Microstructure Analyses of Metal-Filled Carbon Nanotubes Synthesized by Microwave Plasma-Enhanced Chemical Vapor Deposition

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Abstract—Pd/Co-based metal-filled carbon nanotubes (MF-CNTs) were synthesized by a microwave plasma-enhanced chemical vapor deposition method using a bias-enhanced growth technique. Pd/Co-based MF-CNTs were analyzed by scanning electron microscopy (SEM), transmission electron microscopy (TEM) electron energy loss spectroscopy (EELS), and Raman spectroscopy. MF-CNTs were well-aligned and uniform in size on a Si substrate. Both multiwall nanotube carbon nanotubes (CNTs) and herringbone (or stacked cups structure) structures were observed. High-resolution TEM revealed that MF-CNTs were composed of highly ordered graphite layers, and the elemental maps of EELS indicate that both Co and Pd metals are present inside the nanotubes. TEM results clearly showed that both Pd and Co metals were successfully encapsulated into the CNTs. We observed a low value for the Raman intensity ratio between D (1355 cm^{-1}) and G (1590 cm^{-1}) bands with no shift of the G-peak position and no broadening of the G-peak, indicative of high-quality Pd/Co-based MF-CNTs. Based on TEM characterization, we propose a description for the encapsulating mechanisms.

Index Terms—Electron energy loss spectroscopy (EELS), metal filled carbon nanotubes (CNTs), microwave plasma-enhanced chemical vapor deposition (MP-CVD), scanning electron microscopy (SEM), transmission electron microscopy (TEM).

I. INTRODUCTION

THE INTRODUCTION of various materials, such as metals, their compounds, or fullerenes, into carbon nanotubes (CNTs) may lead to a variety of industrial applications. CNTs filled with ferromagnetic metals such as Fe, Ni, or Co have significant potential in high-density magnetic storage devices due to their size, anisotropic behavior, and enhanced magnetic coercivities [1]–[6]. In addition, the graphite sheets provide an effective barrier against oxidation, and consequently

ensure a long-term stability of the ferromagnetic core metal. Moreover, CNTs filled with metals are an effective route to exploit one-dimensional nanocables in various applications [7].

Previously, different groups have tried to synthesize metal-filled CNTs (MF-CNT) and two kinds of methods have been reported so far. One approach has the CNTs filled with molten material through capillary action [8] or with metal oxides using wet chemical techniques after opening the tube top [9]. The other is an *in-situ* filling method, where metals or metal compounds can be filled into the CNTs by the arc-discharge technique using a graphite anode impregnated with the filling material [10]. Recently, the MF-CNTs by a thermal chemical vapor deposition (CVD) method has also been reported [1]–[6]. Although thermal CVD can produce MF-CNTs in large quantities, it is difficult to produce vertically aligned MF-CNTs. From an application view point, it is desirable to use plasma-enhanced CVD to produce MF-CNTs with highly aligned and well-defined diameters and length. Although we have previously reported Pd-based MF-CNTs using bias-enhanced microwave PECVD (BE-MPECVD) [11], we failed to fill Co metal into the nanotubes using only a Co catalyst layer on the Si substrate. The Co particle was detached from the substrate and remained at the tip of the growing Co-based CNTs. In order to realize spin electronic applications by using MF-CNTs, it is required to fill the ferromagnetic metals into the tubes of aligned CNTs.

In this paper, we report the Pd/Co-based MF-CNTs synthesized by a BE-MPECVD using a combination of Pd and Co catalyst layers. BE-MPECVD grown MF-CNTs were characterized in detail by scanning electron microscopy (SEM) and transmission electron microscopy (TEM), as well as energy filtered electron energy loss spectroscopy (EELS), and their growth mechanisms were modeled to describe these results. We also carried out visible Raman spectroscopy to compare the microstructure between Pd/Co-based MF-CNTs and Pd-based MF-CNTs or Co-based hollow nanotubes (CNTs).

