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Growth of carbon nanotubes on stainless steel substrates by DC-PECVD

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ABSTRACT

We report on the fabrication of carbon nanotubes (CNTs) on Ni-coated stainless steel (SUS) substrates by using dc plasma enhanced chemical vapor deposition. The synthesized CNTs have the diameter of about 30 nm and the length of about 1.2 μm. To verify the effects of SUS substrates on the growth of CNTs, CNTs had also been grown on Ni-coated Si substrates. CNTs grown on the SUS substrates were more uniform compared with those grown on the Si substrates. Field emission properties of the CNT films were measured in the diode configuration, and the turn-on electric field of 3.87 V/μm and field enhancement factor β of about 1737 were obtained from the synthesized CNTs at the gap of 500 μm between the SUS substrate and the anode. These results have not only clarified the effects of the substrate on the growth of CNTs, but also shown the potential of CNTs in field emission applications, especially CNT-based cold-cathode X-ray tubes.

1. Introduction

Since the discovery of carbon nanotubes (CNTs) in 1991 [1], they have attracted considerable interest because of their unique physical properties and many potential applications. With the nanometer-size diameter and very large aspect ratio, CNTs exhibit unique electronic properties such as excellent electron emission efficiency and extraordinary mechanical properties. They are the potential building blocks for field emission displays, tips for scanning probe microscopy, X-ray sources using field emission cathode, hydrogen storage, chemical sensors, high-strength mechanical composites, etc. [2–5]. It is reported that X-ray sources with field emission cathodes have several intrinsic advantages over thermionic X-ray tubes, including low temperature, instantaneous response, and the potential for miniaturization. Field emission X-ray tubes with CNT emitters [6] had recently been demonstrated to have significantly improved properties compared to those with metal [7] or diamond tips [8]. For field emission X-ray tubes with CNT emitters, the synthesis of CNTs directly on metallic substrates will greatly simplify the preparation of cold cathodes. However, it has been reported that the synthesis of CNTs on metallic or electrically conducting substrates is rather difficult compared to that on insulators such as glass or silicon wafers. It has been attributed to the high mobility and lack of localization of carbon atoms on metallic surfaces, or to the difficulty of catalyst island formation due to the diffused reaction and interfacial bonding between the catalytic layer and the metallic surface at the growth temperature [9]. Although metal substrates, in recent years, have been more frequently researched [10–12], their effects on growth of CNTs have not been fully understood.

In this research, we report the growth of carbon nanotubes on Ni-coated stainless steel (SUS) substrates by dc plasma enhanced chemical vapor deposition (DC-PECVD). The synthesized CNTs have the diameter of about 30 nm and the length of 1.2 μm, which are more uniform compared with those grown on Ni-coated Si substrates. It is attributed to the higher uniformity of Ni catalyst particles on the SUS substrates after the pretreatment with NH_3 plasma, compared to those on the Si substrates. Field emission properties of the CNT films were measured in the diode configuration. The turn-on electric field of 3.87 V/μm and the field enhancement factor β of about 1737 were obtained from the synthesized CNTs at the gap of 500 μm between the SUS substrate and the anode.

2. Experimental

CNTs had been grown on Ni-coated SUS substrates with a TiN buffer layer by dc plasma enhanced chemical vapor deposition (DC-PEVCD). The Ni layer and the TiN buffer layer with a thickness

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of 50 and 1000 Å, respectively, were deposited by using a radio-frequency magnetron sputtering system. It has been reported that the formation of Ni grains during the pretreatment process plays a key role in growing CNTs; when Ni forms alloys such as NiFe, NiCr, etc. (with SUS substrates) or $NiSi_2$ (with Si substrates), other isomers of carbon such as carbon nanotips are formed instead of CNT [13], and therefore TiN buffer layers with excellent electrical conductivity (resistivity ~25 mΩ cm) and high melting point (~3200 °C) [9] are usually added to prevent the reaction between catalyst layers and substrates [14]. To create uniform Ni particles, NH_3 gas was introduced for 6 min. During this process, the cathode voltage, the temperature, and the flow rate were kept at −550 V, 600 °C, and 60 sccm, respectively. The base pressure of the reactor was maintained 3.4×10^{-6} Torr. Before the CNT growth, we have performed annealing procedures at the temperature of 600 °C in H_2 environments. After the pretreatment and annealing processes, CNTs were grown at 600 °C for 15 min using a mixture of acetylene and ammonia with the flow rates of 30 and 100 sccm, respectively. To examine the effects of SUS substrates on the growth of CNTs, CNTs were grown also on Ni-coated Si substrates at the same synthesizing conditions as above.

The morphology, density, and quality of the CNTs were analyzed using field emission scanning electron microscope (FESEM, Hitachi S-4800), a high resolution transmission electron microscope (HRTEM, JEM 2200FS), and Raman spectroscopy (Ar^+ laser 514 nm, 2.42 eV), respectively. The morphology of Ni particles and Ni catalyst films were investigated using an atomic force microscope (AFM). The field emission properties of the CNT films were measured in the diode configuration in a vacuum chamber with pressure below 3.0×10^{-7} Torr. The anode was a Mo electrode, and the gap between the SUS substrate and the anode was 500 μm.

