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Vertical Alignment of Carbon Nanotubes Using the Magneto-Evaporation Method

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Abstract: We developed a novel method of vertical alignment of SWNTs using a single-step process for the simultaneous vertical alignment of the SWNTs by a magnetic field and the fixation of their alignment by means of the direct evaporation of the films. We fabricated Fe−oxide/SWNT samples that are reacted by iron-oleate complex, oleic acid and cut SWNTs in 1-octadecene. The Fe−oxide/SWNT samples are dispersed in *N,N*-dimethylformamide and the resulting solution was deposited on an ITO glass substrate using the spraying method with magnetic field. After evaporation of the SWNT solution in the presence of a magnetic field, we transferred the nanotubes to the vacuum-evaporator chamber, and titanium is evaporated by e-beam evaporation to hold the vertical alignment of the SWNTs. The resulting SWNTs exhibit the formation of a high degree of vertically aligned SNWTs over a large area. We showed that the degree of orientation of the SWNTs is strongly influenced by the field strength, film thickness of the evaporating molecules and evaporating rates. This technique takes significant advantages of the alignment of SWNTs with high aspect ratio at room temperature, without any organic binders and without the need for further alignment procedures. Moreover, this method might be applicable to other anisotropic materials with high aspect ratio.

The vertical alignment of carbon nanotubes (CNTs) on device surfaces is a key issue in CNT research and represents a topic of great importance in the microelectronics industry.[1−4] This is because most CNT-based functional devices, including transistors,[5−7] sensors,[8,9] emitters,[10−13] and energy units,[14] require vertically aligned CNTs with a high length-to-diameter aspect ratio. At present, the vertical alignment of CNTs relies entirely on two different fabrication methods: (i) the conventional screen-printing technology from a paste mixture of CNTs[15−17] and (ii) direct growth by chemical vapor deposition (CVD) to fabricate vertically aligned CNTs.[18−21] However, these approaches have critical limitations. The paste technology inevitably uses additional components, such as organic binders and additives, and the resulting residues degrade the opto-electronic properties of the nanotubes. Moreover, the process requires an additional alignment of the CNTs, which includes taping, peeling, rubbing, and adhesive cleaning. Using current methods, direct

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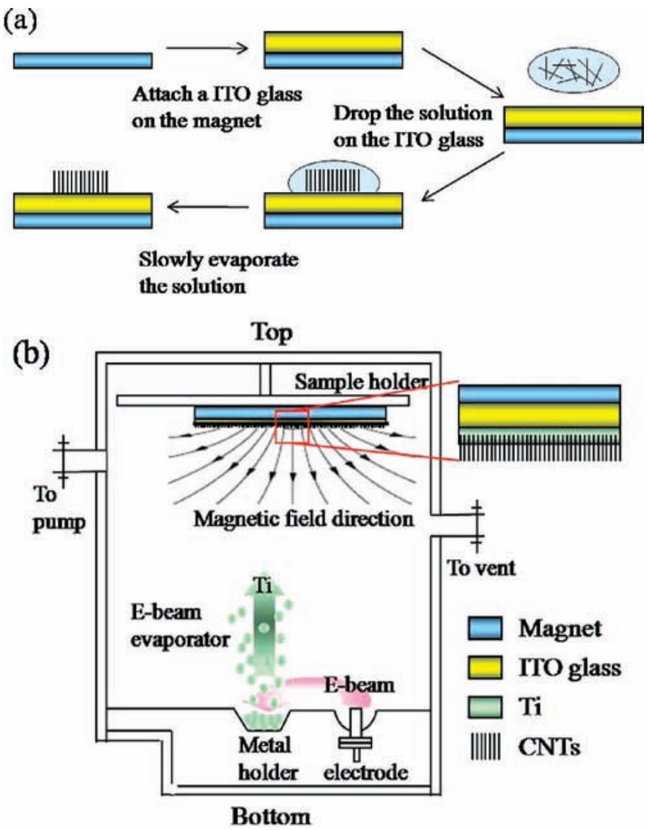

Figure 1. (a) Schematic representation of the fabrication of vertically aligned CNTs. First, we prepare a magnet and attach it to an ITO glass. Then, we drop the solution containing the CNTs (dispersed in DMF) onto the ITO surface and heat it slowly to 80 °C. (b) Scheme of the magneto-evaporation alignment (MEA) method. The vertically aligned carbon nanotubes, placed on an ITO glass attached to a magnet, are inserted into an e-beam evaporator. The aligned CNTs are fixed to a Ti metal, which is evaporated using the e-beam.

growth is inefficient because of the high temperatures and the reactive environment required, as well as the inefficient use of materials for mass production.