II. EXPERIMENTAL

The MF-CNTs were grown by MPECVD using a 2.45 GHz, 1.5 kW microwave power supply. The substrate temperature was controlled by a radio frequency graphite heater. A negative bias was applied to the substrate holder to grow vertically aligned MF-CNTs on the substrate. The Si substrates with a thin native oxide were cleaned in acetone by ultrasonication followed by deionized water and, finally, dried using a nitrogen

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blower prior to the catalyst deposition. The substrates were then treated in an acid solution ($\text{H}_2\text{SO}_4 : \text{H}_2\text{O}_2 = 4 : 1$) to enhance the thickness of the oxide layer. Thin films of Co (6 nm) and Pd (9 nm) were deposited on Si substrates by a thermal evaporation. After deposition of the catalyst layers, the substrates were transferred in air to the CVD chamber. The chamber was pumped down to a base pressure of 10^{-2} Torr. The substrates were heated to 750°C and held at this temperature for 10 min to sinter the catalyst layers. The feed gas, methane (CH_4), was introduced immediately and the concentration of H_2 gas was adjusted to achieve a CH_4/H_2 ratio of 50/50 sccm at a total pressure of 20 Torr. A microwave power of 600 W was employed and a negative bias was applied to the substrate holder to grow vertically aligned MF-CNTs on the substrate. The MF-CNTs were grown for 10 min under the conditions where a negative bias of 400 V was applied.

The MF-CNTs were observed using scanning microscopy (Hitachi S-300, FEI Philips XL30 sFEG) and analyzed using a Philips CM200TEM with a LaB₆ filament, fitted with a GIF2000 spectrometer. The Raman spectra were measured in the back-scattering geometry using the 514.5 nm line of an Ar⁺ ion laser at room temperature in the spectral range from 900 to 1800 cm^{-1} with a resolution of 1.0 cm^{-1} .

III. RESULTS AND DISCUSSION

Fig. 1(a) shows the SEM images of the Pd/Co-based MF-CNTs grown on Si substrates, and in Fig. 1(b), the evolution of the catalyst after just 3 min growth of MF-CNTs. The final MF-CNTs are well aligned and uniform in size on the Si substrate. At lower negative substrate biases below 300 V, randomly oriented sparse CNTs (not MF-CNTs) were observed. This result indicates that a high negative substrate bias above 400 V is necessary to obtain well-aligned and dense Pd/Co-based MF-CNTs. Fig. 1(a) shows that MF-CNTs are well aligned with uniform diameter and length ($1\text{ }\mu\text{m}$) on Si substrates. It is seen from Fig. 1(b), that the Pd/Co breaks into small size (about 60–100 nm diameter) uniformly due to surface tension, as well as the stress due to mismatch of the thermal expansion coefficients during the initial growth stage. For the case of only Co catalyst layers, the particle was found to form spherical islands; different from that observed for the Pd/Co catalyst layers. The catalyst for the growth of Pd/Co-based MF-CNTs should be in a molten state to encapsulate metals inside the tubes. The temperature of growth of Pd/Co-based MF-CNTs in our case is far lower than the melting point of bulk Pd (1552°C) and Co (1495°C). It is well known that the surface-to-volume ratio with respect to nanosize particle can lower the melting point. However, Pd/Co-based MF-CNTs can grow even from particles with diameters from 60–100 nm.

TEM images revealed both multiwall CNTs (MW-CNTs) and the herringbone (HB) or the stacked cups structure structures on the same substrate, as shown in Fig. 2(a). In both structures, there is the presence of the HB structure. It is reported that the formation of HB-shaped CNTs could be attributed to nitrogen doping, in which nitrogen atoms are generally bonded to carbon atoms in sp^2 pyridine-like and sp^3 bridgehead-nitrogen types [12]. Residual nitrogen may be a nitrogen precursor for

Fig. 1. (a) Scanning electron microscopy images of Pd/Co-based MF-CNTs grown on Si substrates and (b) the evolution of the catalyst of Pd/Co layers after 3 min growth of MF-CNTs.

