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SEM and TEM Observation of Carbon Nano-Fibers Prepared by Hot Filament Assisted Sputtering

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Carbon films were prepared by hot filament assisted sputtering. Pure argon was used as the sputtering gas. The substrate temperature was 600 °C and the filament temperature was about 2000 °C. Sample was inhomogeneous. Scanning electron microscope (SEM) and transmission electron microscope (TEM) images showed that some part of films consisted of carbon nano fibers (not hollow but solid). Amorphous and polycrystalline phases were also detected by these measurements. No tube structure was observed by high resolution TEM. The diameter of the fiber was 10 – 30 nm.

Keywords: fiber; sputtering; nano structure; hot filament; carbon

INTRODUCTION

Carbon films have been obtained by chemical vapor deposition (CVD), ion beam deposition and sputtering[1-5]. However, their main subjects seem to be focused on obtaining diamond structure.

In this work, a new method was developed for depositing carbon films. The carbon thin films prepared by this method have a columnar structure like that of carbon nano-tube. The films were characterized by scanning electron microscope (SEM) and transmission electron microscope (TEM) observation. The results showed that the column was not hollow (not tube) but solid (fiber).

EXPERIMENTAL

Carbon thin films were deposited using a DC magnetron sputtering system

with a hot filament, as shown in Fig. 1. The target was a graphite disk. The diameter was 5.7 cm. A tungsten filament was placed between the target and substrate. The filament temperature was about 2000 °C, which is high enough for thermal emission of electron. The substrate was quartz glass or single crystal silicon. The substrate was heated by a carbon ribbon, which acted as a substrate holder and a resistance heater. The substrate temperature was 600 °C. Sputtering was carried out under pure argon gas pressure of about 10 Pa. The sputtering power was about 25 W.

The deposition rate was estimated from deposition time and film thickness and was about 5 nm/min. Samples were characterized by SEM and TEM.

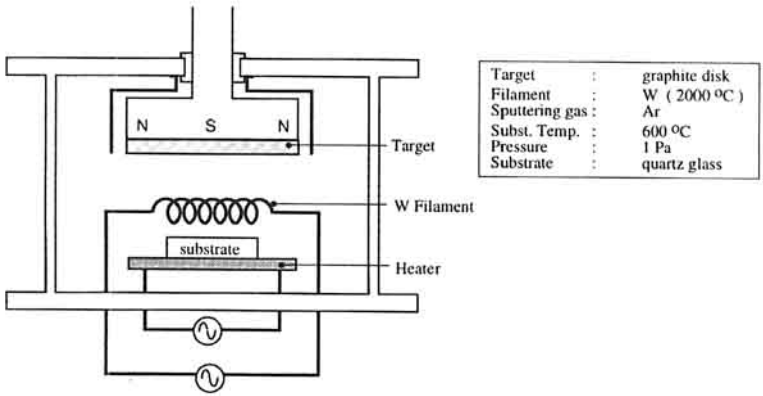


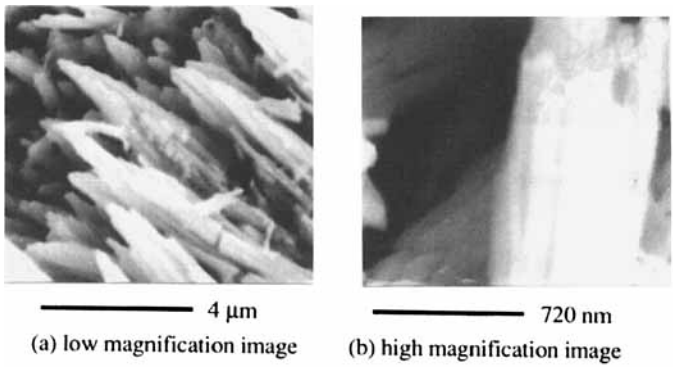
FIGURE 1 Schematic of sputtering apparatus.

RESULTS AND DISCUSSION

Figure 2 shows SEM images of the carbon films prepared by hot filament assisted sputtering. Columnar structure was observed by SEM measurement. Figure 2 (b) is higher magnification observation for the same sample shown in Fig. 2 (a). Figure 2 (b) indicated that each column is constructed by many smaller columns.

Figure 3 shows TEM image of individual columns. The sample was pealed from the glass substrate by HF solution, and set on copper mesh for TEM observation. High resolution observation indicated that the column was not hollow, but solid. As shown in Fig. 3, the diameter was estimated to be 10 - 30 nm. The carbon thin film consists of carbon nano-structure.

