Carbon Nanotubes on SiC Powder Surface Grown by a Vacuum Heating Process

Hirofumi TAKIKAWA, Ryuichi MIYANO¹, Miki YATSUKI and Tateki SAKAKIBARA

Department of Electrical and Electronic Engineering, Toyohashi University of Technology,

1-1 Hibarigaoka, Tempaku, Toyohashi, Aichi 441, Japan

¹Satellite Venture Business Laboratory, Shizuoka University, 3-5-1 Johoku, Hamamatsu, Shizuoka 432, Japan

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Silicon carbide (SiC) powders were baked on a resistively heated tungsten (W) boat at 1,600–1,700°C for 10–15 min in vacuum. The surface morphology of the treated SiC powders was observed using a high resolution scanning electron microscope (HR-SEM). Carbon nanotubes were observed on the surface of 40% of SiC powder. The nanotubes were dense, but slightly dispersed and their growth direction was almost perpendicular to the original surface. The nanotubes on SiC were considerably shorter than that prepared by carbon arc method, but significantly longer than that prepared by laser sublimation of SiC. Energy dispersive X-ray spectrometory (EDX) analysis suggested that W and O were doped on or in the vicinity of the powder surface.

KEYWORDS: carbon nanotubes, SiC powder, sublimation, vacuum heating, W boat

1. Introduction

Carbon nanotubes with a nanometer-scale structure are of great interest as potential materials for future generation electric and electronic devices, such as field emitters, 1,2) semiconductors, and electrodes for electrochemical capacitors. In particular, carbon nanotube field emitters are very attractive for their application. With respect to their application, several methods have been reported to prepare large-area aligned nanotube films. 3,5-7)

At the outset, carbon nanotubes were discovered in the cathode deposit of carbon arc discharge for fullerene production⁸⁾ and later massive quantities of nanotubes were obtained by the same method.⁹⁾ Catalytic pyrolysis of hydrocarbons^{10–12)} and condensed-phase electrolysis^{13,14)} were also found to be effective methods for nanotube synthesis. Recently, a new method for nanotube synthesis has been reported.¹⁵⁾ Nanotubes were epitaxially grown on silicon carbide (SiC) particles (about 1 μ m¹⁶⁾) by laser beam irradiation on the SiC surface and silicon sublimation from the surface. This transformation of SiC to nanotubes is attractive due to the potential application for manufacturing a nanotube film over a very wide area on the large SiC substrate which can be prepared by the chemical vapor deposition (CVD) method.

In this study, prior to using the expensive SiC substrates, the possibility of the transformation of SiC to nanotubes on larger SiC particles by means of baking the particles on a resistively heated tungsten (W) boat in vacuum, instead of the laser irradiation heating method, was investigated.

2. Experimental Setup and Procedure

Figure 1 shows the experimental setup. A W boat (3 mm wide, 35 mm long, 0.1 mm thick) was located in vacuum belljar (Pyrex; 290 mm in diameter, 285 mm in height). 0.05 g of SiC powder (300 mesh; about 50 μ m in diameter) was sprinkled on the W boat. The bell-jar was evacuated down to about 10^{-2} Pa and then the boat was gradually heated by increasing the current for about 5 min. The temperature of the W boat was kept constant at 1,600–1,700°C for 10–15 min. The current was about 45 A. The current was then gradually reduced to 0 A for 10 min. The treated SiC powder was taken out when it was cooled down to room temperature. The temperature was monitored by a pyrometer (Minolta; TR-630) during the experiment.

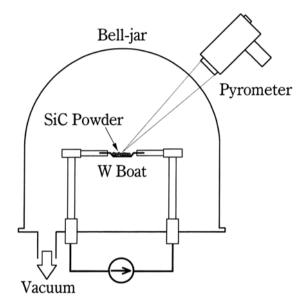


Fig. 1. Experimental setup for carbon nanotube formation on SiC powder.

3. Results and Discussions

The color of the powder changed from pale green to dark black after the heating process. The morphology of the heated powder was observed using a high-resolution scanning electron microscope (HR-SEM; Topcon, ABT-150F). A typical photograph is shown in Fig. 2. The particle surface was covered by dense nanotubes without any obstacle nanoparticles, as seen by arc method. Such nanotubes were found on the surface of 40% of the SiC particles treated. Most nanotubes grew almost perpendicular to the particle surface, as shown in Fig. 3 which is the magnified image of central region of Fig. 2. The tube top was closed and the size was typically 20 to 50 nm in diameter and up to $1 \mu m$ in length. These nanotubes were much shorter than those produced by the arc method (about $100 \, \mu \text{m}^{17}$). On comparing these to the nanotubes obtained by laser sublimation of SiC, 15) it was observed that the former were much longer and more moderately dispersed than the latter.

A SEM image of another particle is shown in Fig. 4. There are no nanotubes on the upper-left side of the photograph (left side of the particle jag), whereas many nanotubes appear on