Nanotechnology **17** (2006) 1087–1098 doi:10.1088/0957-4484/17/4/041

Establishing Ohmic contacts for *in situ* current–voltage characteristic measurements on a carbon nanotube inside the scanning electron microscope

Qing Chen, Sheng Wang and Lian-Mao Peng

Key Laboratory for the Physics and Chemistry of Nanodevices, Department of Electronics, Peking University, Beijing 100871, People's Republic of China

E-mail: qingchen@pku.edu.cn

Received 11 August 2005, in final form 29 September 2005
Published 30 January 2006
Online at stacks.iop.org/Nano/17/1087

Abstract
Multi-walled carbon nanotubes (CNTs), either on an SiO_2 substrate or suspended above the substrate, were contacted to W, Au and Pt tips using a nanoprobe system, and current–voltage (I–V) characteristics were measured inside a scanning electron microscope. Linear I–V curves were obtained when Ohmic contacts were established to metallic CNTs. Methods for establishing Ohmic contacts on a CNT have been developed using the Joule heating effect when the tips are clean and e-beam exposing the contacting area of the tip when the tips are covered by a very thin contamination layer. When the contact is not good, non-linear I–V curves are obtained even though the CNTs that have been contacted are metallic. The resistance measured from the metal tip–CNT–metal tip system ranges from 14 to 200 kΩ. When the CNT was contacted via with Ohmic contacts the total resistance of the CNT was found to change roughly linearly with the length of the CNTs between the two tips. Field effect measurements were also carried out using a third probe as the gate, and field effects were found on certain CNTs with non-linear I–V characteristics.

1. Introduction

Carbon nanotubes (CNTs) and other quasi-one-dimensional nanotubes and nanowires are potential building blocks for nanodevices. To design and optimize the properties of the nanodevices made of nanotubes and nanowires it is essential to know the electronic properties of their building blocks, i.e. nanotubes and nanowires. Although there exist several methods that can be used for studying the properties of bulk materials, electronic characterization of a single nanotube or nanowire is not easy. Knowing the properties of an individual CNT is especially important for its application in nanodevices because, until now, there is no suitable way to control the chirality (which decides the electronic properties) of the carbon nanotube. The first problem for measuring a CNT electronically is to connect it to metal electrodes. Growing CNTs directly on pre-patterned electrodes is a useful method [1], but such an approach is limited to some specific electrode materials, e.g. Fe, Ni and Co. Using a scanning probe microscopy (SPM) probe together with liquid metal Hg, contacts had been made without using lithography [2]. But so far, such methods only provide two-terminal current–voltage (I–V) characteristic measurement. Direct measurements inside the transmission electron microscope (TEM) have been performed [3, 4]; however, due to the limited space inside the TEM, it is difficult to make three- or more-terminal measurements or make other modifications. In most of the cases, the lift-off method including electron beam lithography and metal deposition is employed to set up the nanoelectrodes onto the nanotubes. This procedure is not only tedious but also not versatile. The as-grown carbon nanotube sample usually contains both metallic and semiconducting nanotubes. Until the contacts have been made and measurements have been performed, the properties of the carbon nanotube and