In the cace of carbon nano-tube, the center part of tubule is brighter than outer part because of low density. The tube wall results in dark line near the boundary.



(a) low magnification image (b) high magnification image

FIGURE 2 SEM photograph of carbon film.

In the present sample, as shown in Fig. 3(b), the center part was darker than the outer part. This indicated that the column is not hollow tube but solid fiber. The value of about 0.32 nm was estimated as the lattice spacing tangential to the fiber axis (radial direction) from the periodic line spacing in Fig. 3 (b). This value was almost the same as that for the carbon allotropes such as graphite and usual multiwall nano-tubes.

Figure 4 shows selected area diffraction pattern measured for the same sample in Fig. 3. The parallel lines were observed in the diffraction patterns. This indicates one dimensional structure, as expected from the lattice image shown in Fig. 3 (b). The lattice constant longitudinal to the fiber direction was determined to be about 0.33 nm from the diffraction line spacing. The lattice constant tangential to

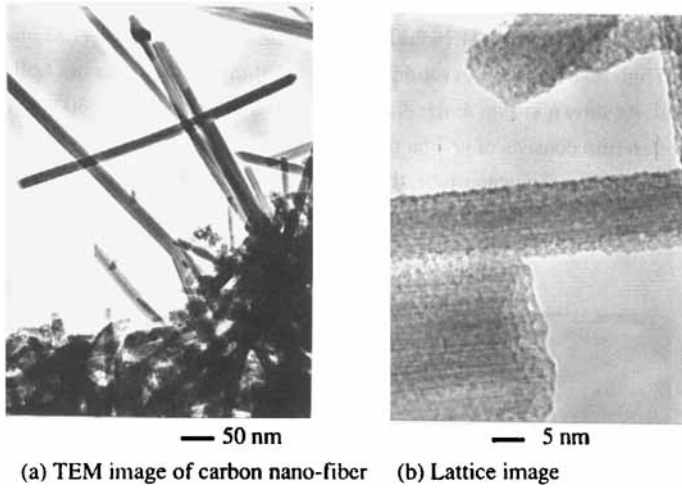


FIGURE 3 TEM image of carbon film.

the fiber direction was also estimated from the periodicity of brightness in diffraction line. The value was 0.31 - 0.35 nm, and was consistent to the value determined from lattice image shown in Fig. 3(b). Since the sample was not homogeneous, spot, ring and diffused patterns were also observed in the electron beam diffraction for the other part of the same sample. The spot pattern was

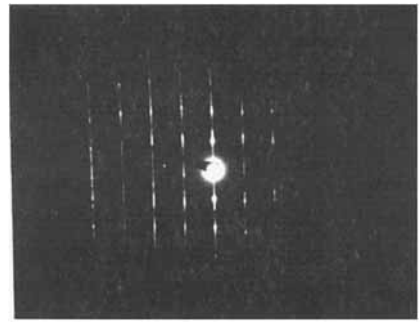


FIGURE 4 Selected area diffraction pattern of carbon nano fiber.

consistent to the 100 diffraction of cubic diamond. This indicates that the film consist of mixture of nano-fiber, diamond phase, graphite phase and also amorphous phase.

CONCLUSION

Carbon films were prepared by hot filament assisted sputtering. Pure argon was used as the sputtering gas. The substrate temperature was 600 °C and the filament temperature was about 2000 °C. SEM and TEM images showed that some part of films consisted of carbon nano-fibers (not hollow but solid). Amorphous and polycrystalline phases were also detected by these measurements. No tube structure was observed by high resolution TEM. The diameter of the fiber was about 30 nm.

Acknowledgement
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References

[1] M. L. Kaplan, P. H. Schmidt, C. H. Chen and W. M. Waish, Jr. Appl. Phys. Letters, 36, 867 (1980).
[2] Y. Onuma, Y. Kato, M. Nakao and H. Matsushima, Jpn. J. Appl. Phys, 22, 888 (1983).
[3] M. Danno and M. Hanabusa, Mater. Letters, 4, 261 (1986).
[4] B. S. Chao, and S. R. Ovshinsky, J. Appl. Phys., 78, 3015 (1995).
[5] R. Ramesham, T. Roppel and J. Mater, Mater. Rev., 7, 1144 (1992).