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Cap Closing of Thin Carbon Nanotubes**

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Carbon nanotubes (CNTs) are the subject of intense research due to their special properties, such as their nanometer dimensions, high aspect ratio, high Young's modulus, and high thermal and electrical conductivities.[1] One of the main hurdles for the successful application of CNTs in electronics today is the inability to precisely engineer their structure at the nanometer/atomic scale. For example, processes are under development which filter post-deposited nanotubes based on their chirality[2] in order to separate semiconducting from metallic nanotubes.[3,4] For applications such as electron emitters[5,6] and probe tips,[7,8] control is required over the structure of the nanotube tip, also referred to as its "cap". CNTs can either be open or closed directly after their growth; some processes allow the caps to be opened,[9] sharpened,[10] and even functionalized with specific chemical groups.[11] It has been proposed that open nanotubes can also be forced to close, and indications of such a process have been published.[9,12] Here, we provide experimental evidence that it is possible to close open nanotubes and we present a detailed investigation of the cap-closing mechanism of individual, thin, multiwalled CNTs (3–8 walls). The closed-cap nanotubes exhibit high current stability, which is of advantage for their use as electron sources.

A single nanotube protruding from a sharp edge was selected from an ensemble of nanotubes, prepared either by chemical vapor deposition (CVD) or arc discharge, and attached to a tungsten support tip. This nanotube was then broken by running a current through it—the break-off occurred at a weak spot along the length of the nanotube, possibly a defect in its structure (see Fig. 1). Individual, short, freestanding nanotubes on the tungsten tips were obtained using this procedure.[13] The caps of six CNTs (nos. 1–6) were imaged using transmission electron microscopy (TEM). After breaking the nanotubes, we found that four of the six displayed closed caps at their broken ends (see Fig. 2, Table 1). Note that obtaining high-resolution TEM images of freestanding individual nanotubes is very difficult, as many nanotubes are lost in the process or vibrate[8] too much to obtain atomic resolution. The nanotubes with closed caps were the ones with the smallest outer diameters and up to five walls, whilst the nanotubes with open caps had at least eight walls; dependence on the inner-wall diameter was not observed. Since all of the nanotubes

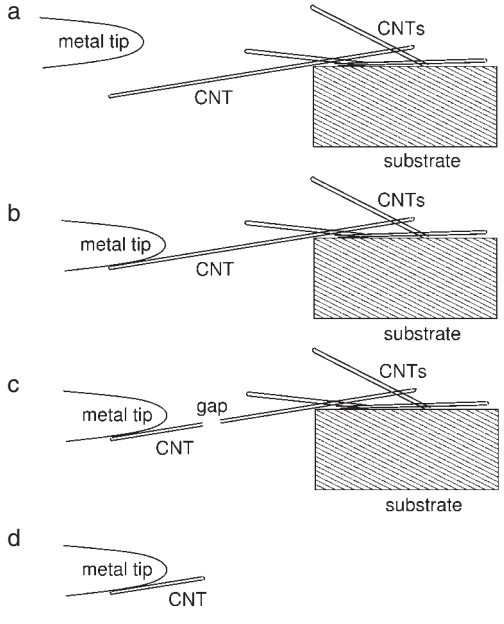

Figure 1. Mounting procedure of a CNT electron source as performed inside a scanning electron microscope. a) A CNT protruding from a thin substrate containing many nanotubes is selected and b) attached to a tungsten support tip. c) The CNT is broken by Joule heating. d) The open tube end is finally closed.

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[**] We thank T. H. Oosterkamp, D. B. Peckys, and D. Tomanek for fruitful discussions, as well as G. Amaratunga, M. Meuwese, T. van Rooij, A. Teh, M. van Wely-Dieleman, and M. Yangs for experimental help, and A. G. Rinzler for kindly providing the arc-discharge nanotube sample. This work was supported by FEI Company, the Dutch Ministry of Economic Affairs, the EPSRC, and the EC.