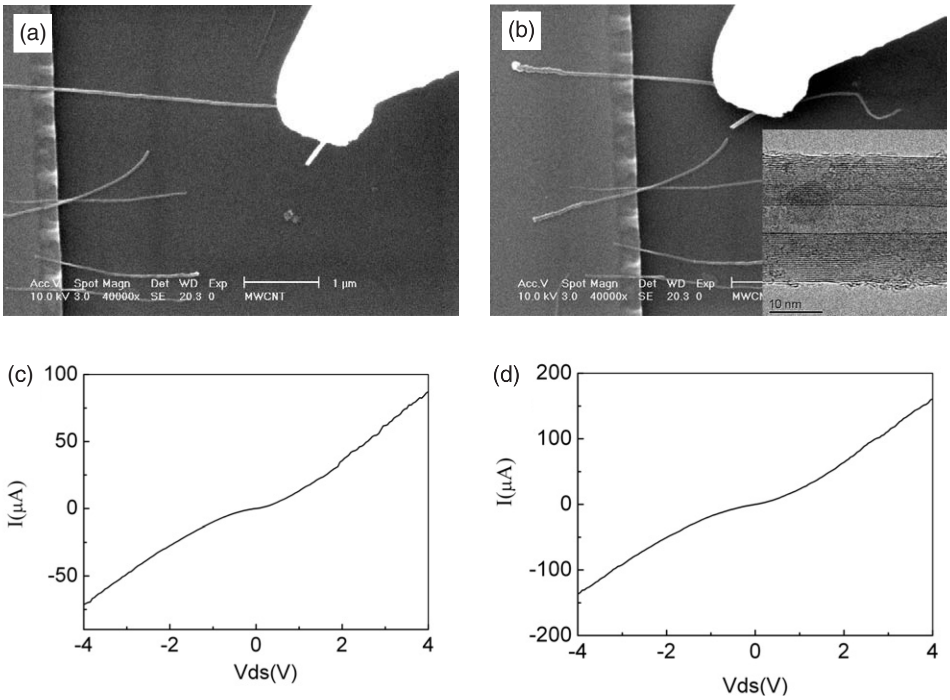

Figure 1. (a) and (b) are SEM images showing a W tip (right) was contacted to the same CNT that was connected to an electrode (left). The inset in (b) is a HRTEM image showing the structure of the CNT. (c) and (d) are corresponding I–V curves measured for the configurations shown in (a) and (b) respectively.

the properties of the device are unknown. When using the lithography method to make the contacts, once the contacts are made, the device is fixed and its properties are hardly changeable. Without knowing the properties of single devices in advance, complex circuits are hard to design and fabricate. To further understand the working mechanism of the nanodevices, it is also highly desirable to know the effect of such parameters as the length of the tubes, the thickness of the insulator layer, etc on the performance of the devices, but for a fixed device such kinds of studies are not easy. Using a conductive AFM as a movable electrode is one way to study device properties and some valuable results have been obtained [5]. However, such a method is not very easy to use for studying suspended CNTs, where the effect of the substrate can be avoided [6–8]. Only a couple of groups have the state of the art technique to perform electronic measurements using AFM or STM on suspended CNTs [9–12].

In our previous work we demonstrated that, using a probe system inside a scanning electron microscope (SEM), contacts can be made directly between the nanostructures (like nanotubes and nanowires) and the tips, and electronic measurements can be performed [13]. The main question is whether or not the measured results reflect the intrinsic properties of the nanostructure. The vital problem is the contact problem, which has been one of the most important problems for electronic measurements on carbon nanotubes. Using the lithography method, the contact properties have been studied extensively [5, 14–16]. It is now well known that different metals can result in different contact properties, such as Ohmic contact or with a Schottky barrier. The contacts have also been found to be diameter dependent [15, 16]. For the measurements on free standing CNTs using an SEM manipulator, it has been found that non-linear semiconductive I–V character, not conducting with more than a 10 V bias, as well as high conductivity Ohmic behaviour, can be obtained [17]. But whether such a behaviour is fundamental to the CNT or caused by the tube–tip contact is not clear. Direct probing has been used for electronic measurements on surfaces. Using a four-point probe system inside an ultra-high vacuum SEM, the electronic transport through surface states have been studied [18, 19]. In these cases the surface is fixed and has large area, that can be used to make reliable contact. Ohmic contact can be easily made by pressing the probes onto the surface. But for nanotubes and nanowires, due to the nanometre sized contact area, making good contacts is far more difficult.

In the present work, through a systematic study, we provide a few procedures for establishing reliable contacts to the CNTs either on an insulating substrate or suspended using a probe system inside an SEM. We have successfully performed two- and three-terminal I–V characteristic measurements, and our results provide a quick way to characterize the electronic properties of nanotubes and nanowires and to study the effects of various parameters on the device performance.

2. Experimental details

A four-MM3A-nanoprobe system manufactured by Kleindiek company was used for the measurement. The system was installed inside an FEI XL 30F SEM. A detailed description of the system has been given previously [20]. Multiwalled carbon nanotubes (MWCNTs) with diameters ranging from 20 to 50 nm were synthesized using the CVD method. The inset in figure 1(b) is a high resolution transmission electron microscopy (HRTEM) image showing the structure of a typical

MWCNT. Sharp tungsten tips were made by etching tungsten wires in NaOH solution. Gold tips were made by evaporating 20 nm Au onto freshly made W tips. Sharp Pt tips were made by electrochemical etching using $CaCl_2 + HCl +$ acetone solution, and normal Pt probes with larger radius of curvature were made by cutting Pt wires with 0.3–0.5 mm diameter using scissors.