3. Results and discussion

Fig. 1(a) and (b) shows the AFM images of the Ni catalyst layers on SUS and Si substrates after the NH_3 plasma pretreatment, respectively. The catalyst layers on both substrates were observed to aggregate and form Ni nanoparticles. However, Ni nanoparticles on SUS substrates (Fig. 1(a)) seem to be more uniform in size than those on Si substrates: most nanoparticles on SUS substrates have diameter from 20 to 40 nm while those on Si substrates have from

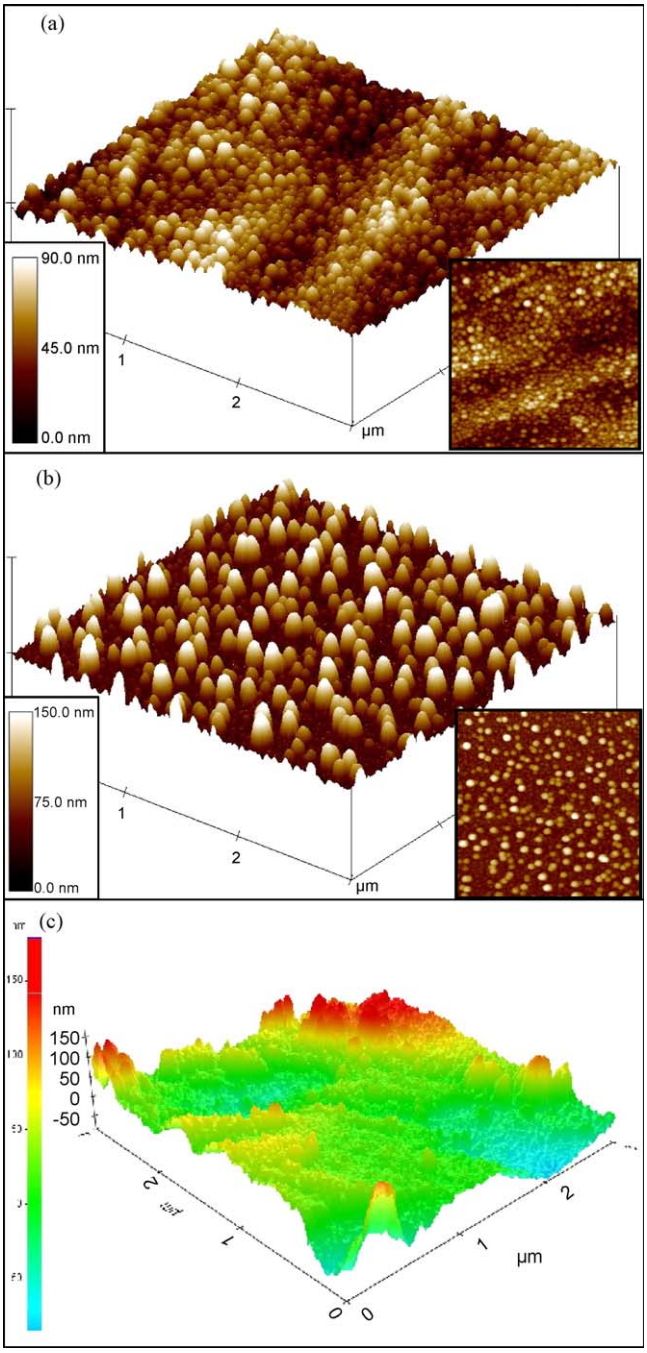

Fig. 1. Typical AFM images of Ni catalyst layers (a) on SUS substrates and (b) on Si substrates after the NH_3 pretreatment process, and (c) on SUS substrates before the NH_3 pretreatment process.

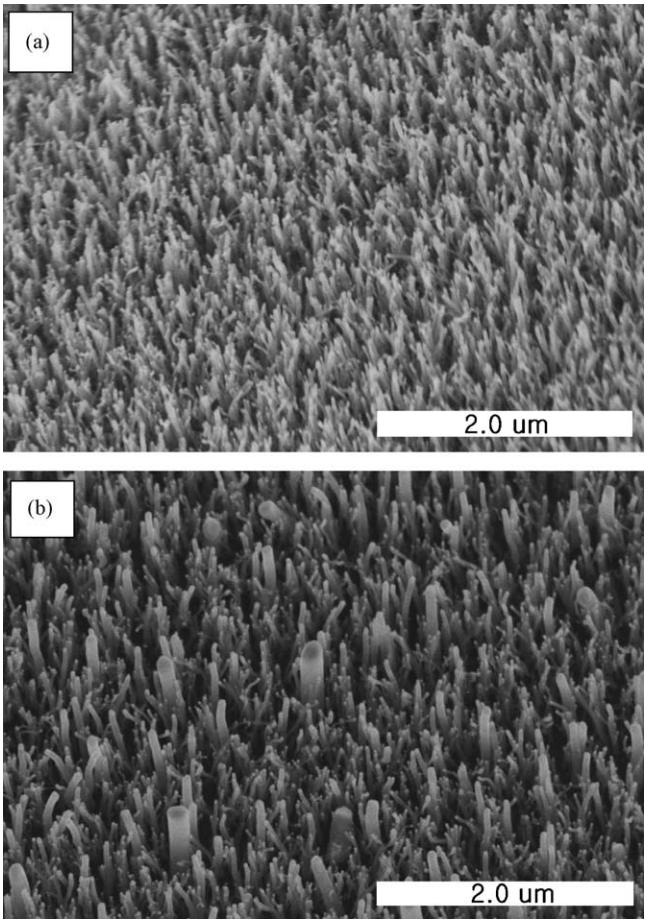

Fig. 2. Typical SEM images of CNTs grown (a) on SUS substrates and (b) on Si substrates.