Jpn. J. Appl. Phys. Vol. 38 (1999) pp. L 401–L 402
Part 2, No. 4A, 1 April 1999

Synthesis of Silicon Oxide Nanofibers by Sublimation of SiC in Medium Vacuum with Oxygen Flow

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(Received December 10, 1998; accepted for publication February 12, 1999)

Silicon carbide (SiC) particles with/without cobalt (Co) powder as catalyst were heated on a carbon (C) boat at temperatures ranging from 1,300 to 1,550°C for 10 min in medium vacuum with low oxygen (O_2) flow rate. A large quantity of fiberlike products was found on the SiC particles. The products were observed using a high-resolution scanning electron microscope (HR-SEM) and a high-resolution transmission electron microscope (HR-TEM). Their compositions were also analyzed by electron diffraction analysis, energy dispersive X-ray spectroscopy (EDX) equipped with HR-TEM, and X-ray diffraction (XRD) analysis, to be amorphous SiO_2.

KEYWORDS: SiO_2 nanofiber, SiC particle, Co catalyst, vacuum sublimation, oxygen flow

1. Introduction

Kusunoki *et al.*[1] reported the production of carbon nanotubes[2] with good orientation by sublimation decomposition of silicon carbide (SiC) particles. Recently, they fabricated a well-aligned carbon nanotube film on a SiC wafer by heating in a vacuum electric furnace.[3] We have also produced a large quantity of carbon nanotubes by heating SiC powder on a tungsten boat in vacuum.[4] If oxygen could play an important role in promoting Si sublimation from the SiC surface and the transformation of SiC to nanotubes, not only residual oxygen (O) but also excess oxygen might be considered to accelerate nanotube formation. In this study, SiC particles were heated in medium vacuum with low oxygen (O_2) flow rate. Furthermore, SiC particles mixed with Co powder were also tested, since metalic catalysts such as iron (Fe), cobalt (Co), and nickel (Ni), have been used to produce a single-wall carbon nanotube in the carbon arc method.[5–7]

2. Experiment

A carbon (C) boat was placed in a water-cooled stainless steel vacuum chamber 300 mm long and 200 mm in diameter, which was usually used for reactive vacuum arc deposition.[8] The semicylinderical carbon boat was 4 mm in diameter and 10 mm long. SiC particles (α type, roughly 20 μm in diameter) with/without Co powder (0.03 μm in diameter) (SiC : Co = 2 : 1 by weight) were sprinkled on the boat. The chamber was evacuated with a turbomolecular pump and a rotary pump. The oxygen gas flow rate of 0.1 ml/min (1 atm, 300 K) into the chamber was regulated with a mass flow controller, and the chamber pressure was regulated at 0.1 Pa with a conductance exhaust valve. The particles and powder were heated on the C boat at temperatures ranging from 1,200 to 1,650°C for 10 min, after 5 min of temperature elevation. Temperature was measured with a pyrometer (Minolta, TR-630A). The resultant samples were taken out from the chamber when the boat temperature reached room temperature (approximately, 1 h).

3. Results and Discussions

Figure 1 shows an image of products on the surface of a SiC particle heated with Co powder at 1,500°C, as observed by a high-resolution scanning electron microscope (HR-SEM; Topcon, ABT-150F). The SiC particle was covered by a large quantity of fiberlike products that were not aligned perpendicular to the SiC surface, similar to nanotubes.[4] The fiberlike products were also observed using a high-resolution transmission electron microscope (HR-TEM; JEOL, HR-2010). A typical micrograph is shown in Fig. 2. The products were obviously not tubular and had no crystalline phase, and were typically 70 nm in diameter and up to 10 μm long. Figure 3 shows a magnified image of the product edge, where a black sphere and a relatively electron-transparent fiber were observed. Most products had a similar structure at their edge when SiC particles were heated with Co powder. The chemical compositions of the sphere and the fiber were analyzed by energy dispersive X-ray spectroscopy (EDX; NORAN Inst., Voyager II) equipped with the HR-TEM. The sphere was identified as Co. From the fiber, Si and O were detected and the atomic ratio of Si to O was found to be almost 0.5. Also, from electron diffraction analysis with HR-TEM, it was found that the electron diffraction pattern of the sphere showed a crystal lattice structure, whereas that of the fiber showed only an amorphous halo pattern. Moreover, SiC particles covered with fiberlike products were analyzed with an X-ray diffraction analyzer (XRD; Rigaku, RINT-2500) for further verification. Only the diffraction peaks due to SiC and Co were detected. From those results, the products on the SiC surface were concluded to be amorphous SiO_2 nanofibers.

The SiO_2 nanofibers were observed on more than 50% of

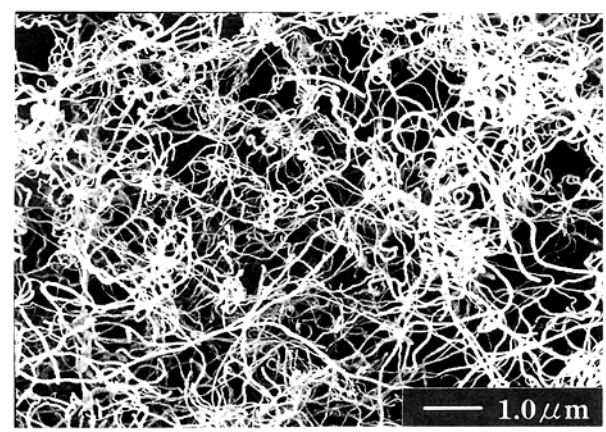

Fig. 1. High-resolution SEM image of surface of SiC particle heated at 1,500°C for 10 min with Co powder under medium vacuum with O_2 flow.