MWCNTs were dispersed either randomly on a SiO_2 substrate by dropping a drop of CNT solution onto the substrate, or with certain order (aligned) on the edge of the electrodes using the electric field assisted assembly technique. The electrodes are Al (with a small amount of Si) electrodes pre-prepared on the SiO_2 substrate using micro-lithography. Suspended CNTs were picked up by Pt probes and moved into the SEM. The method for picking up CNTs using Pt probes is as follows: putting the Pt probe into CNT samples and moving the probe back and forth a couple of times and then drawing the probe out of the CNT sample. CNTs were attracted to the Pt probes by van der Waals forces when they touched the probes and the adhesion between the Pt probes and the CNTs was enhanced by moving the probe back and forth within the CNT samples. At the edge of the Pt probes, some CNTs were found protruding from the tip. Those CNTs were used as a suspended CNT source. Au tips and W tips were also used to pick up the CNTs, but due to the weaker attracting force between these tips and the CNTs the number of CNTs that have been picked up using Au and W is much less than that using the Pt tips. The CNTs were observed and the movement of the nanoprobes was monitored using SEM. The electronic properties were measured using a Keithley 4200 semiconductor characterization system.

3. Results and discussion

CNTs either on the substrate or suspended were measured using the probe system. Ohmic contacts to the CNTs were established and repeatable results were obtained.

3.1. Measuring CNT sitting on the substrate

Our experience shows that to establish good contact is a relatively easy task for CNTs sitting on the substrate, since the position of the CNTs is fixed and external force can be applied to improve the contact by pressing the CNT using the tip. The main challenge here is to make the contact properly and at the same time not to break the CNT. Figures 1(a) and (b) are two SEM images showing the same CNT being connected by the same tips at different positions. The left side of the CNT was contacted to a pre-prepared electrode by the electric field assisted assembly technique. A freshly made W tip (not shown in the image) was contacted to the left electrode with some force added. The right side of the CNT was sitting on a SiO_2 substrate and connected by another freshly made W tip. The contact was made by moving the tip to a position just touching the CNT and by pressing the tip gently onto the CNT. To avoid breaking the CNT, a bias was added between the tips touching the left electrode and the right W tip and the current between the two tips was monitored during the movement of the right W tip. Before the right W tip touched the CNT, there was no current passing through the system. Once the tip touched the CNT, there was a current passing through the system. When the current exceeded a threshold value (10 nA in our experiment) the movement of the tip was stopped by a feedback circuit. The feedback circuit was then switched off and a small force was added to the CNT by moving the tip one fine step (0.5–1 nm) further. Once a reliable contact was established I–V curves were then measured, and the two curves shown in figures 1(c) and (d) correspond respectively to figures 1(a) and (b). The two I–V curves are seen to be non-linear at low bias, but become linear at higher bias, and this behaviour may be explained by the existence of a potential barrier between the electrodes and the CNT [3]. The resistance of the CNT can be estimated from the linear regime at high bias [3]. When a CNT was about 3 μm (figure 1(a)), the resistance was estimated to be about 60 kΩ (figure 1(c)). When the CNT was reduced to about 1 μm (figure 1(b)) the resistance was reduced to about 14 kΩ (figure 1(d)). The fact that the resistance is higher for a longer CNT (figure 1(c)) than a shorter CNT (figure 1(d)) indicates that the CNT we measured is not a perfect ballistic conductor and scattering due to phonons and imperfections of the CNT played a noticeable role in this case. We noticed that the non-linear regions are almost the same in figures 1(c) and (d). Since the left contact is the same for both figures 1(a) and (b) and our experience shows that this type of contact (made by using the electric field assisted assemble technique) is in general of rather good quality, it is reasonable to conclude that the right contact between the CNT and the right W tip is of the same quality for both figures 1(a) and (b), i.e. repeatable contacts had been realized on different positions on the same CNT.

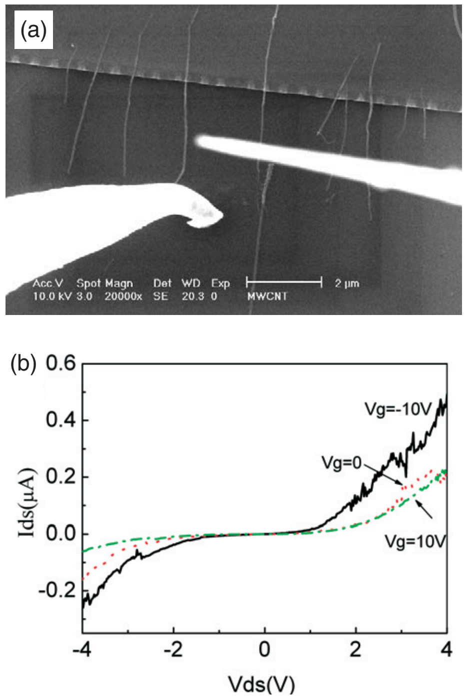

Figure 2. (a) SEM image showing a third tip near the CNT; the tip was separated from the CNT and used as a gate in field-effect measurements. (b) I–V curve measured with different gate voltage added to the third tip.

