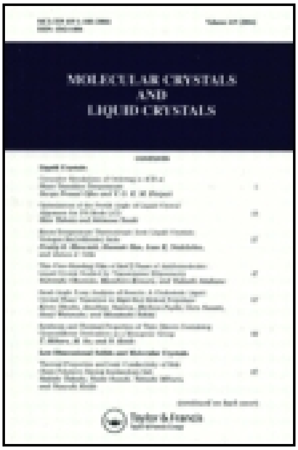

Molecular Crystals and Liquid Crystals
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/gmcl20

Structural analysis of carbon nanofiber by transmission electron microscopy and image processing

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Version of record first published: 18 Oct 2010

To cite this article: Kyoichi Oshida, Kozo Osawa, Tatsuo Nakazawa, Yasuo Fukai, Takuya Hayashi & Morinobu Endo (2002): Structural analysis of carbon nanofiber by transmission electron microscopy and image processing, Molecular Crystals and Liquid Crystals, 387:1, 145-150

To link to this article: http://dx.doi.org/10.1080/713738849

Mol. Cryst. Liq. Cryst., Vol. 387, pp. [369]/145–[374]/150

1058-725X/02 $12.00 + .00
DOI: 10.1080/10587250290113691

STRUCTURAL ANALYSIS OF CARBON NANOFIBER BY TRANSMISSION ELECTRON MICROSCOPY AND IMAGE PROCESSING

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Recently, the lattice image of well ordered carbons can be observed pretty easily by high-resolution transmission electron microscopy (HRTEM). In this study, the cross section of the new type carbon nanofiber, of which the special structure was unknown, was observed by HRTEM and the detailed structure was analyzed by image processing. Defects were observed in the ordered structure of this material, that is, the (00·1) lattice planes show large fluctuation in alignment of them and the (10·0) lattice planes make several angles with the (00·1) lattice planes. The average repeat distance d_{002} was estimated by HRTEM image and image analysis.

Keywords: carbon nanofiber; transmission electron microscopy; image analysis; first Fourier transform; power spectrum

INTRODUCTION

Wall structures of cup-stacking type carbon nanofibers [1,2], variations of carbon nanotubes, are different from that of the conventional multi wall carbon nanotubes [3] and nanofibers [4]. The layer planes of the former materials have an angle to the fiber axis, while those of the latter are parallel to the fiber axis.

Carbon nanotubes generally have a layer structure, which is formed parallel to the fiber axis. However, the layer stacking of the cup-stacking type nanofibers are slanted from the fiber axis, and many edge sites of stacking layers will exist at the sidewall of the tube. This new type nanofibers have possibilities of wide application because small molecules can be intercalated to the side wall. The nanofiber expected to be applied to the material for the cathode electrodes of lithium ion batteries and for the other energy storage system.

In this report, a partial structure of the nanofibers produced by the vapor growth method was investigated by high resolution transmission electron microscopy (HRTEM) combined with image processing [5,6].

EXPERIMENTAL

The lattice structure of the nanofibers was analyzed as follows.

1) Lattice images of the samples were observed by HRTEM (JEM2010FEF, 200 kV).
2) Power spectrum was obtained from the lattice images by means of 2-dimentional fast Fourier transform (2D-FFT) [7].
3) In order to verify the lattice structure, the real space images were reconstructed by 2-dimentional inverse fast Fourier transform (2D-IFFT), by using the selected area of the 2D-FFT data in correspondence to the specific frequencies [8,9].
4) Power spectrum features were analyzed quantitatively by using the graph of the relation between spacing and peak intensity, which is obtained by means of integration of the power spectrum around its central point.

RESULTS AND DISCUSION

TEM Observation

Figure 1 shows the low magnification (×10,000) TEM image of the hollow shape of the cup-stacking type carbon nanofibers with diameters of around 100 nm. The lattice image of them were observed by HRTEM with a magnification of ×400,000 is shown in Figure 2(a). The right part in the image is the cross section of the wall of nanofiber, where (00·1) lattice planes are slanted from the fiber axis, and the left part is the hollow region. The enlarged image of the region enclosed by square in Figure 2(a) is shown in Figure 2(b). The short lines, which appear in between the (00·1) lattice planes, are presumed to correspond to (10·0) lattice planes.