We report here a new method for the vertical alignment of CNTs on indium tin oxide (ITO) surfaces that is based on the direct evaporation of an evaporating molecule upon the application of a magnetic field. This procedure, designated magneto-evaporation alignment (MEA), uses a single-step process for the simultaneous vertical alignment of the single-walled nano tubes (SWNTs) by a magnetic field and the fixation of their alignment by means of the direct evaporation of the films. This strategy leads to the formation of a high density of vertically aligned SWNTs over a large area. The high degree of vertical alignment, uniformity, and stability achieved using this approach can be further verified by assessing the field-emission characteristics of the SWNT arrays. Unlike the aligning techniques reported to date, the proposed method affords the alignment of SWNTs with high aspect ratio at room temperature, without any organic binders and without the need for further alignment procedures. Thus, this aligning technique has proven to be truly cost-effective and might be applicable to other anisotropic materials with high aspect ratio. We suggest that this approach could be suitable for mass production.

The experimental setup used in the magneto-evaporation alignment method is shown in Figure 1. The as-prepared SWNTs (HiPco, Carbon Nanotechnologies Inc.) were purified using heat and an acid treatment.[22,23] They were heated at 365 °C for 90 min under an air atmosphere to remove any amorphous carbon and sonicated in hydrochloric acid for 60 min to remove the metal catalyst. After these purification steps, the clean nanotubes were cut by immersing them in a piranha solution ($H_2SO_4/H_2O_2 = 4:1$ vol/vol %) for five hours at room temperature.[24] We attached a small amount of magnetic nanoparticles, such as iron or iron oxide, onto the SWNT surfaces to obtain a system that could be well aligned by the magnetic fields. This was achieved by means of thermal decomposition (see Figure 2a).[25,26] The iron-oleate complex (as a metal precursor) was prepared by reacting iron chloride and sodium oleate at 70 °C and kept at that temperature for four hours. After completion of the reaction, the solution was cooled to room temperature and the upper organic layer—containing the iron-oleate complex—was washed three times with 30 mL distilled water in a separatory funnel. After washing, hexane was evaporated off using a vacuum pump over 12 h. The iron-oleate complex (12 g), oleic acid (2.83 g), and the cut SWNTs (150 mg) were dissolved in 1-octadecene (130 mL) at room temperature. The reaction mixture was heated to 320 °C, at a constant heating rate of 3.3 °C/min, and then kept at that temperature for 30 min. The resulting solution (containing the nanocrystals) was then cooled to room temperature and ethanol (250 mL) was added to precipitate the Fe−oxide/SWNTs. After the completion of the reaction, the solution was centrifuged at 12,000 g for one hour to separate the SWNT/Fe−oxide from the unreacted Fe−oxide nanoparticles. We obtained $\sim$ 300 mg of the precipitate SWNT/Fe−oxide and the unreacted Fe−oxide nanoparticles from the supernatant solution (see Figure 2b). The transmission electron microscopy (TEM) image (see Figure S1, Supporting Information) of the supernatant solution shows only Fe−oxide nanoparticles, indicating that most of the cut SWNTs were reacted with Fe−oxide. Thus, $\sim$300 mg of the SWNT/Fe−oxide samples are likely to contain a 150 mg of SWNTs and a 150 mg of Fe−oxide (1:1 wt %). The iron−metal precursors located within the defects of the SWNTs were thermally decomposed, thereby forming Fe−oxide nanoparticles on the SWNT surface. The resulting Fe−oxide/SWNT samples were dispersed in *N,N*-dimethylformamide (DMF) ($<$100 mg/L) by means of ultrasonication for several hours. The solution was centrifuged for one hour at 12 000*g* (to obtain only well-dispersed and separated SWNT solutions lacking flocculated SWNT particles) and the supernatant solution was collected. The resulting solution was deposited on an ITO glass substrate using the spraying method. We positioned a magnet behind the ITO substrate to apply a magnetic field to the samples (see Figure 1a).