(This figure is in colour only in the electronic version)

Figure 2(a) shows a field-effect or three-terminal measurement configuration. A CNT was contacted directly by the top electrode and the lower (left) W tip, and two-terminal I–V curves were measured as before. In a field-effect transistor (FET) the current in the conducting channel (usually made of a semiconductor) may be modulated or controlled by a gate which is separated from the conducting channel by an insulating layer. In our experiment the conducting channel is the CNT, and the gate is the W tip to the right of figure 2(a). This gate is electronically separated from the CNT, yet at the same time it is close enough to the CNT that an applied gate voltage on this tip may affect the electron conductance in the CNT. The advantage for using the movable tip as the gate is that the effect of gate position may be studied. Figure 2(b) shows that the gate does have observable effects on the I–V characteristics of the CNT. But so far, we have not obtained any repeatable result on field effect yet. The main difficulty involved in this case is that the height of the tip cannot be determined accurately by looking at the SEM image and the distance between the tip and the CNT is hard to control. Although moving the tip to just touching the CNT or the substrate near the CNT can give a reference height, this procedure may easily break the CNT under investigation.

Besides using the electric field assisted assembly technique, we have also made contact between the electrode and the CNT deposited on the substrate with a pre-designed electrode by just dropping CNT solution on the substrate. We found that the obtained contact barrier between the CNT and the electrode is much higher than that found using the electric field assisted assembly technique. The reason might be that the electric field applied during assembling adds an additional force to the CNT that improves the contact.

We also tried to contact both ends of the CNT directly using the tips. With the feedback circuit, once one end of the CNT is connected to a tip, it is possible to touch the other end of the CNT as described above. The main problem we found is how to make the first contact on the CNT without breaking it. For electronic measurement, the CNT must be placed on an insulating substrate. To establish the first contact using our probe system, the only method we can use is to judge whether or not the tip touches the CNT by looking at the SEM image. Although the position of the tip in the plane perpendicular to the electron beam can be resolved with an accuracy of better than 2 nm, the height may only be estimated by the focusing position of the image resulting in an uncertainty of the order of a micron. Due to the error in height estimation, it is very hard to touch the CNT without breaking it. Furthermore, even when we successfully contact the CNT with one tip, such contact is not stable due to the small contacting area. Quite often, when we try to make contact to the other end of the CNT, the first contact has already failed due to vibration.

3.2. Measuring suspended CNTs

Studies have shown that the substrate can affect the electronic properties of CNT enormously [6–8]. To avoid the effect of the substrate, suspended CNT devices have been fabricated using the lithography method [6, 9, 11, 21, 22] and studies have been performed. In the present work, by using the probe system, we also performed measurements on suspended CNTs to avoid the effect of the substrate, and such measurement can also avoid breaking the CNT during establishing contacts.

3.2.1. Picking up a CNT. Suspended CNTs were brought into SEM using Pt probes as described in the experimental section. By looking at the secondary electron image inside the SEM we often found some CNTs protruding from the tip of the Pt probe, and shown in figure 3(a) is an example where a protruding CNT from the top Pt probe was connected by another sharp W tip from the bottom of the figure. After the contact was established, we then added a bias between the top Pt probe and the bottom W tip and measured the current going through the CNT. At the beginning, the current was lower than 10^{-7} A for a bias between 3 and 5 V. After keeping the bias for a while (several seconds to a few minutes), the current went up suddenly to 10^{-6}–10^{-5} A. Figure 3(b) shows the current versus time curve measured with a bias of 4.5 V. Initially, the current was at the 10^{-8} A level; after a while, the current went up to the highest current limit we set, i.e. 30 μA. The I–V curve was then measured and the result is shown in figure 3(c). A linear I–V curve was obtained, indicating the Ohmic characteristics of the system. We then raised the current limit to 50–100 μA and kept the bias at 5 V for a while; the CNT was then broken due to the large current passing through the CNT (the current density for a 30 nm diameter CNT at 30 μA current is more than 4×10^{10} A m^{-2}). A similar method has been used to attach CNTs to AFM tips [23]. Figure 3(d) is an SEM image showing that the CNT was broken with the lower part of the tube being connected to the sharp W tip. For two-terminal I–V measurements another tip was then moved to connect the top or the broken end of the CNT and I–V curves were measured as before.

