DOI: 10.1017/S1431927605505300
Microsc Microanal 11(Suppl 2), 2005

Structure determination of isolated single-walled carbon nanotubes by electron diffraction and diffraction microscopy

T. Beetz*, L. Wu*, Y. Zhu*

* Center for Functional Nanomaterial, Brookhaven National Laboratory, Upton, NY 11973-5000

Iijima discovered carbon nanotubes (CNTs) in 1991 as a by-product of the preparation method to produce fullerenes [1]. The exceptional mechanical, electrical and chemical properties of CNTs offer significant advantages over many existing materials. The structure of a CNT determines its properties and therefore much work has been done on determining the exact structure. Here we report the unambigous structure determination of isolated single-walled CNTs that were previously studied using Rayleigh scattering to probe the electronic transitions [2].

Electron diffraction patterns of isolated single-walled CNTs were collected using a JEOL3000F electron microscope at a reduced operating voltage to minimize radiation damage. We used a parallel illumination with a diameter of about 60 nm as described by Gao *et al.* [3]. Diffraction patterns were recorded on Fuji image plates which offer a good dynamic range and no 'blooming' effect compared to CCD cameras. This acquisition technique results in very clear diffraction patterns as shown in FIG.1.b). To determine the chiral vector of the nanotube, the experimental diffraction patterns were fitted with simulated ones of nanotubes that have a diameter consistent within the diameter range determined at imaging conditions. FIG.1.c) shows the experimental diffraction pattern overlaid with the simulated diffraction pattern of a (27,11) tube, which is perfectly matched and the structure is also consistent when the method used by Gao *et al.* [3] is applied to the experimental data.

To further extend the sensitivity to local defects in CNTs and to image catalysts attached to CNTs we have used diffraction microscopy [4]. In diffraction microscopy, one has to make sure that the field of view is illuminated coherently and that the area surrounding the region of interest is empty. It is then possible to retrieve the phase, which is lost when the diffraction pattern (intensity) is recorded, using iterative algorithms [5],[6]. This method has yielded an atomic resolution image of a double-walled CNT by Zuo *et al.* [7].

We show results of this method applied to diffraction data of single-walled CNTs under the same experimental conditions described above. Phase retrieval was obtained using Elser's difference-map algorithm [6]. We further discuss the effects of the lenses, used to record the diffraction pattern, such as aberrations and distortions, and how they effect and limit the ultimate achievable resolution. To overcome these limitations we present diffraction microscopy experiments that were conducted without any post-specimen lenses. Using this experimental technique, we show that the achievable resolution is only limited by the coherent properties of the illumination incident on the specimen and ultimately by the radiation dose that can be accepted by the specimen before structural alterations take place.

[1] S. Iijima, Nature (UK) **vol.354, no.6348**, (1991).
[2] M. Y. Sfeir, F. Wang *et al.*, Science **306** (5701), (2004).
[3] M. Gao, J. M. Zuo *et al.*, Applied Physics Letters **82** (16), (2003).
[4] J. W. Miao, P. Charalambous *et al.*, Nature (UK) **400** (6742), (1999).
[5] J. R. Fienup, Opt. Lett. **3** (1), (1978).
[6] V. Elser, J. Opt. Soc. Am. A-Opt. Image Sci. Vis. **20** (1), (2003).

[7] J. M. Zuo, I. Vartanyants *et al.*, Science **300** (5624), (2003).
[8] We would like to thank M.Y. Sfeir, F. Wang, L. Huang, C.-C. Chuang, J. Hone, S.P. O'Brien, T.F. Heinz, L.E. Brus at Columbia University for the CNT sample and J.M. Zuo for helpful discussions. This work was funded by the U.S. Department of Energy under contract DE-AC02-98CH10886 and FG02 96ER45610.