Fig. 2. (a) Transmission electron microscopy image of Pd/Co-based MF-CNTs grown on the Si substrate and (b) Co elemental map inside tubes measured by energy filtered electron energy loss spectroscopy. Metal encapsulation is clearly visible in Fig. 1(b).

the synthesis of HB-shaped CNTs. The exact reason for existing both the tube with HB and the HB structures in the same substrate is not known up to now. Combining the TEM image [Fig. 2(a)] and the EELS elemental map of Co [Fig. 2(b)], the Co metal was successfully filled inside the MW-CNTs. In the case of HB-shaped structure, Co metal was encapsulated only in the tips. A magnified HRTEM images, as shown in Fig. 3, reveal that the interlayer spacing of a graphite layer is 0.34 nm, consisting with the (002) plane lattice parameter of graphite.

Fig. 3. High-resolution transmission electron microscopy images of Pd/Co-based MF-CNTs grown on the Si substrate.

The graphite layers are parallel to the tube axis. Although some amorphous material was observed inside the tube walls for the Pd-based MF-CNTs [11], almost no amorphous material is observed for Pd/Co-based MF-CNTs. Pd metal is generally considered as an inactive metal and Co metal is considered as an active metal in catalyst. Therefore, the reactivity of the catalyst is enhanced by combining Co with Pd and leads to a reduction of the amorphous nature inside the tube walls for the Pd/Co-based MF-CNTs. A large number of linear contrast features were observed around the tip for Pd-based MFCNTs due to a lattice strain induced by the solidification of Pd during cooling after growth [11]. In contrast to the Pd-based MF-CNTs, no linear contrast around the tip for Pd/Co-based MF-CNTs. This indicates no lattice strain was induced by the solidification of Pd/Co during cooling after growth.

EELS analysis was performed to identify the composition of the material inside the CNTs, as shown in Fig. 4. On the basis of elemental analysis, we have observed not only Pd and Co but Si and Mo (not shown in Fig. 4) elements. Si and Mo might be incorporated into the metal within the CNTs from the Si substrate. In the previous study, we have also observed Si and Mo atoms for the Pd-based MF-CNTs [11]. The elemental maps also clearly reveal that both Pd and Co metals are found inside tubes uniformly. This result indicates that combining Co with Pd is essential to fill Co metal inside tubes for producing Co filled CNTs.

The first-order Raman spectra of MF-CNTs consists of two broad peaks located at 1590 cm^{-1} (the G peak) and 1355 cm^{-1} (the D peak), as shown in Fig. 5. In comparison with Pd/Co-based MF-CNTs, the Raman spectra of Pd-based MF-CNTs reported previously [11], and multiwall

Fig. 4. C, Co, Pd, and Si elemental maps inside tubes measured by energy dispersive X-ray spectroscopy.

CNTs (MWCNTs) grown using Co catalyst particles where the catalyst metal was not filled inside the tubes are shown in Fig. 5(a) and (b), respectively. The G and D peaks are attributed to the in-plane symmetric C-C stretching (E_{2g}) and the graphite materials with small crystallite sizes or so-called disordered graphite, respectively. Raman spectra show an additional peak at about 1620 cm^{-1} (denoted as D'). The origin of the D and D' bands in other forms carbon materials has been explained as disorder-induced features, caused by the finite-particle size effects or lattice distortion [13]. Although, the Raman intensity ratio between D and G bands (I_D/I_G) of Pd/Co-based MF-CNTs is always larger than that from the MWCNTs in the experiment, a low intensity of the D band, as well as a low value of the I_D/I_G , indicates high quality of Pd/Co-based MF-CNTs, as shown in Fig. 5(c). While the broadening of G -peak was observed for Pd-based MF-CNTs [11], the full-width at 1/2 maximum (FWHM) of the G -peak for Pd/Co-based MF-CNTs was almost the same as that of MWCNTs. Furthermore, no shift of the G -peak position was observed indicating that the structural change from graphite to

Fig. 5. Raman spectra of Pd-based MF-CNTs, Co-based CNTs multiwall CNTs, and Pd/Co-based MF-CNTs.