 DOI: 10.1002/adma.200400266

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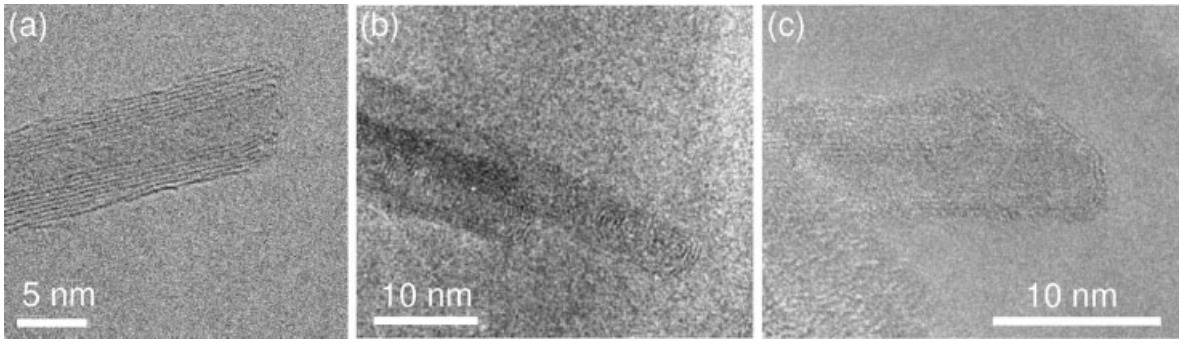

Figure 2. High-resolution TEM images of freestanding CNTs mounted individually on tungsten support tips. a) Image of nanotube no. 1 with an open cap and eight walls. b),c) Images of nanotubes no. 2 [6] and no. 5, respectively, with closed caps.

Table 1. Results of the investigations of the nature of the caps of nanotubes nos. 1–7* in a transmission electron microscope. The electron emission of nanotube no. 7* was investigated in situ in the microscope. Nanotubes grown by both arc-discharge and CVD methods were investigated. For nanotubes with up to five walls, closed caps were observed, while those with eight or more walls resulted in open caps. The inner diameter, d_{in}, and outer diameter, d_{out}, are also indicated.

Nanotube No.	Type	d_{out}	d_{in}	No. of walls	Cap	Figure	Ref.
1	arc	7.5±0.1	2.6±0.1	8	open	1a	
2	arc	5.4±0.1	2.6±0.1	4	closed	1b	[6]
3	arc	15.7±0.2	4.3±0.2	8	open		[13]
4	arc	7.0±0.3	3.3±0.3	3	closed		
5	cvd	4.6±0.1	2.1±0.1	3	closed	1c	
6	cvd	4.4±0.2	2.6±0.2	3	closed		
7*	arc	5.2±0.2	2.0±0.2	5	closed	2	

were broken off in the mounting procedure, it was initially expected that all the nanotubes would have open caps. Thus, the observation of closed caps shows that a cap-closing mechanism exists for small-diameter nanotubes.

We now demonstrate the in-situ opening and closing of a nanotube (no. 7) in a transmission electron microscope. A high-resolution image of the nanotube taken at a position close to the tungsten support tip revealed that it had five walls (see Fig. 3a). Initially, the nanotube was 0.69 μm long. A voltage difference of 270 V was then applied between the nanotube and the electrode for a short time, leading to a strongly fluctuating emission of 5–7 μA. This caused the nanotube to break in the middle, probably at a weak spot. The remaining nanotube (with a length of 0.10 μm) had an open cap, as clearly seen in Figure 3b. In this figure, the nanotube was emitting at a current of 0.05 μA at a voltage of 190 V. It was not possible to use the high-resolution mode of the transmission electron microscope while the nanotube was emitting, due to the vibrations of the tube. Running a current of 0.5 μA at 220 V for three minutes did not lead to any changes in the open cap. At a voltage of 240 V, the nanotube emitted a current of 1 μA with strong fluctuations of 0.5 μA (ca. 50 %). Over a period of approximately five minutes, the current slowly increased to 3 μA, although the same voltage was maintained at the electrode. Two possible explanations exist: the structure may have been slowly changed (it may have become slightly sharper at the end, for example), or the temperature may have increased slowly, for example, by small changes in the contact resistance. Then the nanotube closed suddenly, as seen in Figure 3c. The length of the nanotube did not change, within the experimental error of 0.5 nm, during this experiment. The current dropped to 1.5 μA after the closing, as expected for a reduction of the sharpness of the emitter on account of the closing. An image recorded at a lower emission current is shown in Figure 3d. This closed-cap nanotube was then tested for emission of currents up to 10 μA without breaking or failure.