3.2.2. Establishing Ohmic contacts. Freshly made W tips were used to contact CNTs for electronic measurement. Figure 4(a) is an SEM image showing a CNT being connected to two W tips. The CNT being measured in this case was about 1 μm long. The contact between the CNT and the upper W tip was made during transferring the CNT from the Pt probe to the W tip as described in the previous section. To make the lower contact, we moved the lower W tip to touch the CNT, and then added 3 V bias between the two tips. Initially the current was very low at about 10^{-8} A. However, after a few minutes, the current went up quickly to a level that was higher than the upper limit we set, i.e. 100 μA (see figure 4(b)). We then measured the I–V curve from -0.25 to $+0.25$ V and the so obtained linear I–V curve is shown in figure 4(c). The linear I–V curves were stable and had been repeated several times. The resistance calculated from this linear I–V curve is about 27 kΩ, indicating that good and Ohmic contacts had been made to the CNT.

By connecting the two sharp nano W tips directly, we measured the short circuit resistance of the system to be less than 1 kΩ. Therefore, the 27 kΩ resistance we obtained comes almost entirely from the part between the two tips. Besides the resistance of the CNT, there are also two contact resistances at the two contacting points. The Ohmic property of the system indicates that the two contacts are indeed Ohmic contacts.

In the Ohmic regime the resistance of a CNT does not change with the bias; the high resistance (or low current) we observed at the beginning (more than 10^8 Ω; see figure 4(b)) was mainly due to the contacts instead of the CNT. The contact between the upper tip and the CNT (figure 4(a)) was made

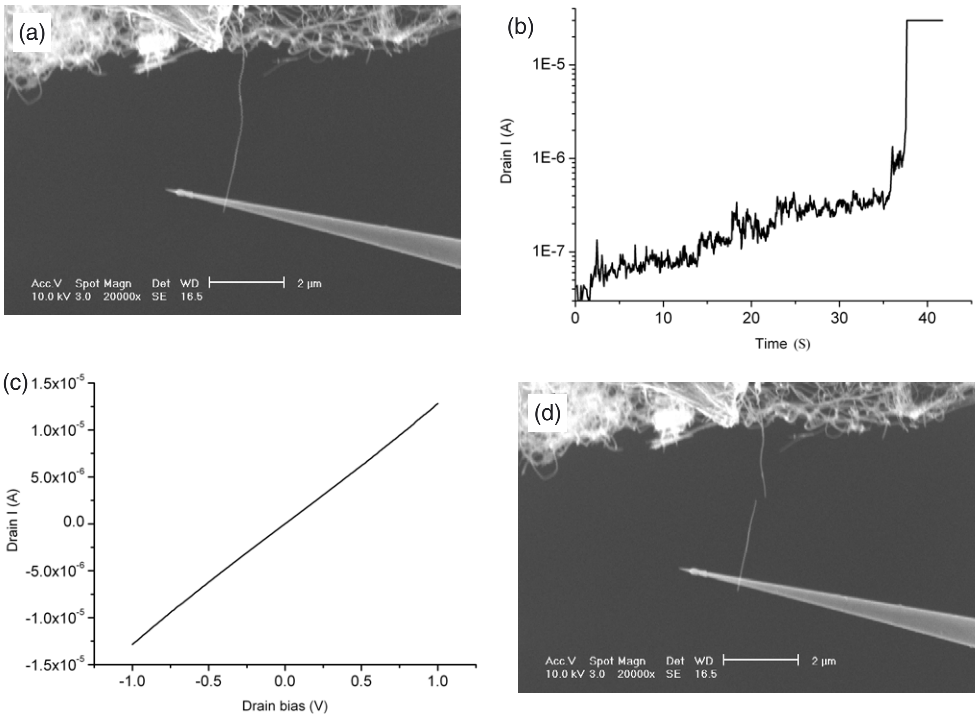

Figure 3. (a) SEM image showing a CNT protruding from the top Pt probe and being connected by a sharp W tip from below. (b) Current versus time curve measured with 4.5 V bias added between the W tip and the Pt probe. (c) I–V curve measured after the current went up to the 10^{-5} A level at 4.5 V bias. (d). SEM image showing the CNT was broken due to a large current and a part of the CNT was left on the W tip.