Fig. 6. Summary of the proposed growth model of Pd/Co-based MF-CNTs.

nanocrystalline graphite may not be occurring by filling Pd/Co metals inside tubes [14].

Most reports so far, conclude that the catalyst particles are encapsulated on the tip of the MWCNTs. The question arises if there is any reason preventing the metallic catalyst present inside the MF-CNTs. Based on TEM and SEM investigations, we propose the following model to explain the Pd/Co-based MF-CNTs, as schematically shown in Fig. 6. After Pd and Co layer deposition, the mixture of Pd and Co catalyst layers

become fragmented into nanoparticles Fig. 6(a). The decomposition of CH_4 on the surface of Pd/Co nanoparticle results in the formation of bimolecular hydrogen and carbon atoms, and the growth of Pd/Co-based MF-CNTs is believed to occur via diffusion of carbon atoms through the Pd/Co particle leading to the formation of graphite layers at the lower colder zone of the particle. The axis of a MF-CNT growing perpendicular to the substrate coincides with the direction of the applied electrostatic force, resulting in a uniform tensile stress across the entire graphite layer/catalyst particle interface [Fig. 6(b1) and (b2)]. Therefore, carbon uniformly precipitates across the interface and the CNT continues to grow vertically (perpendicular to the substrate) [16]. The key feature of this growth model for the tube with HB is that the precipitation of carbon atom does not occur on the bottom surface of the catalyst particle [Fig. 6(b1) and (b2)]. Therefore, the Pd/Co catalyst metals are trapped in the CNT from the basal side when the CNT was filled by Pd and Co metals [Fig. 6(c1)]. On the contrary, the precipitation of carbon atom occurred on the bottom surface of the catalyst particle leads to elevation of the particle to the tip [Fig. 6(c2)]. If the carbon species are supplied in a steady-state manner, the tube will grow vertically for the tube with HB structure, and the HB structure will appear periodically [Fig. 6(c1) and (c2)]. The growth continues until the catalytic activity of the Pd/Co. In previous reports, metals were considered to be encapsulated in the hollows of CNTs by the capillary force [8], [15]. Although the capillary force was also active in our experiments, encapsulation was thought to start at the beginning of growth of MF-CNTs.

IV. CONCLUSION

In conclusion, we report the synthesis and characterization of Pd/Co-based MF-CNTs. We have successfully grown well-aligned Pd/Co-based MF-CNTs with uniform diameter and length (1 μm) on Si substrates. TEM revealed multiwall nanotubes and the HB or stacked cups structure on the same substrate. HRTEM revealed that MF-CNTs were composed of highly ordered graphene layers. Based on TEM observation and analysis by EELS showed that Co metal was successfully encapsulated into the CNTs from the basal side by combining Co with Pd metals. Raman results showed a low intensity of the D band, as well as a low value of the I_D/I_G indicate the high quality of Pd/Co-based MF-CNTs. Moreover, no broadening and no shift of the G-peak were observed indicating that the structural change from graphite to nanocrystalline graphite may not be occurred by filling Pd/Co metals inside tubes. A growth model for the Pd/Co-based MF-CNTs is proposed based on these experimental results. The key feature of the growth model for the tube with HB structure is that the precipitation of carbon atom does not occur on the bottom surface of the catalyst particle. Finally, owing to the unique properties of the Pd/Co-based MF-CNTs, they have potential application for tips of scanning probe microscopy or high-density magnetic storage devices.

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