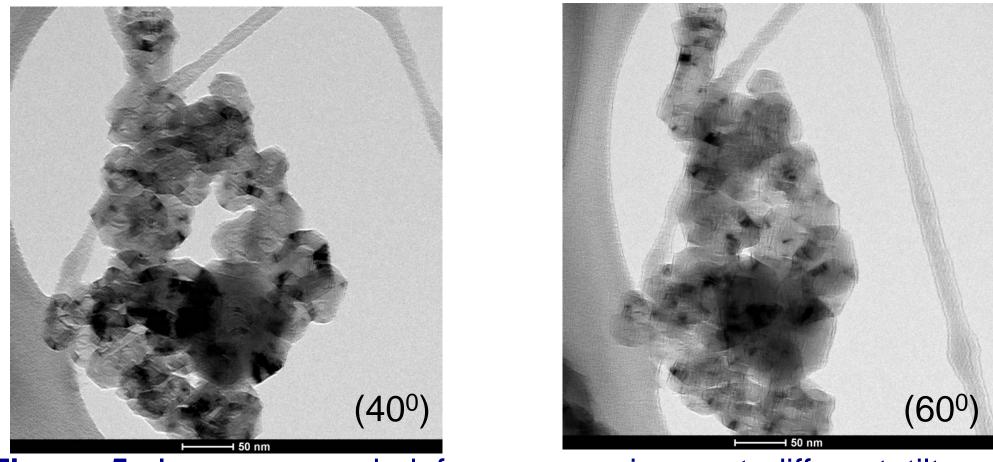


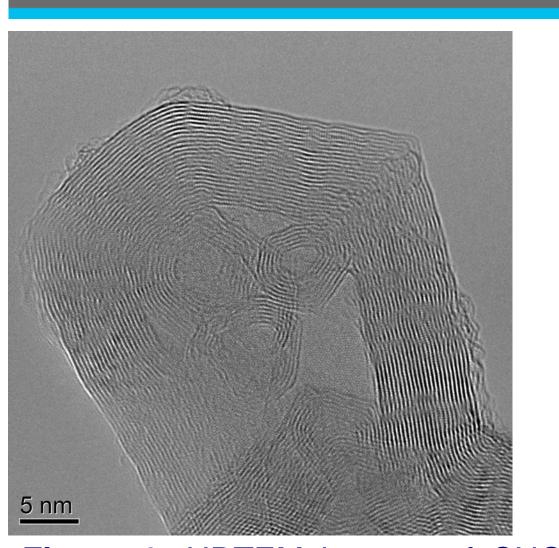
Figure 5. Images recorded from a specimen at different tilt-angle positions (as labeled)