Image Analysis

The power spectrum shown in Figure 2(c) was obtained by means of 2D-FFT. The spectrum is symmetrical about the center point. The bright parts labeled A and A′, which are thought correspond to (10·0) lattice planes,

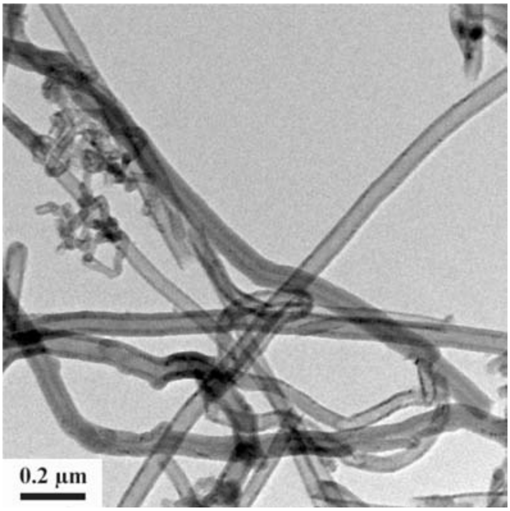

FIGURE 1 TEM image of carbon nanofibers.

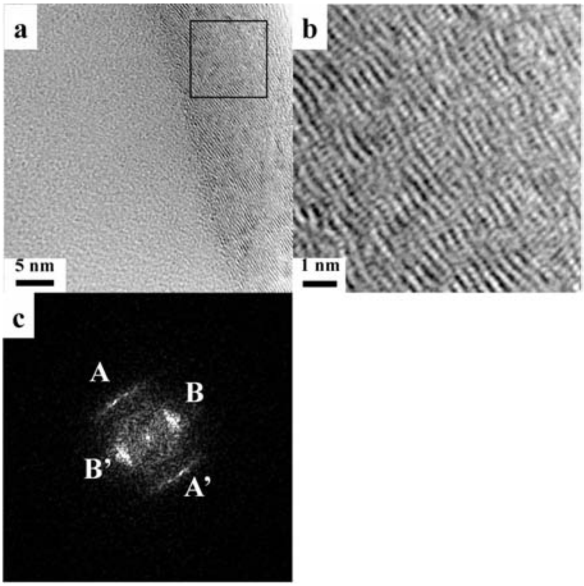

FIGURE 2 (a) HR-TEM image of the carbon nanofibers. (b) The enlarged image of the square in Figure 2(a). (c) The power spectrum of (b).

appear linearly. The spectrum labeled B and B′, on the other hand, is distributed in the crescent like shape, which shows large fluctuation in alignment of the (00·1) lattice planes. Since the coordination of carbon atoms in the benzene ring is rigid, the linear aligned 100 bright parts in the power spectrum indicate that the (00·1) lattice planes glide each other. In detail observation of the spectrum, the slight fluctuation in the 100 linear bright parts is recognized. The disorder of (00·1) lattice lanes thought to cause the fluctuation of 100 bright lines.

Figures 3(a), (b) and (c) show the reconstructed real space images corresponding to the (00·1) lattice planes, the (10·0) lattice planes, and the both lattice planes, respectively. These images were reconstructed by means of 2D-IFFT from each selected specific space frequencies by mask patterns inserted in Figure 3. These results support that the spectrum labeled A and A′, B and B′ shown in Figure 2 correspond to the (00·1) lattice planes and the (10·0) lattice planes, respectively.