Field emission microscopy (FEM) was used to investigate the tube caps of twelve other CVD-grown nanotubes (nos. 8–19). FEM images provide information on the electronic structure and the local tunneling probability of an emitting

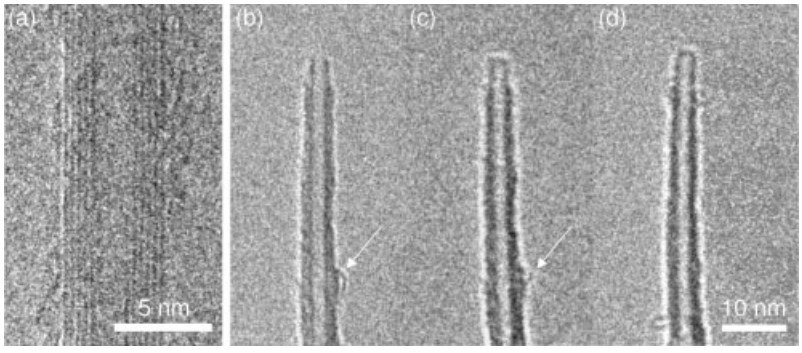

Figure 3. Results of the in-situ electron emission experiment in the transmission electron microscope for nanotube no. 7. a) High-resolution image showing five walls. b) Image of the nanotube end taken in the presence of a strong electric field, resulting in electron emission of a current of 0.2 μA. c) Image recorded directly after the nanotube had closed. The nanotube emitted a current of 1.5 μA. The arrows in (b) and (c) indicate a piece of amorphous carbon that showed the same distance from the nanotube end in both images. d) Image of the closed cap recorded while the nanotube was emitting a current of 0.08 μA.

area.[14,15] Figures 4a–d,f show that the FEM patterns of nanotubes 8–12 are almost round and have several local maxima within the emission pattern, but they do not contain sharp transitions from regions of low to high intensity. These patterns are interpreted as the typical emission patterns of single-walled or thin multiwalled nanotubes with closed caps.[15] These patterns were highly stable with time for emitted currents up to 1 μA, as expected for closed-cap nanotubes.[9] The emission patterns of five other nanotubes (nos. 13–17) showed the patterns of closed caps as well (not shown). It is important to point out that a different type of emission pattern, showing rings with five- and sixfold symmetry and interference fringes, has been observed previously for bundles containing thick multiwalled CNTs with highly symmetric caps.[16] Such patterns are not likely to occur for our nanotubes, as these caps do not have a completely regular and symmetric structure due to the breaking process, as can be seen in Figures 2b,c. For comparison, three patterns not related to stable, closed caps are shown in Figures 4e,g,h. The pattern in Figure 4e is the initial emission pattern of nanotube no. 12 and shows a single round spot that changed its position every few minutes. This pattern changed to the pattern of Figure 4f only after repeated heating to 700 °C and extracting an emission of 10 μA. The unstable pattern of Figure 4e can be interpreted as belonging either to a strongly adsorbed species, amorphous carbon, or an open nanotube. The FEM images of Figures 4g,h are the patterns of nanotubes nos. 18,19, respectively, and display separated and uncorrelated spots. These spots changed their positions and intensities every few seconds; the pattern of Figure 4h even rotated. Since additional heating and emission of several microamps of current did not change this behavior, we interpret these patterns as being caused by open nanotubes, whose wide emission patterns and fluctuating emissions were undesirable for field-emission applications. To summarize, ten out of twelve nanotubes showed the emission patterns associated with having closed caps. In the whole process of mounting the nanotubes, transferring them to the FEM, and imaging them, only a few additional nanotubes were lost. We consider this success rate highly satisfactory.