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HRTEM & Fringe Analysis



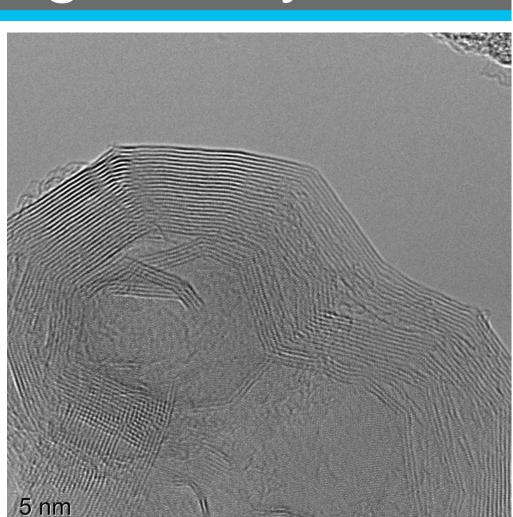


Figure 6. HRTEM images of CNOs acquired at a magnification of 500,000x

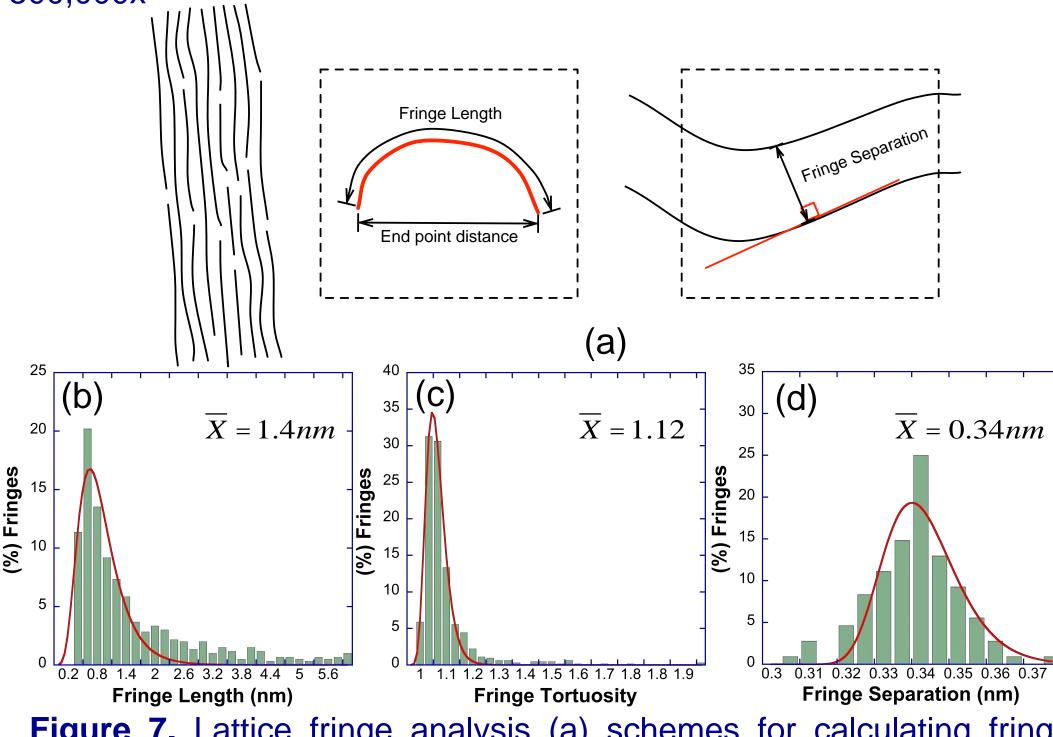


Figure 7. Lattice fringe analysis (a) schemes for calculating fringe length, tortuosity and separation, (b)–(d) histograms and overlaid lognormal fits and parameterized mean values, respectively

FFI S

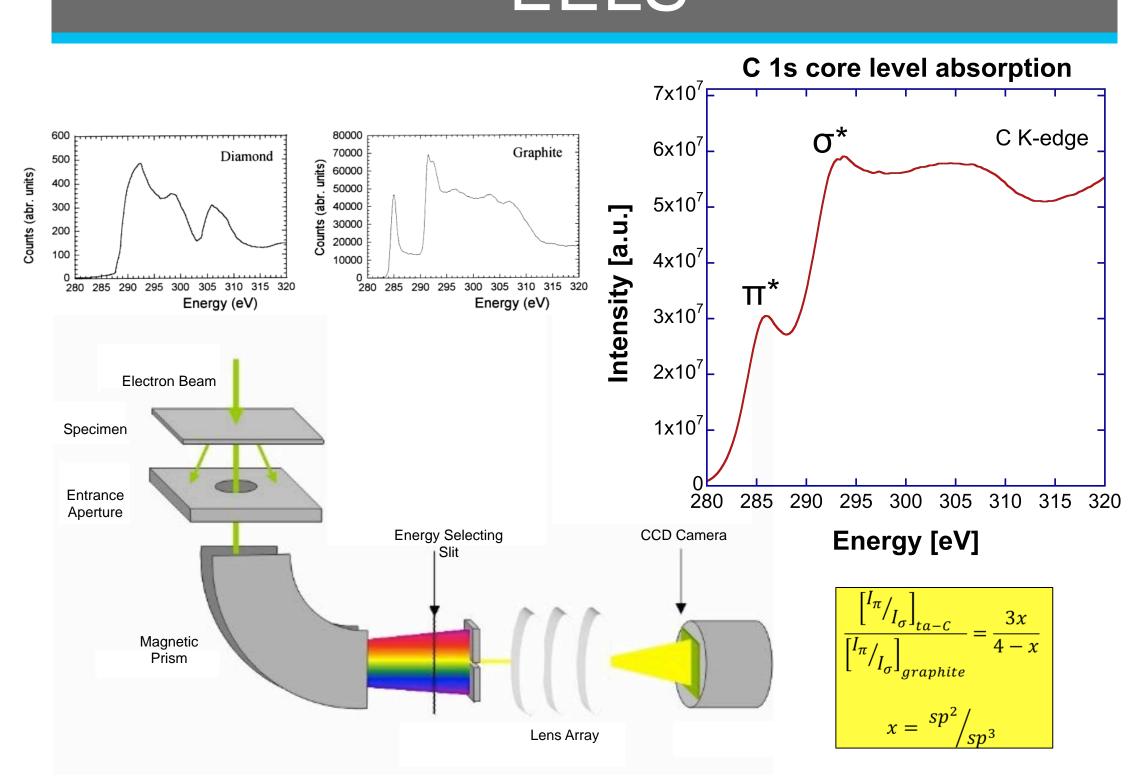


Figure 8. A schematic of the EELS and EFTEM experimental setup for a Gatan imaging filter (GIF)

Conclusions

- Bright field images show ordered lamella
- SAED "spotty" pattern demonstrates well developed crystal structure (along with lattice structure)
- Dark field shows that structure is spatially localized FELS indicates bonding is prodominantly sp²
- EELS indicates bonding is predominantly sp²
- Image analysis algorithms permit quantification of crystallite parameters
- Tomography confirms 3D nature of this novel carbon morphology

References

- 1 D. Haarto Naturo 350(1002)707-709
- D. Ugarte, Nature, 359(1992)707-708
 R. L. Vander Wal, Combust. Sci. and Tech., 126(1997)333-357
- 3. Y. Gao et al., Carbon, 51(2013)52-58
- F. Banhart et al., Nature, 382(1996)433-435
- F. Baimart et al., Nature, 362(1996)433-433
 W. A. de Heer et al., Chem. Phys. Letters, 207(1993)480-486
- D. Pech et al., Nature, 5(2010)651-654S. Tomita et al., Chem. Phys. Letters, 305(1999)225-229
- S. lijima, J. Crys. Growth, 50(1980)675-683
 K. Yehliu et al., Combust. Flame, 158(2011)1837-1851