Pulsed Plasma Synthesis of Nanorod Filled Iron Carbide

Soemsak Yooyen\textsuperscript{a} and Yasuyuki Suzuki\textsuperscript{b}

\textsuperscript{a}Department of Mechanical Engineering, Faculty of Engineering, King Mongkut’s Institute of Technology Ladkrabang

\textsuperscript{b}Graduate School of Engineering, Mie University,

1 Chalongkrung Rd., Ladkrabang, Bangkok 10520 THAILAND e-mail: kysoemsa@kmitl.ac.th

1577 Kurimamachiyacho, Tsu City, Mie Prefecture, 514-8507 JAPAN

Introduction

Since the discovery of carbon nanotubes (CNTs) [1] and because of its applicable useful characteristics in various fields of research, many researchers have been successfully used many methods to synthesis CNTs and nanorods filled with ferromagnetic metals like iron, cobolt or nickel. Plasma enhanced chemical vapor deposition (PECVD) is one of a cost-effective method with controllability of process parameters and ease to scale up [2]. Diamond-like Carbon (DLC) was grown using pulse-DC PECVD [3] which a polarity of substrate was negative, opposite pole to the usual DC PECVD. CNTs were grown by pulse-DC [4] under high vacuum chamber. In this study, the synthesis of nanorod by means of pulsed plasma chemical vapor deposition (PPCVD) however under low vacuum chamber directly on Iron (Fe) substrate acted as both the catalyst and supporter for the growth of nanorods was studied. The optimum time for growing nanorod as well as its surface morphology were also studied. The surface morphology was observed by field emission scanning electron microscope (FESEM) while the crystal structure was evaluated by x-ray diffraction (XRD) and transmission electron microscope (TEM).}

Experimental

The experiment was taken under the discharge frequency of 1000 Hz, with discharge duty 10% and discharge distance 10 mm. Pure Fe plate 10 x 10 x 0.5 mm was cleaned by ultrasonic degreasing for 30 min before placed in a chamber and evacuated to the initial pressure. The substrate temperature was maintained about 600\degree C during the experiment. The synthesis using methane (CH\textsubscript{4}:H\textsubscript{2}) 7% was performed from 1 to 30 min. The gas pressure was 4.1 kPa and the gas flow rate was 30 sccm. The surface morphology and the average diameter and length of the particles, which were determined by measuring the diameter and length of the particles on the FESEM image were observed by using Hitachi S4000 while the crystal structure was evaluated by using Rigaku Cu-K\alpha radiation 4.8 kW. TEM using Hitachi H-9000 operating at 200 kV.

Results and Discussions

Nanoparticles found on pretreatment of CNTs growth [5] were also observed on the substrate after treatment by H\textsubscript{2} plasma for 10 min. The average diameter was about 40 nm, which corresponds to that of nanorods. Nanorods were rapidly grown after further treatment by CH\textsubscript{4}:H\textsubscript{2} plasma. A period time of growth were agreed with previous studied [6], [7] but the structural behavior was vertically aligned. The rods were rapidly grown in the first 3 min and gradually reduced when CH\textsubscript{4}:H\textsubscript{2} plasma period was 10 min. The rods density were also rapidly increased in the first 3 min and continual increased until CH\textsubscript{4}:H\textsubscript{2} plasma period was 10 min. Nanorod can be initiated growing when treatment by CH\textsubscript{4}:H\textsubscript{2} plasma because of bombardment [4]. The proper time of CH\textsubscript{4}:H\textsubscript{2} plasma treatment was until the length and the number of rod were decreased which was about 10 min. The diffraction of the different CH\textsubscript{4}:H\textsubscript{2} treatment period were the diffraction of Fe. The other peaks were well matched with iron carbide (Fe_{3}C) diffraction (ICDD no. 01-077-0255). From TEM, C-plane on top of nanonod was not well aligned while C-plane on the side wall, however were well aligned parallel to the core of nanorod. Dark Field Image technique was used to observe nanorods in longitudinal and vertical direction. Bright and dark observation meant that C-plane were well aligned. The core of nanorod diffraction ring revealed d space similarly to iron carbide. The rods were vertically oriented around the middle of substrate agreed with the effect of current flow line [8] which was perpendicular to the surface of electrode. The rods on the upper, lower, left and right outer side were oriented to their outer direction because the effect of equipotential.

References

Fig. 1. TEM observation of nanorod filled iron carbide.

Fig. 2. Crystal structure of iron carbide.

Fig. 3. TEM revealed well-aligned nanorod.

Fig. 4. Overall structure of nanorod.

Fig. 5. The growth mechanism of nanorod filled iron carbide.