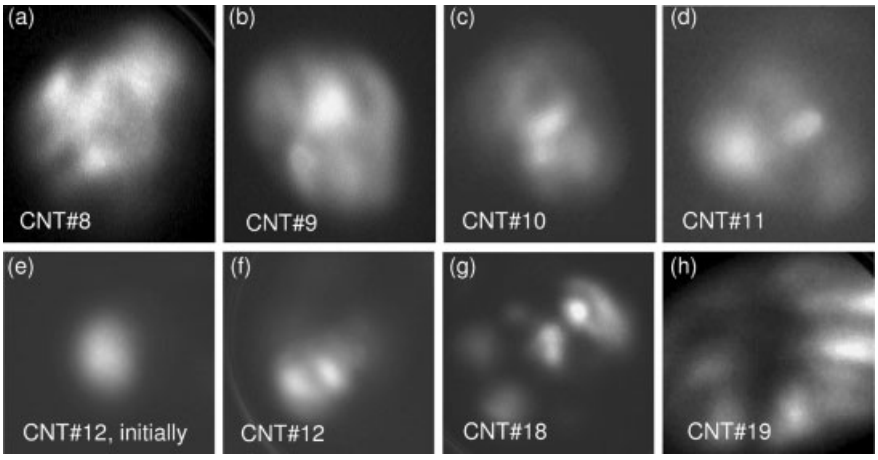

Figure 4. FEM images of CNT electron sources. The emission patterns have an approximate width of 1 cm. The images a)–f) correspond to nanotubes nos. 8–12, showing patterns of closed caps. Image e) shows the initial emission pattern of nanotube no. 12, while image f) presents the emission pattern after additional treatment (see text). Images g) and h) of nanotubes nos. 18,19, respectively, are the emission patterns of CNTs with open caps.

The electron emission from a closed-cap nanotube with no dangling bonds is expected to exhibit a high current stability in contrast to emission from open-cap nanotubes.[9] Figure 5 shows plots of current versus time, which exhibit high stability, for three nanotubes (nos. 8,11,12). The maximum deviation of the current from the mean current, $\Delta I/I$, is smaller than 1 %, measured over a frequency range from 1 mHz to 2 Hz. All the other nanotubes that had emission patterns associated with having closed caps showed similar stability (data not shown). The initial measurement of nanotube no. 12 is related to the emission pattern in Figure 4e; it shows much larger fluctuations, with a maximum $\Delta I/I$ value of 9 %. Such instability is common for cold field emitters made from materials such as tungsten, which have much lower activation energies for surface migration than CNTs.[17] Note that the difference in current is not the cause of the larger fluctuations–closed nanotubes still showed highly stable emission for currents up to 500 nA (data not shown). The emission of nanotubes nos. 13,14, which had FEM patterns associated with open caps, showed large fluctuations, of over 100 %.

Having demonstrated the existence of a cap-closing mechanism, two important questions remain. The first question is whether the cap always closes by forming bent graphene layers, as expected for a "pure" cap structure of a nanotube, or whether the cap is formed by amorphous carbon. The TEM images of Figures 2b,c show graphene cap structures. In addition, the emission patterns of closed caps were observed and the emitted current was found to be highly stable for a cold

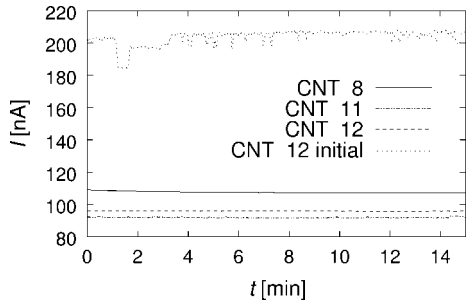

Figure 5. Plot of the emitted current versus time for CNTs nos. 8,11,12. The initial trace of nanotube no. 12 is also shown (see text). The data presents current measurements every 0.5 s.

