Influence of Injection Temperature on the Structure of Carbon Nanotubes Synthesized by Floating Catalyst Method

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Introduction

Floating catalyst method is a semi-continuous process of producing MWNTs, which have benefits of high yield and purity[1-2]. Many researchers reported that the diameter of carbon nanotubes produced by floating catalyst method was determined by the reaction temperature[3-4]. However, other reaction parameters also affects on their diameter and structure[5]. In this study, we tried to find out the influence of injection temperature on the structure of carbon nanotubes including diameter.

Experimental

MWNTs were synthesized by the floating catalyst method using a mixture of ferrocene and xylene at the range of 750~850° in a quartz tube reactor[1]. The mixture of ferrocene and xylene was fed continuously into a tubular quartz reactor using a syringe pump. The feed solution was heated while passing through a capillary tube in the range of 165~270° and fed into the quartz tube reactor. The mixture exiting the capillary was immediately volatilized and swept into the reaction zone of the quartz tube reactor by the gas mixture of nitrogen and hydrogen. The ferrocene-xylene mol ratio was 0.065, and the flow rates of hydrogen and nitrogen were 40 and 250 sccm, respectively. The flow rate of ferrocene-xylene mixture was 5 ml/hr.

Results and Discussion

When the injection temperatue was in the range of sublimation pt. of ferrocene, the obtained MWNTs had broad distribution of diameter but some MWNTs had very thin diameters under 10 nm. As the reaction temperature was increased, the diameter of carbon nanotubes became larger as shown in Fig. 1. As shown in Fig. 2, at the injection temperatures between the melting and boiling pt. of ferrocene, the diameter of carbon nanotubes became smaller as the temperature increased. Surprisingly, over the boiling pt. of ferrocene, the diameters became smaller and uniform. Moreover, at this condition, the diameter of carbon nanotubes were not changed with the variation of reaction temperature and the average diameter was about 30~40 nm. The degree of MWNTs' order was analyzed using Raman Spectroscopy and the crystalinity was also influenced by the injection temperatue, namely the phase of ferrocene at the injection part, as shown in Fig. 3.

Conclusions

The carbon nanotubes produced near sublimation pt. of injection temperature had a wide range of diameter and curly structure, but the diameter of carbon nanotubes produced over the boiling pt. became smaller and more uniform than those produced at other injection conditions. We found that the phase of ferrocene at the injection part was important factor that could affect the carbon nanotubes' structure.

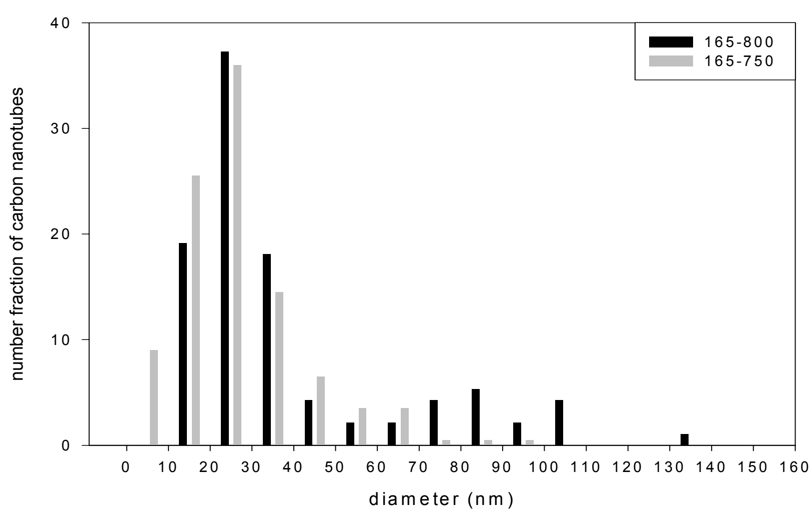

Fig. 1. Diameter distributions of carbon nanotubes produced at different reaction temperatures and 165° of injection temperature.

(a) 200° (b) 210° (c) 230°

Fig. 2. FE-SEM images of carbon nanotubes synthesized at different injection temperatures between melting and boiling pt. of ferrocene

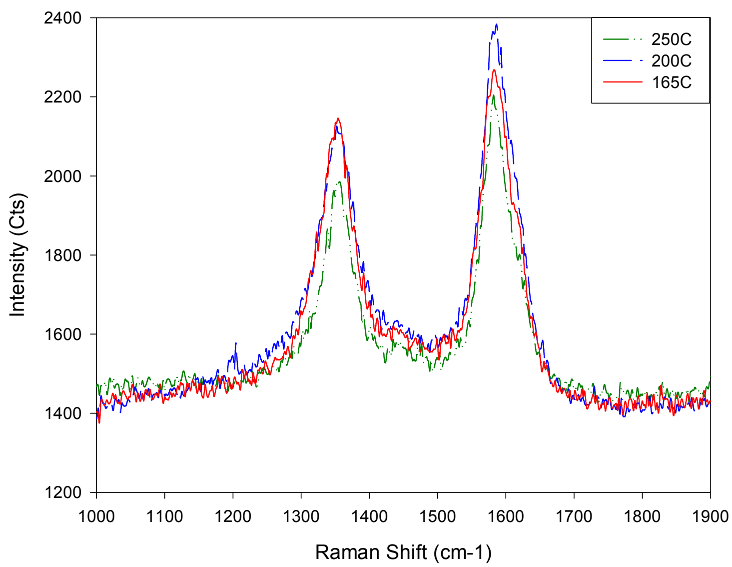

Fig.3. Raman spectrum of carbon nanotubes produced at different injection temperatures.

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Acknowledgments
This work was supported by the RRC program of MOST and Gyeonggi-do.