big and the wetability is good. Whereas the MoS_2 powder, produced by ball milling, is spheroidal, the surface energy is low and the wetability is poor. Between the powder and substrate or between the powder and powder, the bonding is not very good so that friction causes the powder to easily flake off. Therefore the tribological properties of the MoS_2 coating were not as good as for the graphite coating.

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Synthesis of high quality single-walled carbon nanotubes at large scale by electric arc using metal compounds

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Many prospects of applications of single-walled carbon nanotubes (SWNTs) [1–3] rely on the development of a cost-effective and large-scale production of high-quality SWNTs. Among all the available synthetic techniques [4–7], the electric arc (EA) discharge method is one of the most efficient techniques for large-scale synthesis of as-prepared (AP) SWNT soot.

Since the initial breakthrough in the large-scale EA synthesis [4], significant efforts have been directed toward improving the synthesis of AP SWNT by optimizing parameters such as the buffer gas [8], electrode composition [9] and geometry [8,10] and the temperature field [11]. It is interesting to note that although various metal catalysts were studied using CVD method [3,5,6], so far all the reports for EA method have been using element metal(s) for the synthesis of SWNTs [9]. In this letter we report the application of metal compounds including metal oxides and/or their carbonates as catalyst to produce SWNTs using EA method with high quality at large scale.

In our experiments, we firstly tested a catalyst from a mixture of NiO and Y_2O_3 with 4.2:1 atom (at) % Ni:Y. The consumable anodes were prepared by finely mixing the ground catalyst with the carbon source materials on the basis of a procedure described in the literature [9]. The electrodes were 12–20 mm in diameter and 200–250 mm in length. All of the EA experiments were carried out under a He buffer gas at a pressure of

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530–550 Torr and the arc discharge was created by a current of 90–120 A with a distance of ~3 mm maintained between the electrodes. Typical run time was 30–150 min, leading to the production of ~10–50 g of AP SWNT soot per anode, depending on the electrode size and length.

The EA product from our experiments consists of three major parts as reported earlier [4,9,10]: low-density web-like material that spreads across the chamber (web), a more dense material that condenses on the wall of the chamber (chamber); and a grayish deposit that forms on the cathode and falls on the floor of the chamber (deposit). The deposit contains only a few SWNTs. Both the web and chamber fractions were evaluated using Raman spectroscopy, thermogravimetric analysis (TGA), near-IR spectroscopy (NIR), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The yield of the AP SWNT product, defined as the ratio of the weight of web and chamber fractions to the total weight of material collected, was in the range of ~45–50%. The web fraction comprises 10–25% of the total mass of the AP SWNT soot.

Fig. 1 shows the typical Raman spectra of the web fraction SWNTs prepared from the new catalysts. A strong and a weak shoulder peaks appear in the high-frequency region at ~1584 and ~1560 cm^{-1}, respectively, which are associated with the *G*-band. In the low-frequency region, there are two main peaks at ~147 and 162 cm^{-1}, assigned to the band of the radial breathing mode (RBM). According to the relationship between the RBM frequency with the tube diameter [12], $d = 234/(\omega - 10)$, these two main peaks correspond to the SWNT diameters of 1.70 and 1.54 nm, respectively. The distinctive left shoulder peak at 1560 cm^{-1} and low intensity of the *D*-band around 1336 cm^{-1} ($G/D > 80$) indicate high purity of the AP SWNTs. The chamber fraction gave almost the same Raman spectra as the web fraction except for a lower *G*/*D* value.

Fig. 2 shows the typical SEM images of the AP SWNTs from the web (Fig. 2A) and chamber (Fig. 2B) fractions. It can be found that the web fraction contains very little impurity and the SWNTs are made up of tangled bundles. The diameter and size of the SWNT bundles were investigated with high-resolution (HR) TEM and it was found that the diameter range of SWNTs was ~1.3–1.8 nm and the bundle size ranged from ~5–30 nm. Fig. 3 shows a HRTEM image of an isolated SWNT with a diameter of ~1.4 nm.