After evaporation of the SWNT solution in the presence of a magnetic field, we transferred the nanotubes to the vacuum-evaporator chamber, which operates at a pressure of around 10^{-6}

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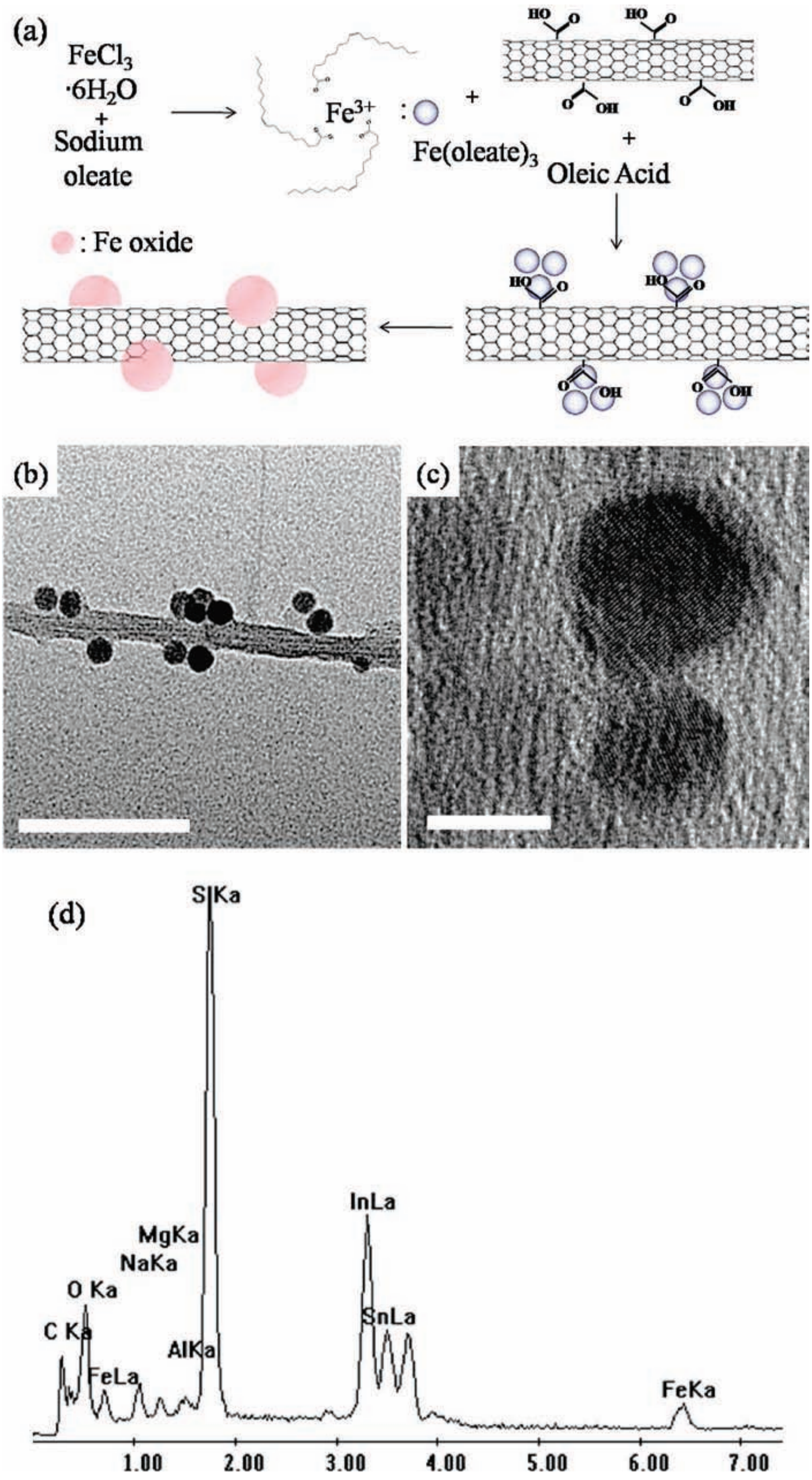