The area of fan-shaped mask patterns (see Figure 4), which include 002 and part of 100 spectrum, were superimposed on the power spectrum in

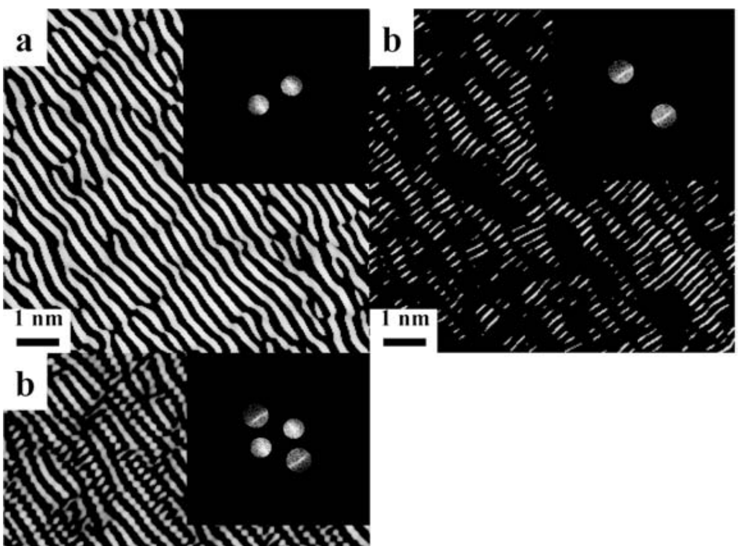

FIGURE 3 2D-IFFT images correspond to (a) (00·1) lattice planes, (b) (10·0) lattice planes, and (c) both (00·1) and (10·0) lattice planes.

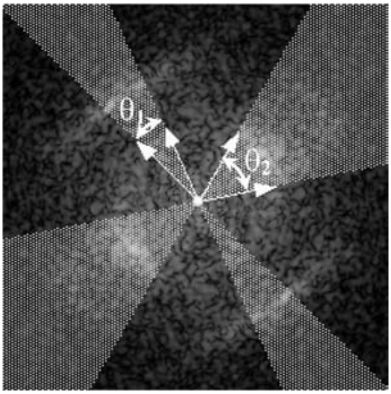

FIGURE 4 Radial mask patterns corresponding to (10·0) lattice planes (in the angle θ_1) and (00.1) lattice planes (in the angle of θ_2).

Figure 3(c). The angle of θ_1 must be enough small to estimate the repeat distance of the (10·0) lattice planes (d_{100}) as the standard. The fan-shaped area in θ_2 angle determines the region contains the spectrum corresponding to (00·1) lattice planes. Figure 5 shows the relation between spacing and power spectrum intensity obtained by integration of the masked area around the center point. The average repeat distance d_{002} was estimated as 0.346 nm on the basis of $d_{100} = 0.213$ nm. The values of d_{002}

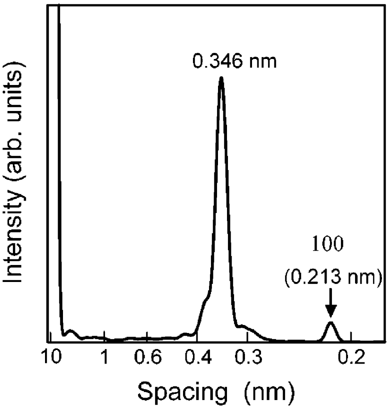

FIGURE 5 Figure 4 was represented by graph obtained by integration of the power spectrum in the masked area around the center point of it. The average repeat distance d_{002} was estimated at 0.346 nm.

were extended from 0.339 to 0.352 nm. Since the d_{002} is larger than that of graphite, the hexagonal carbon layers of the nanofiber are distorted.

SUMMARY

The lattice images of the cup-stacking type nanofibers were observed clearly by HRTEM. Details of structure of the slanted (00·1) layers, which appear at the cross section of the fiber wall, were revealed by this image analysis technique. Two structures of the nanofibers can be assumed; one is the real cup-stacking structure and the other is the spiral ribbon structure, which will affect largely to the electrical properties of them. In the next step, we plan to analyze the central part of the cylindrical fiber in the TEM image, perpendicular to the fiber wall in order to determine the correct structure of the nanofiber.

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