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field-emission process. A cap formed by amorphous carbon, with its undefined structure and manifold dangling bonds, would probably result in less stability under the harsh conditions of field emission (i.e., heat, free electrons, ion bombardment, and very large electric field). Thus, it is likely that most caps consist of graphene layers and not of amorphous carbon.

The second question relates to the physical process that causes the nanotube cap to close. In this discussion, it is important to define two temperatures related to the structure of CNTs. Heating a CNT to the carbonization temperature (ca. 700 °C) leads to the removal of volatile species, such as oxygen, nitrogen, and hydrogen atoms bound to carbon atoms.[1] New C–C bonds are formed at somewhat higher temperatures, resulting in the formation of basic structural units of graphene. At the graphitization temperature (ca. 2300 °C), interplanar site correlation is established and nanotubes with stiff and straight carbon layers are obtained. We expect that the formation of a cap structure requires a temperature in between the carbonization and the graphitization temperatures. Indeed, others have observed that heating to the carbonization temperature stabilizes the cap structures of CNTs.[18] In the mounting procedure inside the SEM, nanotubes were broken at a weak spot by running a current through them. At the instant of breaking, the local temperature at the breaking point is likely to have reached the carbonization temperature or higher. In the in-situ experiment of cap closure under electron emission conditions at an emitted current of 3 μA, the temperature probably reached values of over 1000 °C.[19] Indeed, from measurements of the energy spectra of individual CNTs, it was deduced that the temperature had already increased to about 800 °C at an emission current of 500 nA (data not shown). A strong electric field at the weak spot may also have influenced the closing. Such a field is present for a short period of time, as a potential drop of several volts occurred over the weak spot at the instant of breaking. A strong electric field was applied at the nanotube end in the in-situ experiment. The presence of free electrons and of hydrocarbons may also play a role in the growth of a small layer of amorphous carbon prior to the closure. This layer could serve as a source of the carbon atoms that are transferred into the graphene structure of the cap at the instant of closure. Alternatively, it is also possible that carbon atoms already present in the nanotube reorganize at the elevated temperature and snap into a closed cap. Others have observed the reorganization of straight graphene layers into bent layers for heated CNTs,[20] as well as for carbon nanofibers under tensile strain,[21] in both cases under electron-beam irradiation. To further investigate these possible mechanisms, closing experiments should be carried out at different vacuum levels and it should be determined whether the closing can also take place by applying thermal energy only. Finally, we did not observe closed nanotubes with more than five walls. Thus, the closing is more likely to occur for thin nanotubes, presumably because the energy barrier to go from an open to a closed cap increases with the diameter of the nanotube and the number of walls. The existence of a closing mechanism is favorable from an energetic point of view. A closed cap has a lower total energy than an open cap due to the reduction of the number of open bonds[22,23] (20 eV for a single-walled CNT with a diameter of 0.8 nm).[22]

In conclusion, we have demonstrated the existence of a cap-closing mechanism for thin nanotubes of up to five walls, and have observed the closure of an open nanotube in situ in a transmission electron microscope. It proved feasible to apply the closing mechanism to a highly precise mounting process, thus routinely providing individually mounted CNTs with closed caps, short lengths, and small diameters, which served as electron point sources with a high current stability (1 %). This is technologically important for the production of regular tips for next-generation electron sources for electron microscopes, in order to enhance their resolution.

Experimental

Sample Preparation: Two types of CNTs were investigated. Sample 1 contained nanotubes grown by arc discharge in the group of Smalley [24], from which thin nanotubes were selected in the experiments. Sample 2 contained thin (mainly 1–4 walled) CNTs grown by chemical vapor deposition (CVD) on an oxidized silicon substrate [25]. The nanotubes from both samples had a low concentration of defects and amorphous carbon.

