



The characteristics of carbon nanotubes grown at low temperature for electronic device application



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ABSTRACT

For the application of carbon nanotubes (CNTs) in flexible electronic devices, the CNTs were grown on Corning 1737 glass substrate by microwave plasma enhanced chemical vapor deposition (MPECVD) method. To deposit the catalyst layer, TiN buffer layer of 200 nm thickness and Ni catalyst layer of 60 nm were first deposited on the glass by r.f. magnetron sputtering method. The CH₄ and H₂ gases are used as the synthesis gas of CNTs and the working pressure was about 2.13 kPa, and the substrate bias was about –200 V. The growth time was from 2 min to 5 min and the microwave power was about 800 W. The substrate temperature as the main parameter was changed from 400 °C to 550 °C. The structural properties of CNTs synthesized with the substrate temperature were investigated using Raman, field emission scanning electron microscopy, and transmission electron microscopy methods. The surface and electrical properties of CNTs grown by MPECVD method were studied by scanning probe microscopy and four-point probe methods. We obtained the multi-walled CNTs (MW-CNTs). Multi-walled CNTs were vertically grown on Ni/TiN/glass substrates below 500 °C without any glass deformations. As the substrate temperature was increased, the crystallinity of CNTs was improved. Ni catalyst was found at the tip of CNT by the TEM observation and the grown CNTs were found to have a multi-walled with bamboo like structure.

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1. Introduction

Carbon nanotubes (CNTs) that consist of sheet(s) of graphene (a hexagonal lattice of carbon) rolled into a cylinder, were discovered in 1991 by Iijima [1]. Numerous investigators have reported CNTs, its development of commercial applications such as scanning probes [2], field emission display (FED) [3], anode for lithium ion batteries [4], nanoelectronic devices [5], supercapacitors [6], molecular sensors [7], and hydrogen storages [8] have been stimulated tremendously. Several methods, including arc-discharge [9], chemical vapor deposition (CVD) with [10–12] or without plasma enhancement [13,14], and laser ablation [15], have been used for the growth of CNTs in the presence of the catalyst. For CVD process, CNTs can directly grow on the predefined catalyst layer, and have high yield and uniformity [16]. For FED application, the growth temperature should be below 550 °C to avoid the deformation of the sodalime glass substrate [17]. Recently, many studies for low temperature growth of multi-wall CNTs by CVD methods have been reported [18–20].

In this work, we have synthesized vertically aligned CNTs on the glass substrate at low temperature (<550 °C). The effect of the substrate temperature on the properties of CNTs grown by microwave plasma

enhanced chemical vapor deposition (MPECVD) with methane (CH₄) and hydrogen (H₂) gas was investigated.

2. Experimental details

The CNTs were catalytically grown on Corning 1737 glass substrate by the MPECVD system with CH₄/H₂ as source gases. No deformation or change in material properties of glass substrate was observed during or after the whole process. The sputtered TiN film of 200 nm was employed as the buffer layer in this work. Ni (60 nm) was applied to catalyze the growth of CNTs on the TiN/glass substrate by the sputtering method. CNTs were synthesized by the following procedure: (1) pretreatment with H₂ plasma for 10 min to make well-distributed nano-sized catalysts, at a pressure of 2.13 kPa and a microwave power of 800 W, and (2) the introduction of CH₄/H₂ (10:80 sccm) process gases at a pressure of 2.13 kPa and a microwave power of 800 W. The substrate temperature was varied from 400 to 550 °C during the growth of CNTs. The growth time was changed from 2 to 5 min. Table 1 presents the synthesis conditions of CNTs.

Synthesized CNTs were subjected to a number of analysis including field emission scanning electron microscopy (FE-SEM) [Jeol, JSM-6700 F], high resolution transmission electron microscopy (HRTEM) [FEI Tecnai G2 200 kV], and Raman spectrometer [Jasco, MRS-3000]. The sheet resistance of CNTs was measured by using four-point probe.

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Table 1
CNT growth conditions.

Parameters	Conditions
Substrate	Glass (Corning 1737)
Gas flow rate	CH ₄ : 10 sccm H ₂ : 80 sccm
Working pressure	2.13 kPa
Microwave power	800 W
DC bias voltage	−200 V
Growth time	2 ~ 5 min
Substrate temperature	400 ~ 550 °C

3. Results and discussion

Fig. 1 shows the morphologies of CNTs grown at different substrate temperatures for 3 min. The SEM images exhibit clearly that the substrate

temperature highly affects the surface morphology of the CNTs. As the substrate temperature is increased, the length and the diameter of CNTs varied markedly. The CNTs grown at low substrate temperature are curly, suggesting CNTs to be highly defective. This is expected since the growth temperature is very low, i.e., carbon atoms deposited at the edge of the tubes do not have enough time to diffuse, forming defective edges with pentagons and heptagons which will eventually induce bending in nanotubes [21]. As shown in Fig. 2, the vertically well-aligned CNTs are grown and the length of CNTs was increased with increasing substrate temperature. The longer CNTs at higher temperature are probably due to the enhanced diffusion and reaction of carbons [22]. The CNT grown at 550 °C are well aligned perpendicular to the substrate, quite long about 3.5 μm , and highly dense, as shown in Fig. 1(d). However, some carbonaceous particles were formed on top of CNTs.