Figure 2. (a) Schematic representation of the fabrication of Fe−oxide/SWNTs. First, the metal precursors are prepared from the metal chlorides and sodium oleate. Then, these metal precursors, the cut SWNTs, and oleic acid are dissolved in a solvent and heated to the boiling point. The Fe−oxide/SWNTs are fabricated by thermal decomposition. Note that the drawing is shown schematically for single nanotubes, in reality they are bundles of nanotubes. (b) TEM images of the Fe−oxide/SWNTs (scale bar: 100 nm). (c) High-magnification TEM images showing the Fe lattice (scale bar: 10 nm). (d) Energy-dispersive X-ray (EDX) analyses of samples with Fe−oxide/SWNTs. The Fe peak is observed between 6.00 and 7.00 keV.

torr (Figure 1b). The SWNT films were positioned between the evaporation source (i.e., the electron-beam source used in this study) and the poles of the magnets, such that the magnetic-field vector and the electron-beam irradiation were mutually

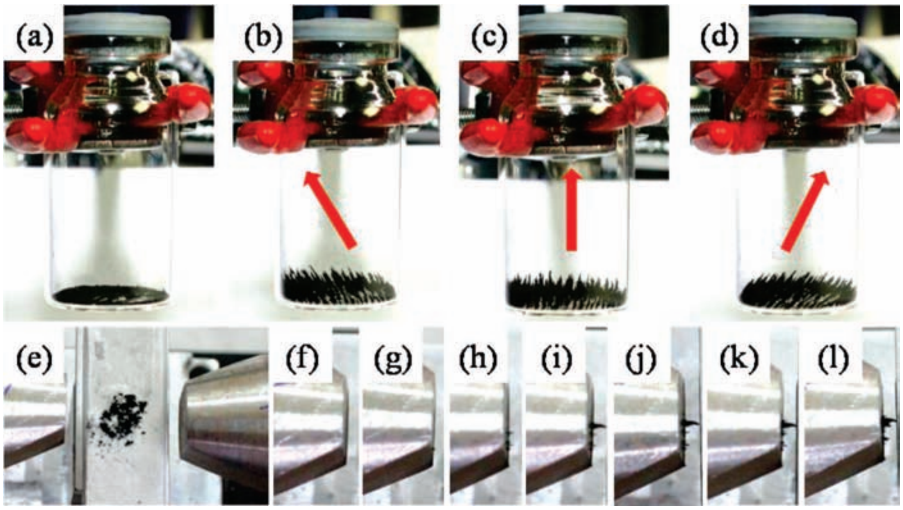

Figure 3. (a−d) Real iages showing the response of the Fe−oxide/SWNTs to applied magnetic fields of different directions. (a) No magnetic field. (b−d) Magnetic fields in the following directions: (b) left, (c) perpendicular, and (d) right. The intensity of te magnetic field is 400 gauss. The red arrows indicate the direction of the magnetic field. (e−l) Responses of the SWNTs to magnetic fields of (e) 50, (f) 100, (g) 150, (h) 200, (i) 230, (j) 250, (k) 350, and (l) 500 gauss (all going from right to left). The SWNTs show a response at strengths above 200 gauss.

parallel. We used titanium as an evaporating molecule—required to hold the alignment of the SWNTs—because titanium has a strong adhesion to both CNTs and ITO electrodes.[27] When the magnetic field was applied from a magnet placed behind the ITO substrate, the SWNTs were observed to align perpendicularly to that substrate. The field strength of the magnets and the evaporating conditions of titanium were varied to achieve a high degree of vertical alignment of the SWNTs on the ITO substrate.