Mounting of Nanotubes: Individual CNTs were mounted on tungsten tips in a scanning electron microscope (SEM), equipped with a nanomanipulator [13]. The vacuum level in the specimen chamber of the SEM was 5×10^{-6} torr (1 torr ≈ 133 Pa), while the current density of the electron beam amounted to 10 nA μm^{-2}. In the mounting procedure, a nanotube was first selected from a manifold of nanotubes protruding from a sharp edge. This nanotube was attached to a tungsten tip, which had been pressed into carbon tape to apply some glue (hydrocarbons) for the attachment of the nanotube. Next, a current of 5–50 μA, which differed for each nanotube, was applied through the nanotube, which caused the nanotube to break at a weak spot. This technique enabled the mounting of individual nanotubes with pre-determined radii and lengths [13]. Each mounted nanotube was finally tested inside the SEM mounting chamber for its electron-emission ability of a current of approximately 100 nA.

TEM Experiments: TEM images of freestanding individual nanotubes mounted on tungsten support tips were taken at an electron beam energy of 300 kV in low-dose mode. To avoid electron-beam-induced damage of the nanotube, the objective astigmatism and focus were adjusted on a position near to, and not on, the nanotube. The total time that a nanotube was illuminated in order to record a high-resolution image was limited to approximately one minute. The current density of the beam was 10–20 nA μm^{-2} and the vacuum level in the specimen chamber was 1×10^{-7} torr. For the in-situ observation of a nanotube while it was emitting electrons, a special TEM specimen holder was constructed with a metal electrode for electron-emission measurements at a distance of 2.0 mm from the nanotube.

FEM Investigations: FEM images of the nanotube caps were recorded with a microchannel plate and a phosphor screen, while the nanotubes were emitting a current of 0.1 μA as a result of a voltage difference between the microchannel plate and the nanotube of 300–500 V; this value was different for each nanotube. The FEM setup was placed inside an ultrahigh vacuum system (10^{-10} torr). A fresh nanotube was always heated first to 700 ± 50 °C in vacuum for 10 min to remove adsorbed species from the tube [17]. The samples were heated to a temperature of about 500 ± 50 °C during the emission experiments to continuously clean the tubes.

Received: February 25, 2004
Final version: October 29, 2004

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Non-Volatile Polymer Memory Device Based on a Novel Copolymer of *N*-Vinylcarbazole and Eu-Complexed Vinylbenzoate

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The ever-increasing pace in the development of information technology requires concurrent development in advanced materials technology to overcome the scaling problem and physical limitations on device components that the semiconductor industry may have to face in the next decade.[1] Organic materials (including dyes, complexes, oligomers, and polymers) are promising candidates for future molecular-scale device applications.[2] Their attractive features include miniaturized dimensions and the possibility for molecular design through chemical synthesis.[3,4] In particular, polymer materials have attracted considerable attention because of their good scalability, mechanical strength, flexibility, and most important of all, ease of processing. As an alternative to the more elaborate processes of vacuum evaporation and deposition of inorganic and organic molecular materials, manufacturers could eventually use ink-jet printers or spin-coaters, for example, to deposit polymers on a variety of substrates (plastics, wafers, glass, or metal foils).[5] Several types of electronic devices can be derived from polymer materials, including light-emitting diodes,[6] transistors,[7] lasers,[8] photovoltaic cells,[9] and switches.[10]

Recently, flash-type and write-once read-many-times (WORM)-type memories based on polymeric materials have been demonstrated, receiving a great deal of attention due to their simplicity in structure, good scalability, low-cost potential, and large capacity for data storage.[5,11] A polymer memory stores information in a manner entirely different from that of silicon devices. Rather than encoding "0" and "1" as the amount of charge stored in a cell, polymer memory stores data, for instance, based on the high and low conductivity response to an applied voltage (electrical bistability).[5] A number of polymeric materials, including poly(allylamine hydrochloride),[12] a mixture of poly(ethylenedioxythiophene)

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 DOI: 10.1002/adma.200401048