Fig. 3(a) shows the Raman spectra of CNTs obtained at different substrate temperatures. The spectra clearly show strong peaks at

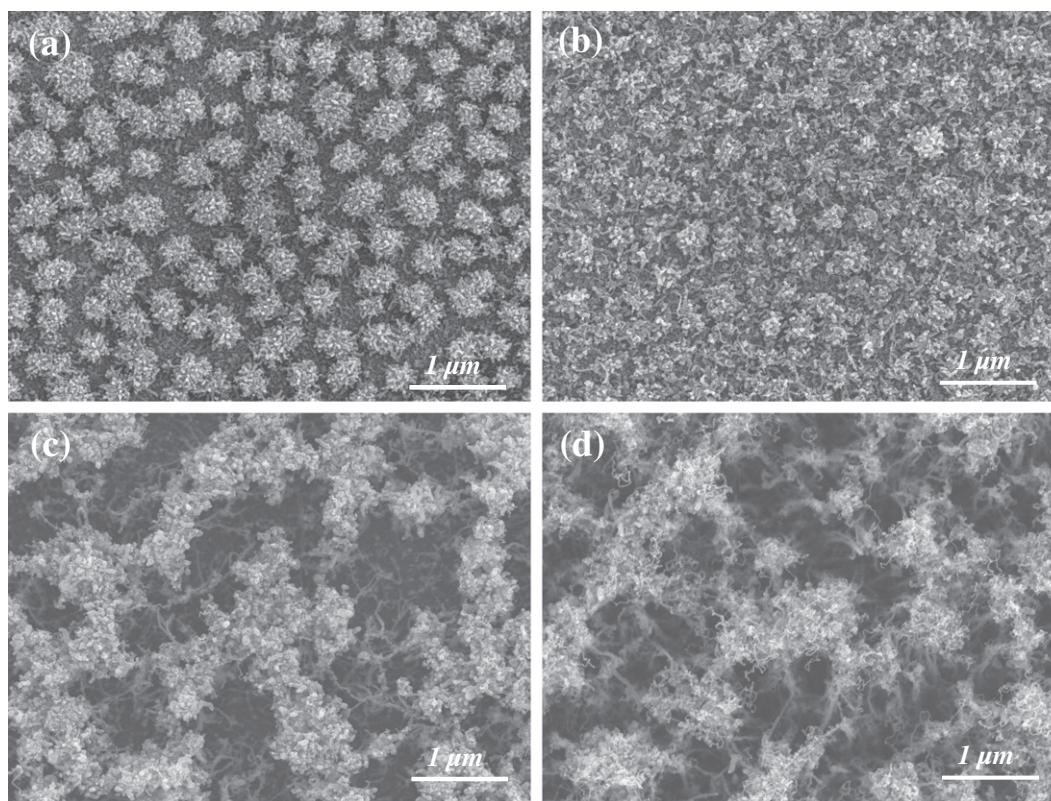


Fig. 1. FESEM micrographs of CNTs grown with d.c. bias voltage of −200 V at various substrate temperatures: (a) 400 °C; (b) 450 °C; (c) 500 °C; and (d) 550 °C.

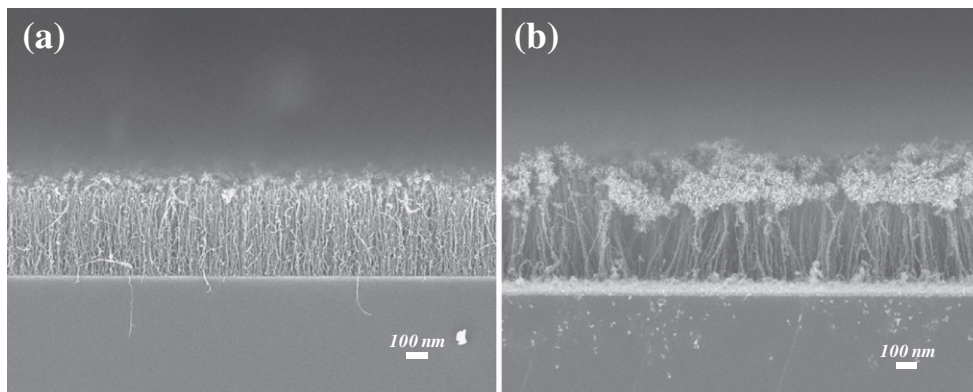


Fig. 2. Cross-sectional FESEM images of CNTs grown at substrate temperatures of (a) 400 °C and (b) 550 °C.

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