The TEM images and corresponding energy-dispersive X-ray (EDX) analyses of the SWNT/Fe−oxide samples [SWNTs/Fe−oxide particles = 5:1 (vol/vol %)] clearly show that the Fe−oxide nanocrystals are formed by the thermal decomposition of Fe ions (Fe^{3+}) on the SWNT surface (see Figure 2b,c). The magnetic particles are efficiently dispersed on the nanotube surface (Figure 2b). These particles are spherical and highly monodisperse, with a mean diameter of about 15 nm, as determined from a total of approximately 300 magnetic particles in different areas of the sample and different TEM images. The number of Fe−oxide nanoparticles in the SWNT film was tuned by varying the ratio of the components in the bulk solution prior to assembly, that is, by varying the nanoparticle concentration while keeping the nanotube concentration constant. A higher magnification image shows that the measured regular *d*-spacing of the observed planes of the lattice is approximately 3.0 Å, which corresponds to the {220} planes of Fe−oxide (see Figure 2c).[28] The EDX pattern (see Figure 2d) shows typical Fe peaks in the range of 6.0−7.0 keV, further confirming the presence of Fe−oxide nanoparticles on the SWNTs.

We investigated the degree of orientation of the samples with SWNT/Fe−oxide particles = 5:1 (vol/vol %) as a functional of the magnetic-field strength at room temperature (see Figure 3). It is noteworthy that the nanoparticle concentration also affects the alignment of the SWNTs. We observed similar alignment behavior for the samples with concentration SWNT:Fe−oxide particles $\geq$20:1 (vol/vol %). To simplify the description of the experiments and the corresponding discussion, we set the concentration of SWNT/Fe−oxide particles at 5:1 (vol/vol %) and extend the conclusions to the other nanotube samples. In this way, we can investigate the effect of the magnetic field and the evaporating conditions on the alignment of the SWNTs.

No preferred orientation was observed for the samples in the absence of an applied field (Figure 3a). However, under the influence of a magnetic field, the SWNTs were found to be aligned along the direction of that magnetic field (see Figures 3b−d). To determine the threshold field strength required to align the SWNT/Fe−oxide samples, we placed them between the poles of magnets with various field strengths (ranging from 0 to 500 gauss) using a magnetic-field generator (Figures 3e−l). We found that the threshold field for aligning the samples was approximately 200 gauss (see Figure 3h) and that the alignment increased only slightly at higher field strengths up to about 230 gauss (Figure 3i). Hence, the magnetic field strength was set at about 400 gauss in all subsequent experiments. However, the SWNTs did not retain their magnetic-field-induced alignment when this field was removed.

Cross-sectional scanning electron microscopy (SEM) images clearly reveal the high degree of vertical alignment of the SWNTs prepared on the ITO substrates using the magneto-evaporation alignment method (**MEA**) (Figure 4). The SWNTs, with interspersed straight nanotubes, are vertically aligned and elongated along their axis over the entire substrate (2 cm × 2 cm). A high degree of alignment of the nanotubes is observed, this alignment being highly uniform across the whole surface. The SWNTs align perpendicular to the substrate—and simultaneously retain their own alignments—so that they stand upright on the surface. We found that the density of vertically aligned SWNTs on the substrate can be controlled by varying either the concentration of the SWNT solutions or the number of coating cycles on the substrates, thus leading to a controlled spacing between the SWNTs. Close inspection of the vertically aligned nanotubes reveals the presence of Fe−oxide nanoparticles on the surface (see arrows in Figure 4a).

Many of the vertically aligned SWNTs are either isolated or form small bundles on the ITO surface (see Figures 4a,