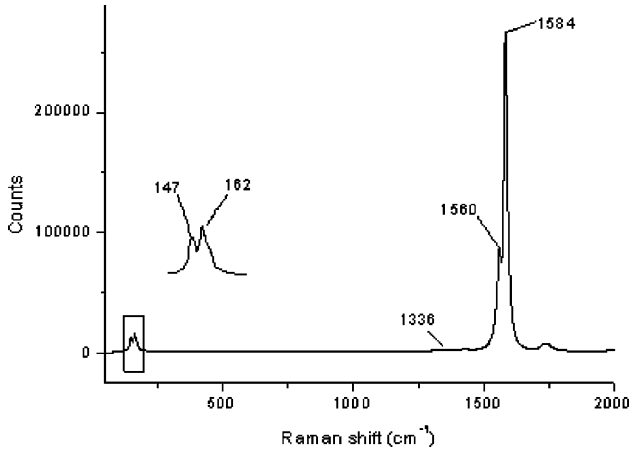

Fig. 1. Raman spectra (excited with 514.5 nm laser) of the web fraction SWNTs obtained using $NiO–Y_2O_3$ as catalyst.

The results of TGA shows that the SWNT product from this process yields an amount of ~30 wt% metal oxide residue, which corresponds to ~6 at% of metals in our SWNT product. This is similar to that of SWNT product using the conventional Ni–Y catalyst [9].

Using the solution-phase NIR spectroscopy method [13], we compared the purity of our SWNTs and that using the conventional Ni–Y catalyst and it is evident that the purity of SWNTs from this new catalyst was quite similar to that from the conventional Ni–Y catalyst and the overall purity was estimated at ~45–60 at% and the overall purity for the web fraction was in the range of ~80–85 at%. These results demonstrate that the new catalyst yields very similar and sometime

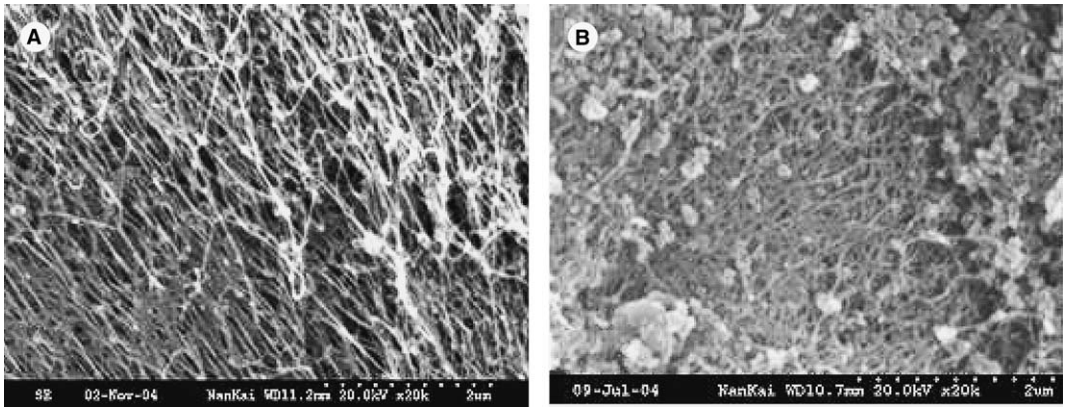

Fig. 2. Typical SEM images of AP SWNTs obtained using $NiO–Y_2O_3$ as catalyst. (A) Web fraction, 20 K, 20 kV. (B) Chamber fraction, 20 K, 20 kV.

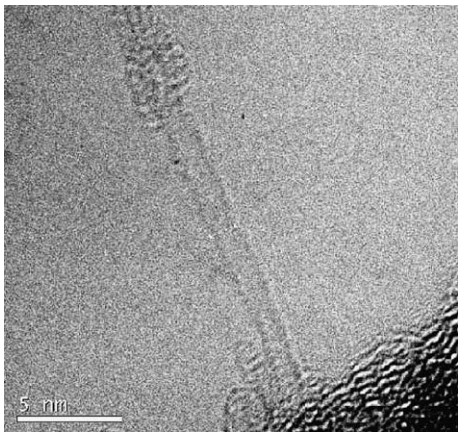

Fig. 3. HRTEM image of AP SWNTs obtained using NiO–Y_2O_3 as catalyst. Scale bar, 5 nm.

better purity and yield, compared to the conventional Ni–Y catalyst [13].

We have also tested different combinations of oxides and/or carbonates of Ni and Y with Ni:Y at 4.2:1 at%, and the results were very similar to that using NiO and Y_2O_3.

In summary, the present work shows that the use of Ni and Y compounds in place of the metal Ni–Y in the EA process leads high quality and good yield of SWNTs and has a substantial reduction in the cost of the catalyst materials. The SWNTs produced using these new catalysts are similar to those produced from Ni–Y catalyst in terms of purity, yield, diameter and bundle size. The overall savings and easily handling for the new catalysts are significant, which may warrant further investigation of the technique on an industrial scale.

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Preparation of PVC pitch from waste pipe

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