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# Molecular Crystals and Liquid Crystals

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Preparation and characterization of carbon nano fibers prepared by hot filament assisted dc magnetron sputtering

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# PREPARATION AND CHARACTERIZATION OF CARBON NANO FIBERS PREPARED BY HOT FILAMENT ASSISTED DC MAGNETRON SPUTTERING

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Carbon thin films were prepared by hot filament assisted sputtering system. The sputtering gas was pure Ar (99.9995%), and the temperature of the filament was 2000°C. The properties of the carbon thin films were investigated by X-ray photoelectron spectroscopy (XPS), scanning electron microscopy (SEM), and transmission electron microscopy (TEM). Nano fibers were observed in the carbon films by SEM and TEM measurement. No catalytic material was detected in the fiber. The diameter was about 10 nm.

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

#### INTRODUCTION

In recent years, the field of carbon materials has received great attention due to the discovery of fullernes and carbon nanotubes. There has been considerable interest in the electrical and physical properties of carbon nanotubes and nano fibers [1]. Carbon nanotubes have been prepared by coevaporating carbon and certain metals in arc evaporation, or by laser vaporization of metal-doped graphite [2].

In our recent works, hot filament assisted sputtering was found to be an effective method for obtaining carbon thin films containing carbon nano fibers [3–5]. In this report, the properties of the carbon nano fibers were characterized by using X-ray photoelectron spectroscopy (XPS), scanning electron microscopy (SEM), and transmission electron microscopy (TEM).

#### **EXPERIMENTAL**

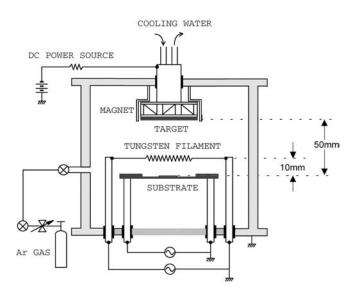
The sample preparation was carried out by a DC magnetron sputtering system with a tungsten hot filament. Figure 1 shows the system used in this work. The target was a graphite disk. The tungsten filament was placed between the target and a substrate, n-Si (110) plate.

The filament temperature was about  $2000^{\circ}$ C, which is high enough for thermal electron emission. The substrate temperature was  $700^{\circ}$ C and the film was deposited onto the substrate for 120 minutes. Sputtering was carried out under pure argon gas pressure of about 150 Pa. The sputtering power was about 35 W.

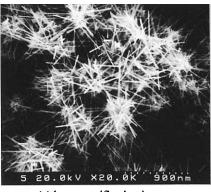
The properties of the films were characterized by using XPS, SEM and TEM. The results were examined into detail referring to the deposition conditions.

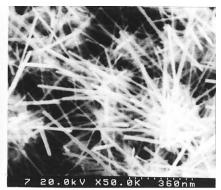
#### RESULTS AND DISCUSSION

In the case of the film deposited using a new filament, diffraction peaks corresponding metal tungsten appeared in the X ray diffraction patterns. Chemical composition of the film was determined by the XPS profile. There were three bands corresponding to the binding energy of  $C_{1\rm s}$ ,  $W_{5\rm p}^{3/2}$  and  $O_{\rm ls}$  in the spectrum.



**FIGURE 1** Schematic diagram of a sputtering apparatus.





(a) low magnification image

(b) high magnification image

FIGURE 2 SEM images of carbon film.

No tungsten appeared in the film prepared after several deposition cycles. Tungsten carbide seemed to be formed at the surface of the filament during this process, and this layer seemed to prevent the evaporation and incorporation of tungsten.



FIGURE 3 TEM image of a carbon nano fiber.

The film surface was observed using SEM. Figure 2 shows the SEM images of carbon films. Fibers were observed in the surface of the film. The diameter of the fiber was estimated to be about 10 nm or less.

Figure 3 shows a TEM image of a carbon nano fiber deposited by hot filament assisted sputtering. The diameter of this fiber was about 10 nm. The film consisted of these fibers and crystalline carbon grains [6]. No catalytic material was detected in the fiber. In the contrast to the nanotubes, the inside of the fiber seemed not to be hollow [7].

### CONCLUSION

Crystalline carbon thin films consisting of nano scale fibrous crystallites were grown by hot filament assisted DC magnetron sputtering. Hot filament assistance was effective to obtain carbon Nano fibers. The inside of the fiber seemed not to be hollow. The diameter of the fiber was about 10 nm or less. No catalytic material was detected in the fiber.

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