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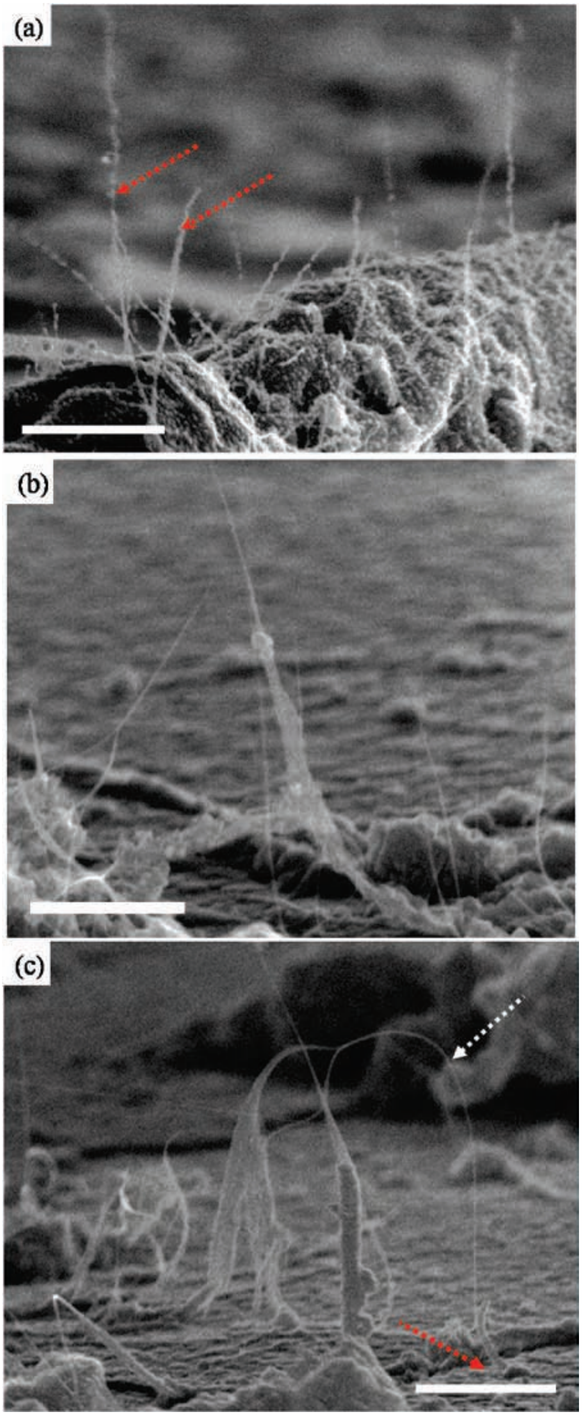

Figure 4. SEM images of vertically aligned carbon nanotubes showing the nanoparticles on the SWNT surface [red arrow, (a)] as well as many individual (or a few bundles of) SWNTs (b). The length of the SWNTs varies between 1 and 3 μm. Because of their high aspect ratio, some SWNTs are bent [see white arrow in (c)]. The red arrows in (c) indicate a strong fixation of the aligned SWNTs by the evaporating molecules [scale bars: (a), (b) 500 nm, (c) 1 μm].

b), although some aligned large bundles are also visible (see Figure 4c). Interestingly, we frequently observed vertically aligned SWNTs with a very high aspect ratio (length/diameter $\approx$ 3.0 μm:15 nm), which indicates a strong fixation of the aligned nanotubes by the evaporating molecules (red arrow in Figure 4c). The SWNTs bend at approximately 1.7 μm (see white arrow in Figure 4c), which is a result of their high bending constant.[29]

The fraction of vertical alignment of SWNTs after titanium evaporation is strongly affected by the film thickness and evaporation rate of the titanium molecules. Independent of the titanium evaporation, if the magnetic field is not applied during the evaporation, no vertical alignment occurs (see Figure S2, Supporting Information). The SEM images in Figure 5 show the degree of vertical alignment of SWNTs containing titanium layers of various thicknesses. Evaporated titanium layers with a thickness below ~10 nm cannot sustain the vertical alignment of the SWNTs (see Figure 5a). Upon increasing the thickness of this layer to about 30 nm (Figure 5b), most of the SWNTs become vertically aligned on the surface. Vertically aligned nanotubes could be observed in everywhere on the film after evaporation of the titanium molecules with ~30 nm thick. Our microscopic analysis results show that more than 80% of the SWNTs are vertical aligned on the surface. However, this vertical alignment is kept only for thicknesses up to about 100 nm (Figure 5c). Titanium layers thicker than that did not produce a sustained vertical alignment, which is possibly a result of the collapse of the vertically aligned SWNTs under the weight of the titanium molecules (Figure 5d). Furthermore, slow evaporation rates of the titanium molecules are likely to be better for aligning the SWNTs. Thus, we tested the degree of alignment of the nanotubes at different titanium evaporation rates (namely, 0.05, 0.15, and 0.3 nm/sec) and found that the maximum current density of field emission decreased with increasing evaporation rate. This result indicates a better vertical alignment at lower evaporation rates (i.e., 0.05 nm/sec), which might be attributed to the better uniformity of the titanium layers produced at low rates relative to those obtained at high rates.[30] Thus, the evaporation rate (~0.05 nm/sec) and film thickness (~30 nm) of the titanium layer were thoroughly controlled to achieve an optimum alignment of the SWNTs over a large area.

