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In situ XAFS Study on Carbon Nanocap Formation by SiC Surface Decomposition

Takahiro MARUYAMA*[1], Kenta AMEMIYA[2]
[1]Department of Materials Science, Meijo University, Nagoya 468-8502, Japan
[2]KEK-PF, Tsukuba, Ibaraki 305-0801, Japan

Introduction

Carbon nanotube (CNT) growth by surface decomposition of SiC is a unique growth technique, because no catalysts are necessary and because zigzag-type CNTs are selectively formed. At the initial stage of CNT formation, nanosized hemispherical structures composed of carbon atoms are formed on the SiC surface [1]. These "carbon nanocaps" determine the structure of CNTs, such as the number of walls, diameters and chirality. Therefore, clarifying the formation mechanism of carbon nanocaps leads to control of the CNT structure. Through STM and XPS studies, we have reported the formation process of carbon nanocaps and proposed a model of CNT formation [2]. However, to clarify the mechanism in detail, *in situ* observation for the carbon nanocap formation is essential. In this study, we carried out *in situ* XAFS measurements for SiC surfaces through the carbon nanocap formation.

Experimental Procedure

Commercial single-crystal wafers of 6H-SiC(000-1) (CREE Research, Inc.) cut into 4.0×7.0×0.2 mm bars 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 (<1×10^{-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 H_2 pressure was maintained between $1\sim4\times10^{-4}$ torr. Then, the hydrogen gas was exhausted and C *K* XAFS spectra were measured at BL-7A in Auger electron yield detection mode, keeping the sample at the heating temperature.

Results and Disccusion

Fig. 1 shows XAFS spectra of SiC(000-1) surfaces at several temperatures. At 925°C, the spectrum was quite similar to that of SiC surface [3], indicating that sublimation of Si atoms was very small and the thickness of carbon layer was less than 1 nm. When the sample temperature rose to 1200°C, the shape of XAFS spectrum considerably changed and a shoulder peak corresponding to C-C π* resonance appeared at 285 eV. At 1275°C, the π* resonance became sharp and the spectrum resembles the XAFS spectrum for CNTs [4]. At this temperature, carbon nanocaps of several nanometers in length should be formed on the SiC surface, which is consistent with the spectrum in Fig. 1.

To investigate carbon structure before carbon nanocap formation in detail, the spectrum at 1200°C after subtraction of that at 925°C is shown at the top in Fig. 1. This spectrum corresponds to that of carbon layers piled up on the SiC surface after sublimation of Si. The spectrum shows weak π* resonance at 285 eV and broad σ* resonance between 290 and 315 eV, indicating that carbon structure at 1200°C is amorphous-like. From the XAFS analysis, it is confirmed that amorphous carbon layers induced by sublimation of Si are crystallized above 1200°C, forming carbon nanocaps on the SiC surface. This result supports our previous model for formation of carbon nanocaps.

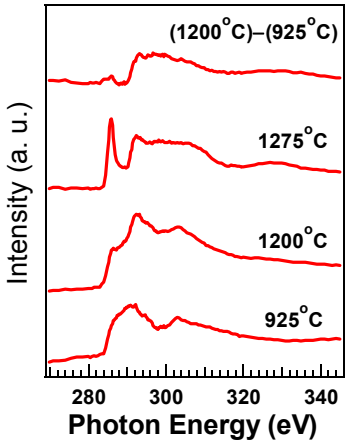

Fig. 1: XAFS spectra of SiC(000-1) during heating. The sample temperatures are shown in the figure. At the top, the spectrum at 1200°C after subtraction of that at 925°C is shown.

References

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* takamaru@meijo-u.ac.jp