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The yield and structure of CNTs synthesized by using Fe-Mo/MgO catalyst

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Introduction
Carbon nanotubes are expected to have wide applications in many fields, because they have excellent properties like high aspect ratio, high mechanical strength and electric conductivity. The technology to fabricate CNTs with homogeneous structure and size in a low cost is still a big challenge for researchers. Chemical vapor deposition (CVD) is a promising method for large-scale synthesis of CNTs. The yield and structure of CNTs depend on many parameters, especially on temperature, catalyst composition and catalyst particle size. It has found that the yield of CNTs synthesized by using Co-Mo/MgO catalyst contained around 10% Mo is higher than other ratio. However, the synthesized CNTs are severely curved and aggregated.

In this study, we investigated the yield and structure of CNTs synthesized by using Fe-Mo/MgO catalyst, and examined the shape and aggregate state of CNTs.

Experimental
To prepare the catalyst, Fe(NO$_3$)$_3$·9H$_2$O, Mg(NO$_3$)$_2$·6H$_2$O and (NH$_4$)$_6$Mo$_7$O$_{24}$·4H$_2$O were mixed at 60-70°C. After the mixture was gelled, it was dried at 110°C for 2 days. The catalyst precursor was pyrolyzed in 100ml/min N$_2$ at 500°C and calcined in air at 700°C for 5h. After reduction in 30ml/min H$_2$ for 30min, CNTs were synthesized by decomposition of C$_2$H$_4$ in 20ml/min at 700-850°C for 30min. Two kinds of catalysts were prepared. The morphology and structure of CNTs were examined using FE-SEM and TEM. The catalyst was examined using XRD. Yield of CNTs was calculated using the following equation.

$$ \text{Carbynyield (g/g-cat)} = \frac{M_{\text{carbon}}}{M_{\text{Fe}2O3} + M_{\text{MoO}}}. $$

Two catalysts, Cat9119 with ratio of (Fe : Mo = 9 : 1) : Mg = 10 : 90 and Cat9128 with ratio of (Fe : Mo = 9 : 1) : Mg = 20 : 80, were prepared. CNT synthesized using Cat9119 is denoted as CNT-9119 and that synthesized using Cat9128 is denoted as CNT-9128.

Results and discussion
Fig.1 shows that relationship between synthesis temperature and the yield of CNTs. The yield of CNTs increased with synthesis temperature, and CNTs synthesized at 750°C showed higher CNT yield than those at 650°C, 700°C and 800°C. The yield of CNT-9119 synthesized at 750°C was 19 (g / g-cat) and The yield of CNT-9128 was 25 (g / g-cat).
Fig. 2 shows TEM image of CNTs. The diameter of CNTs ranged from 10 nm to 50 nm. Fig. 3 shows inner and outer diameter of CNT-9128. Both inner diameter and outer diameter increased with synthesis temperature from 700°C to 800°C. Increase of outer diameter is attributed to pyrolytic carbon deposition and catalyst particle growth. Increase of inner diameter indicates that catalysts cohered.

TEM observations showed that many CNTs are grown from the nanoparticles on a large MgO particle. Since particles are packed together and the space between particles is limited, the growth of CNTs was inhibited by other particles or CNTs. As a result, the CNTs were curved and twisted together, and the CNTs grown from same MgO particles form a large aggregate.

**Conclusion**

CNT showed highest yield at 750°C but both inner diameter and outer diameter increased with synthesis temperature from 700°C to 800°C. CNTs grown from same MgO particles form a large aggregate.