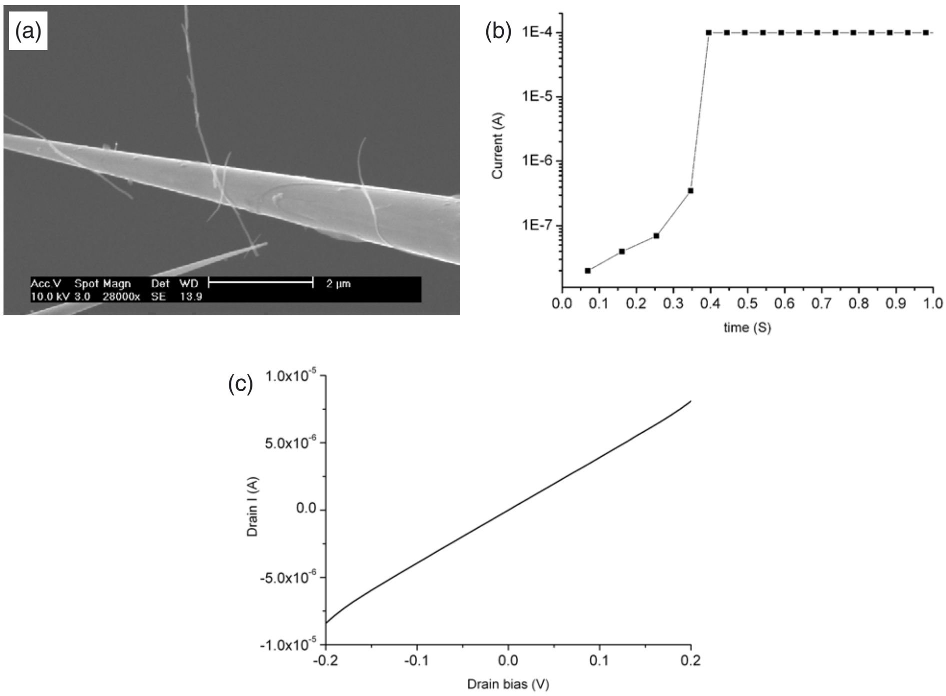

Figure 4. (a) SEM image showing a CNT being contacted by two freshly made W tips. (b) Time versus current curve measured with 3 V bias added between the two tips. (c) I–V curve measured after good contact was established.

during transfer of the CNT from the Pt probe to the W tip. The current in the circuit containing the Pt probe–CNT–W tip had reached the 10^{-5} A level at 3–5 V bias before the CNT was broken, and the total resistance in that circuit was of the order of 10^5 Ω. Therefore, the contact resistance for the upper contact was of the order of 10^5 Ω or less. The total