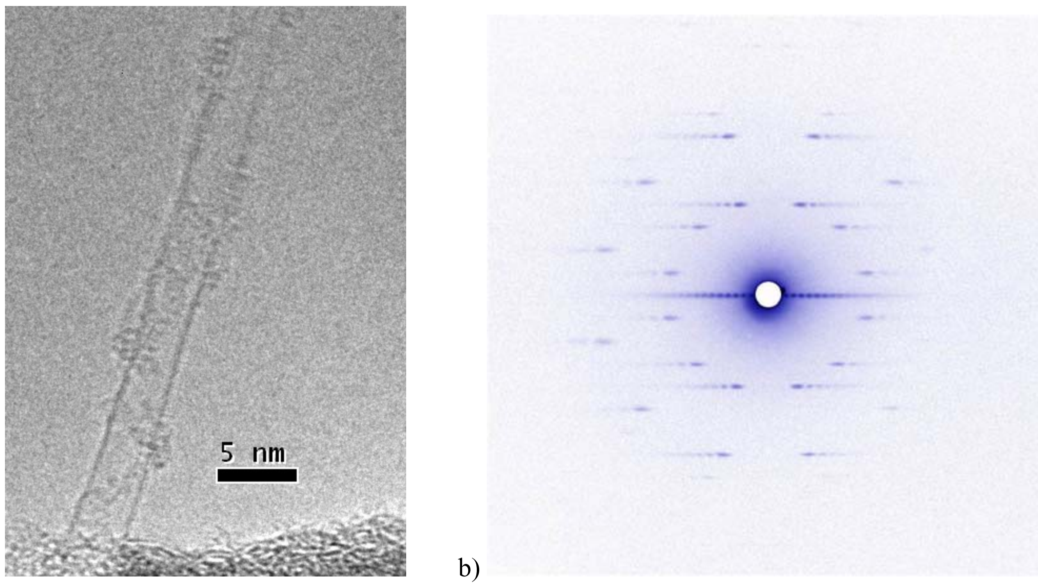

FIG.1. a) Experimental TEM image and b) electron diffraction pattern of an isolated single-walled CNT.

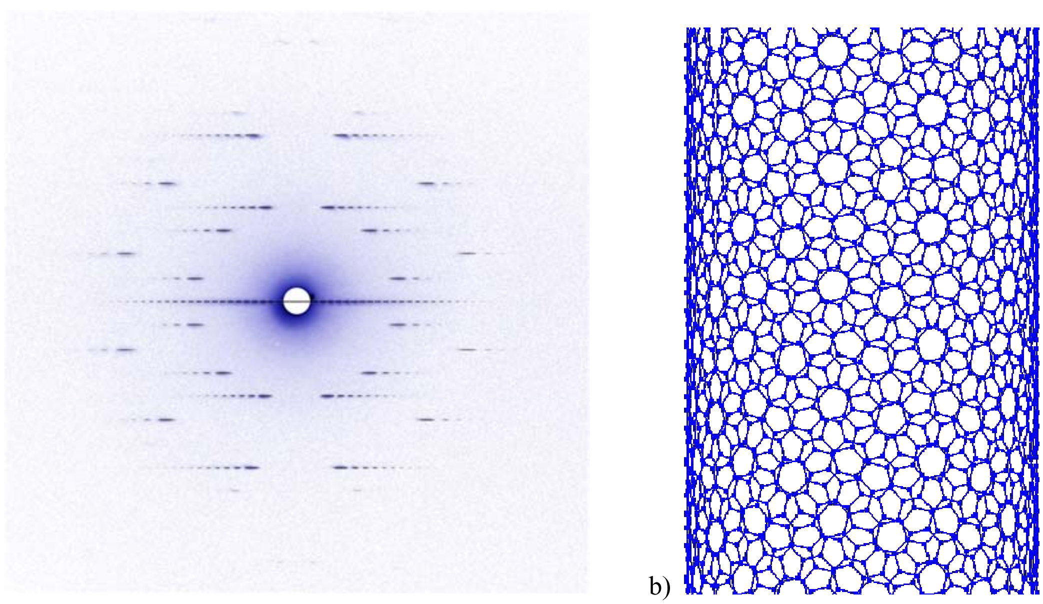

FIG.2. a) Experimental electron diffraction pattern (blue) overlaid on simulated electron diffraction pattern (black). The simulated diffraction pattern was generated from a CNT with chiral vector (27,11) corresponding to a diameter of 2.65 nm and a chiral angle of 16.34°. A structural model of this CNT is shown in b).

https://doi.org/10.1017/S1431927605505300 Published online by Cambridge University Press