The nanotubes were obtained in a single-step process involving vertical alignment by a magnetic field and simultaneous preservation of the arrangement by the evaporation of a titanium layer on the ITO surface. No vertical alignment of the SWNTs was observed when a two-step process was applied. In the first alignment step, the Fe–oxide/SWNT particles moved from the suspension to the device surface—in the presence of a magnetic field of about 400 gauss—and aligned perpendicularly to the surface. In the second step, they were transferred to an e-beam evaporator in the absence of a magnetic field. This was done after evaporating the SWNT solution contained within the substrate in the presence of a magnetic field. The resulting SWNTs did not retain their vertical alignment when the field was removed (data not shown). In other words, it is impossible to achieve a sustained vertical alignment of the SWNTs without simultaneously depositing titanium molecules on the device surface. This is likely due to the high length-to-diameter aspect ratio and the bending rigidity of the nanotubes. The supporting frames of the titanium layer truly keep the tube axis in a fully perpendicular orientation with respect to the surface.

This single-step approach can also be used to vertically align CNTs without magnetic particles if a strong magnetic field

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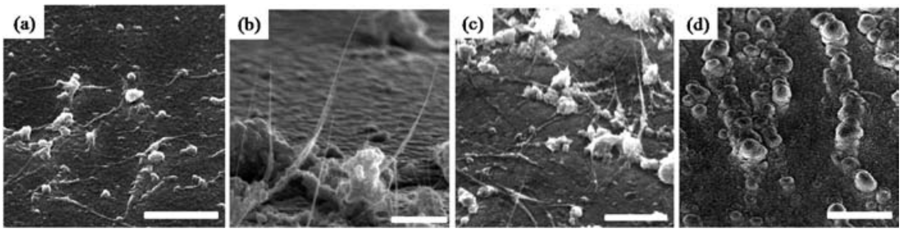

Figure 5. SEM images of aligned Fe−oxide/SWNTs for different titanium thicknesses: (a) 10, (b) 30, (c) 70, and (d) 800 nm [scale bars: (a) 2 μm, (b) 500 nm, (c) 1 μm, and (d) 5 μm].

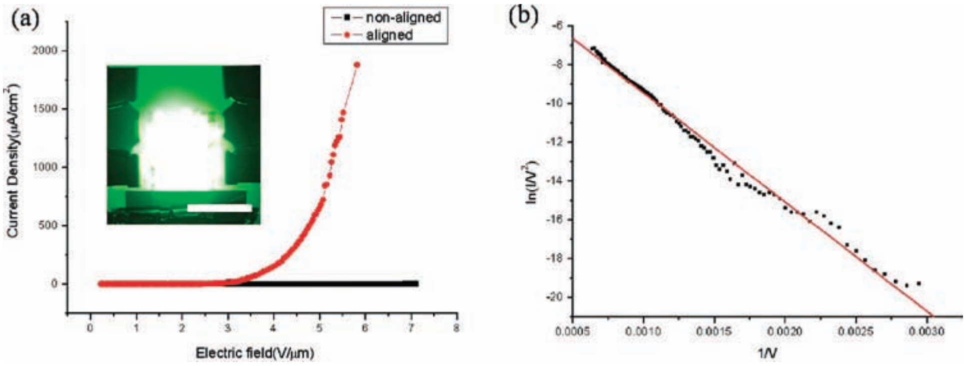

Figure 6. (a) Plot of the applied electric field versus the current density. (b) Fowler−Nordheim plot. The vertically aligned Fe−oxide/SWNTs obtained by means of the MEA process exhibit good field-emission properties (red line). Because the Fe−oxide/SWNTs are not vertically aligned in the absence of a magnetic field, the FEA has no field emission I−V characteristics (black line). In (a), the maximum current density (J) is 1880 μA/cm^2 and the turn-on voltage is 2.45 V/μm. Fe−oxide/SWNT emitters are very uniformly illuminated. The inset shows a real image taken on a 1-cm^2 phosphor anode during the application of a voltage in a vacuum chamber.