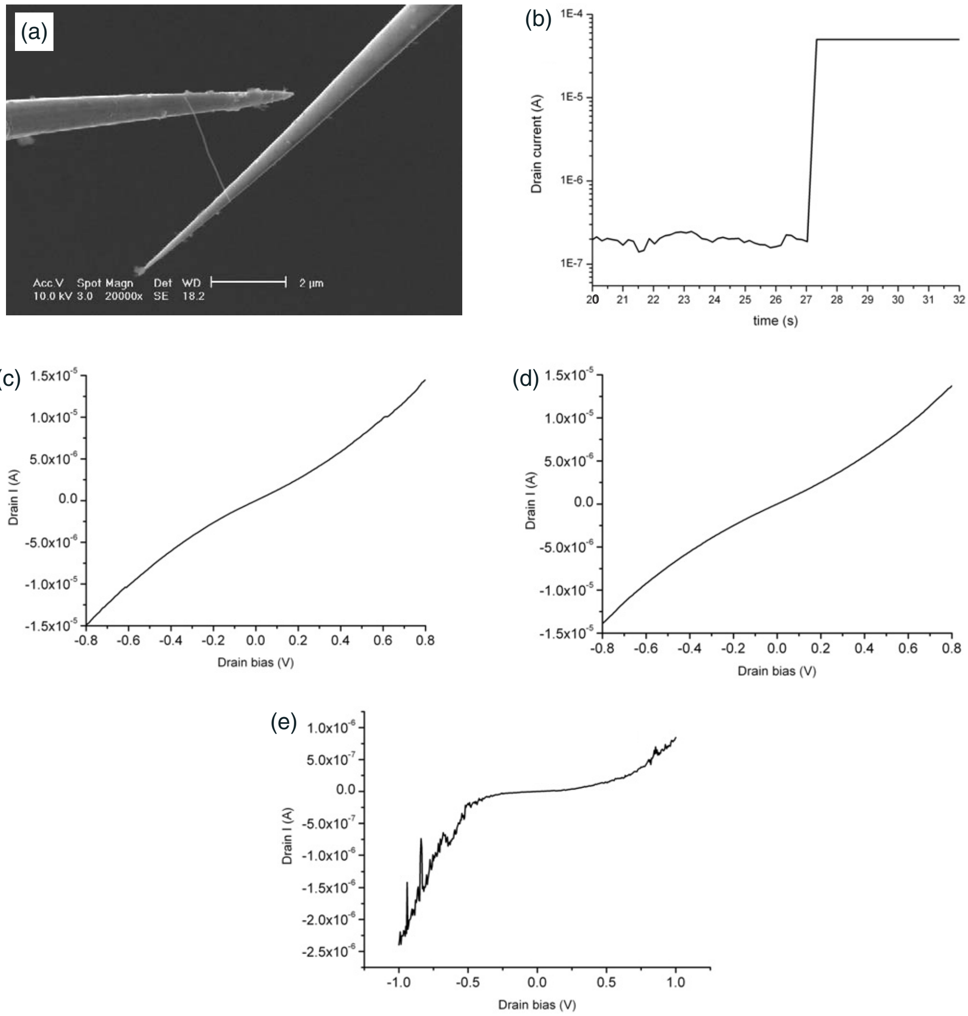

Figure 5. (a) SEM image showing a CNT being connected by two Au tips for electronic measurement. (b) Current versus time curve measured with a 2 V bias added to the two Au tips. (c) I–V curve measured after the contact was established as shown in (b)). (d) I–V curve measured after the lower contact was disconnected and then reconnected. (e) I–V curve measured for the same CNT having the same length but a poor contact.

resistance of about 10^8 Ω at the beginning of our experiment (see figure 4(b)) for the W-tip–CNT–W-tip system came mainly from the contact between the lower W tip and the CNT (see figure 4(a)). The bias added to the circuit dropped then mainly at the high resistance part, i.e. the second or lower contact. When the contact was not good, there existed a potential barrier at the interface between the W tip and the CNT. For a clean tip the interface was less than a couple of nanometres thick. When a high bias of several volts was applied across the interface, electrons might gain sufficient energy from the field to overcome the interface barrier and contribute to the total current. The structure of the interface might also change due to the large momentum exchange between the electrons and atoms of the interface. In addition, the heat generated by the current might also assist the structure change. Oxygen absorbed on the surfaces of the tip and CNT in air before they were moved into the vacuum might rearrange. Carbon atoms of the CNT and the metal atoms at the tip might be bonded strongly together at the interface as a result of the Joule heating effect, via e.g. the formation of tungsten carbites. Resistance at the interface may be reduced as a result of structure change leading to a larger current across the interface or contact. Larger current generates more heat and further lowers the contact resistance. As the contact resistance decreases, the voltage drop at the contact is lowered; finally, a balance is achieved and the resistance does not decrease anymore, leading to a stable current level. Although this effect is not a pure heating effect, we will still refer to it as the Joule effect for the convenience of our following discussions. The formation of the Ohmic contacts as evidenced in figure 4(c) results mainly from this effect. The CNTs we used in the present work have diameters of about 30 nm. The contact area between the CNT and the tip was about 15 nm wide and 100 nm long or shorter. Therefore, the current density at the contact area was around 6×10^{10} A m^{-2} when the current reached 100 μA.

To avoid effects resulting from the presence of the oxidation layer on the W tip, we also coated the freshly made W tip with Au to make an Au tip. The CNT selected for measurement using Au tips was first transferred onto an Au tip from the Pt probe. To establish the second contact to the

CNT, another Au tip was moved to touch the suspended end of the CNT attached to the first Au tip. Due to the adhesive van der Waals force, the CNT was attracted to the tip once it touched it. The connection was confirmed by moving the second Au tip gently and seeing that the CNT was moving together with the tip. Figure 5(a) is an SEM image showing a suspended CNT connected to two Au tips. We then added a bias of 2–5 V between the two tips and monitored the current passing through. Initially the current was very low because the contact was poor. We then kept the bias and let the current run for a while. Due to the Joule heating effect described above, the contact was improved and the current went up at the fixed bias within a second to at least 50 μA (goes up 10^3- to 10^6-fold), as shown in figure 5(b). To prevent burning out of the CNT, we had set the highest current limit to 50 μA. The I–V curve was then measured and the result is shown in figure 5(c). The curve is roughly linear and has a resistance at large bias around 40 kΩ. The same I–V curve was obtained repeatedly several times after good contact was made, indicating that the contact was stable. To verify the repeatability of the contact, the lower contact (figure 5(a)) was disconnected from the CNT (by pulling the lower tip away from the top tip) and then re-established several times and almost the same I–V curves were obtained (figure 5(d)). The weak non-linear behaviour in the low bias regime is the same in figures 5(c) and (d), indicating the existence of a small barrier for electron injection. A strong non-linear curve was also obtained for the same CNT having the same length when the contact was not good. Figure 5(e) shows an example. The CNT being measured in this case is about 2.5 μm long.

