

Vapor Phase Deposited Sub-micron Carbon Tubes

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INTRODUCTION

Tubular carbon fibers with diameters ranging from 100 nm to 9 μm were produced through vapor phase deposition in the presence of a transition metal catalyst. Effects of temperature and time on the growth of carbon tubes were examined. The growth rate and structure were observed to be sensitive to the deposition temperature. Also, smaller sub-micron carbon tubes were observed at longer processing times.

EXPERIMENTAL

The growth of tubular carbon was conducted using a technique similar to that described in references 1 and 2.^{1,2} The gas mixture used was $\text{CH}_4 + \text{H}_2$ with 2 different ratios: $\text{CH}_4/\text{H}_2 = 1/6$ and $2/5$. Five different deposition temperatures of 900, 1000, 1100, 1200, and 1300 $^\circ\text{C}$, and 4 deposition times of 5, 10, 20, and 30 minutes, were used. In each experiment, the gas mixture was introduced when the deposition chamber reached the desired deposition temperature. Carbon tubes thus produced were examined using scanning electron microscopy (SEM) and transmission electron microscopy (TEM).

RESULTS AND DISCUSSION

Figure 1 shows SEM micrographs of sub-micron carbon tubes obtained at 900 $^\circ\text{C}$ and $\text{CH}_4/\text{H}_2 = 2/5$. The (outer) diameters of carbon tubes obtained using different deposition temperatures were measured. It was found that for $\text{CH}_4/\text{H}_2 = 2/5$, longer deposition times did not lead to tubes with larger diameters when $T = 900$ $^\circ\text{C}$ and 1000 $^\circ\text{C}$. For the same gas ratio, diameter was

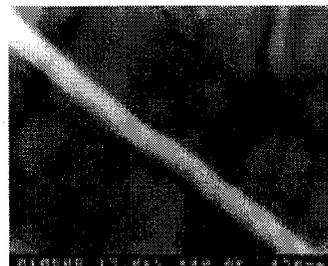


Figure 1. Micrographs of sub-micron carbon tubes obtained at 900 $^\circ\text{C}$ and $\text{CH}_4/\text{H}_2 = 2/5$.

found to increase with increasing time when $T = 1100$ $^\circ\text{C}$ and 1200 $^\circ\text{C}$. For these two temperatures, the lower temperature gave a higher thickening rate as shown in Fig. 2. Figure 2 also shows that when the methane concentration was reduced from $2/5$ to $1/6$, the thickening rate decreased from 300 nm/s to 4 nm/s.

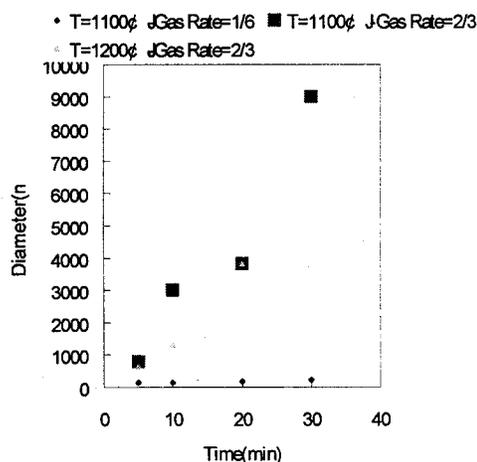


Fig. 2. Growth kinetics of carbon tubes.

In the case of $T = 1300$ $^\circ\text{C}$, beaded carbon tubes were observed in addition to regular carbon tubes, as shown in Fig. 3.³ The tubular nature of the beaded structure was observed between beads using TEM. It is not

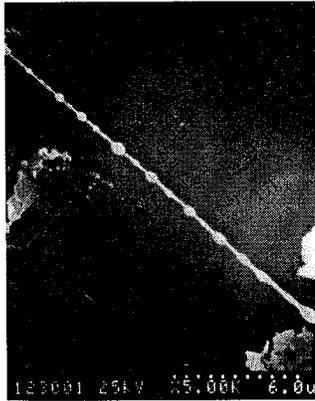


Fig. 3. Micrographs of beaded structure.

known if the hollow tubular center extends into the beads. The mechanism of forming beaded structures is currently not known. However, it is thought that localized temperature variation, coupled with the resulting gaseous transport variation, favors a rapid construction of carbon atoms into bamboo-like structure at lower temperatures⁴ and beads at higher temperatures.

CONCLUSION

Thickening kinetics and structure of vapor phase deposited carbon tubes were examined. It was found that 1100 °C was the

deposition temperature which lead to the highest thicken rate. Unusual, beaded carbon tubes were observed at a deposition temperature of 1300 °C.

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Fig. 1. Micrographs of sub-micron carbon tubes obtained at 900 °C and $\text{CH}_4/\text{H}_2 = 2/5$.

Fig. 2. Growth kinetics of carbon tubes.

Fig. 3. Micrographs of beaded structure.

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