

***In situ* NEXAFS Study on Carbon Nanocap Formation Process at High Temperature by SiC Surface Decomposition**

Takahiro MARUYAMA*¹, Satoshi SAKAKIBARA¹, Hiroaki ITOH¹,
Kenta AMEMIYA²

¹Department of Materials Science, Meijo University, Nagoya 468-8502, Japan

²KEK-PF, Tsukuba, Ibaraki 305-0801, Japan

Introduction

Carbon nanotube (CNT) growth by surface decomposition of SiC is a unique growth technique, because zigzag-type CNTs are selectively formed without any catalyst. At the initial stage of CNT formation, nanosized hemispherical structures composed of carbon atoms are formed on SiC surfaces [1]. These “carbon nanocaps” determine the structure of CNTs, such as the number of walls, diameters and chirality [2]. Therefore, clarifying the formation mechanism of carbon nanocaps leads to control of the CNT structure. Previous TEM and STM studies suggested that these carbon nanocaps were formed by crystallization of amorphous carbon on SiC surface. In this study, we carried out *in situ* NEXAFS measurements and investigated crystallization process to carbon nanocaps.

Experimental Procedure

6H-SiC(000-1) substrates were dipped into a 40% hydrofluoric acid (HF) solution for 15 min to remove surface oxides. After rinsing by deionized water, the samples were placed in a high vacuum chamber ($<1 \times 10^{-7}$ torr). High-purity hydrogen gas of 6N (99.9999%) was then introduced into the chamber and the samples were gradually heated to an intended temperature, and left for 30 min. The sample temperature was measured with a pyrometer, and the H₂ pressure was maintained between $1 \sim 4 \times 10^{-4}$ torr. Then, the hydrogen gas was exhausted and C K edge NEXAFS spectra were measured at BL-7A in Auger electron yield detection mode, keeping the sample at the heating temperature.

Results and Discussion

Fig. 1 shows NEXAFS spectra of SiC(000-1) surfaces at several temperatures. At each temperature, we carried out the NEXAFS measurements with two different incident angles, θ , i. e., 30° and 90°, to investigate the directions of C-C bonds. At 800°C, the NEXAFS spectral shapes at two incident angles were almost the same, and were also similar to that observed for 6H-SiC(000-1). When the sample temperature was 1190°C, the spectral shapes drastically changed and π^* resonance peaks were observed for the two incident angles. It should be noted that there were slight differences between the spectra. At $\theta=30^\circ$, a sharp peak was observed at 291.5 eV, which corresponds to a σ^* bound exciton accompanied by a

broad σ^* resonance in the region of 290 to 315 eV. In contrast, at $\theta=90^\circ$, the σ^* bound exciton peak was blurred and the relative intensity of the σ^* to the π^* resonance peak increased. This suggests that, at the beginning of crystallization, most C-C bonds in the carbon hexagonal networks were inclined with a large angle with respect to the surface normal. At 1250°C, the NEXAFS spectra at the two incident angles were similar, and both the π^* resonance and σ^* bound exciton peaks were observed at 285 and 291.5 eV, respectively. This polarization dependence indicates that carbon nanocaps were formed at this temperature, because they consist of arced graphene sheets [1].

In summary, we succeeded in detecting the crystallization process to carbon nanocaps by *in situ* NEXAFS measurements. The direction change of C-C bonds was observed during crystallization.

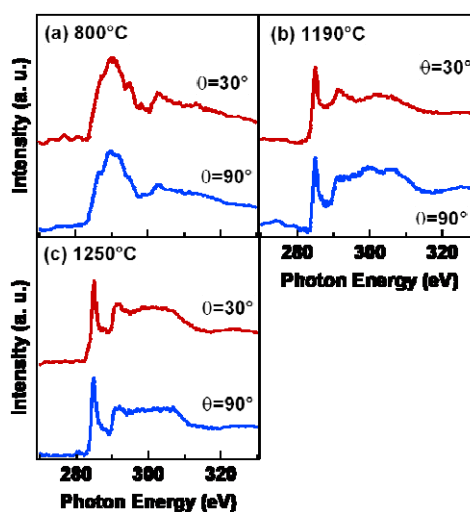


Fig. 1: NEXAFS spectra of SiC(000-1) during heating at (a) 800°C, (b) 1190°C and (c) 1250°C. At each temperature, the spectra measured at two incident angles, 30° and 90°, are shown. (The incident angle, θ , was measured as an angle between the direction of X-ray and the sample surface.)

References

- [1] M. Kusunoki, et al., Appl. Phys. Lett. 77, 531 (2000).
- [2] T. Maruyama et al., Chem. Phys. Lett. 423, 317 (2006).

* takamaru@meijo-u.ac.jp