

# Growth Termination of Carbon Nanotubes at Millimeter Thickness Due to Structural Change in Catalyst

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The water-assisted growth method, so-called "supergrowth [1]", realized millimeter-thick vertically-aligned single-walled carbon nanotubes (VA-SWNTs). Later, it is reported that "supergrowth" rate decreases with reaction time and finally the growth terminates [2]. Our group recently reproduced "supergrowth" [3] and observed similar "supergrowth" termination within a few tens minutes. In this work, we analyzed substrate surface before and after CVD to clarify the mechanisms of catalyst deactivation.

0.5-2-nm-thick Fe catalyst was prepared on Al<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub> substrate by sputtering method. After 10 minutes reduction of this catalyst under 27 kPa H<sub>2</sub>/ 0.010 kPa H<sub>2</sub>O/ Ar balance at atmospheric pressure, 8.0 kPa C<sub>2</sub>H<sub>4</sub> was introduced and CVD was carried out at 1093 K for 3-30 min. After removing CNT by oxidation in air at 1000 K, substrate surfaces were investigated by SEM and XPS. SEM images of substrate surfaces showed that the mean diameter of catalyst particles increased while number density of catalyst particles decreased with an increasing CVD time (Fig. 1). Fe/Al intensity ratio by XPS decreased with reaction time, and this change occurred quickly for thinner Fe (Fig. 2). The decrease in Fe/Al ratio is caused by the increase in Fe particle size rather than by the decrease in the amount of Fe. The coarsening of Fe particles during CVD is possibly the fundamental cause of the growth termination under our experimental conditions. More details will be reported.

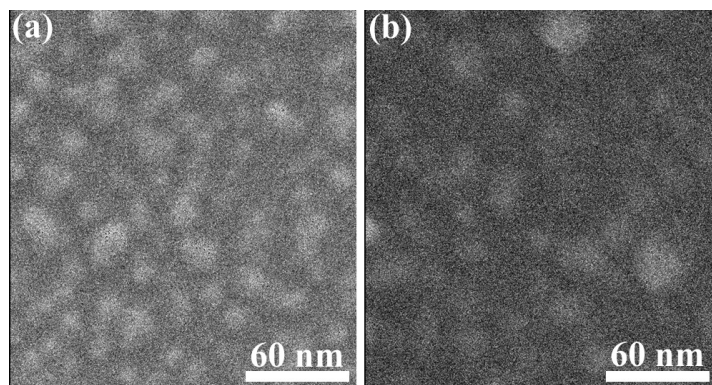


Fig. 1 SEM images of 2 nm Fe/ Al<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub> sample surfaces after CVD for (a) 3 min and (b) 30 min. VA-CNTs were removed by oxidizing under air at 1000 K.

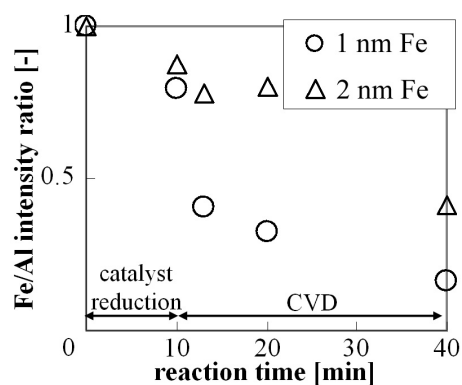


Fig. 2 Change of XPS intensity with reduction/CVD time at 1093 K. Intensity ratio was normalized by the initial value.

## References

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