(between 7 and 26 T) is applied to the nanotubes.[31,32] However, in this case, an ~1000 times higher field strength is required to align the SWNTs compared to the case of the nanotube samples containing small amounts of magnetic particles. Also, a similar approach using an electric field instead of a magnetic one can be employed for the vertical alignment, provided that an appropriate apparatus for applying electric fields is designed in the high-vacuum-evaporator chamber.

The significance of our approach was verified by assessing the field-emission characteristics of the vertically aligned SWNTs (see inset of Figure 6a). A prototype field-emission display device based on the magneto-evaporation alignment method shows a highly uniform illumination over a large area (namely, 1 cm^2). An ITO layer containing the SWNTs was used as the cathode and a phosphor-coated ITO glass served as the anode. The anode was made by screen printing method using conventional cathodoluminescent green phosphor (ZnS:Cu,Al) which was calcined at 430 °C for 30 min. The field emission I−V curve was obtained after sweeping the voltage several times for aging of the field-emission analyzer (FEA) (Figure 6a). The turn-on voltage of the vertically aligned SWNTs on the ITO glass substrate was measured to be about 2.45 V/μm, and the emission current density of this sample was found to be 1880 μA/cm^2 at 5.8 V/μm. The field-enhancement factor (β) was found to be 2551 for an assumed work function of the SWNTs of 4.8 eV.[33] The value of β was determined using the Fowler−Nordheim (FN) relationship [Figure 6b; the slope (k) of the FN plot is given by: $k = -B\phi^{3/2}d/\beta$, where the constant $B = 6.83 \times 10^9$ V eV$^{-3/2}$ m^{-1},[34] ϕ is the work function, and d is the distance between the anode and the cathode]. The I−V characteristics of the vertically aligned SWNTs indicate that this system has excellent field-emission properties. Higher field-emission current densities could be achieved by controlling the density of SWNTs within the emitter.

In summary, we have developed a method for the fabrication of vertically aligned SWNTs on ITO glass substrates that is based on a single-step process involving the simultaneous vertical alignment of the nanotubes by a magnetic field and the fixation of the alignment by means of the direct evaporation of the films. We found that the fraction of vertically aligned nanotubes after evaporation is strongly affected by the titanium film thickness, the evaporation rate and strength of the magnetic fields. The threshold field for aligning the samples was approximately 200 gauss. A large fraction of vertical alignment of the SWNTs was produced after titanium evaporation with ~30 nm-thick. The fraction of vertically aligned tubes was significantly decreased on titanium layers with a thickness below ~10 nm. Also, titanium layers thicker than 100 nm did not produce a sustained vertical alignment. This procedure leads to the formation of vertically aligned SWNTs with good uniformity over a large area. Unlike the aligning techniques reported to date, this method affords the alignment of high-aspect-ratio isolated SWNTs at room temperature without the need for organic binders or further aligning procedures. Thus, our method offers several advantages over previously reported techniques, for example, long-time stability, mild temperature conditions, and good reproducibility. Moreover, it is cost-effective and amenable to mass production, and it could potentially lead to

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the use of CNTs as functional devices. Further studies are in progress to develop a similar single-step process for the simultaneous vertical alignment of SWNTs by an electric field and fixation of the alignment by means of the direct evaporation of the films.

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Supporting Information Available: (1) TEM image of supernatant solution, showing only Fe−oxide nanoparticles, (2) SEM image of SWNTs after the titanium evaporation in the absence of a magnetic field. This material is available free of charge via the Internet at http://pubs.acs.org.

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