We also etched some Pt tips using electrochemical etching. The top right tip shown in figure 6(a) is a Pt tip made by electrochemical etching. An Ohmic contact was also established by the Joule heating effect and the linear I–V curve was obtained as shown in figure 6(b). The resistance is calculated to be 65 kΩ. The CNT being measured is about 3 μm.

3.2.3. e-beam exposure. We had been using a 10–30 keV electron beam in the SEM. With or without electron beam exposure of the CNT, we obtained the same I–V curve, indicating that the electron beam irradiation does not change the electronic properties of the CNTs significantly. However we found that electron beam exposure does have an obvious effect on the contact. The vacuum level in our SEM is about 4×10^{-6} mTorr in the sample chamber, so there exist some residual hydrocarbon molecules in the SEM. The hydrocarbon molecules decompose under the electron beam irradiation, resulting in carbonaceous material deposited on the exposed area. The deposited carbon was amorphous and was found to have resistivity in the order of 10^{11} Ω cm [24]. To avoid such carbon contamination, we had tried our best to avoid e-beam exposure to the tips especially under high magnification using high dose. However we found that after the tips (Au or fresh W, or other conductive tips) were exposed by the electron beam under low magnification for more than one hour only poor contacts were established between the tips and the CNT. When the tip was only covered by a thin layer of carbonaceous material, we found that e-beam exposure could improve the contact dramatically. In figure 7(a), the CNT was contacted to a Pt probe to the left of the figure (not shown). The right tip was an Au tip having been exposed under a regular electron beam for about one hour. When the tip just touched the CNT the current was very low (at 10^{-9} A level) even at 4 V bias. The situation did not change even after the bias had been added to the tips for more than 10 min. We then used the electron beam to intensely irradiate the contacting area; the resistance dropped and current went up dramatically to 10^{-6} A and then to 10^{-5} A. Figure 7(b) is the current versus time curve showing the current went up from 10^{-6} to 10^{-5} A. Unlike the case using the Joule heating effect (where the current went up from below 10^{-7} to 10^{-5}–10^{-4} A within a second), under the e-beam exposure the current went up rapidly but not to the highest current level in one step. When the current was stable (which means that the current stayed at a fixed level at a fixed bias, not shown in figure 7(b)), we measured the I–V curve and obtained a linear curve as shown in figure 7(c). The resistance is calculated to be about 44 kΩ. The result is repeatable and the I–V curve remained linear even at a large bias of 3 V, but the CNT burned out at 4 V.

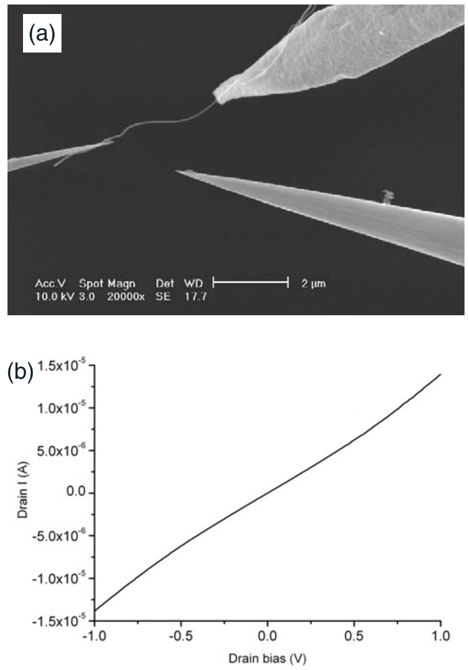

Figure 6. (a) SEM image showing a Pt tip (top right tip) connecting to a CNT. (b) I–V curve measured after Ohmic contact was been established.

When the tip was covered by a relatively thick layer of carbon contamination we found that although e-beam exposure could improve the contact an Ohmic contact could not be obtained any longer. Figure 8(a) is an SEM image showing two W tips being connected to one CNT. The lower contact was made to a fresh W tip using the Joule heating effect, which is an Ohmic contact. The top right W tip was an old tip that was covered by a relatively thick contamination layer. We found that adding a bias is not useful in improving the contact. We then used an electron beam to irradiate the contact area, and the current versus time curve under 1 V bias is shown in figure 8(b). Unlike the thin layer case, figure 8(b) shows that

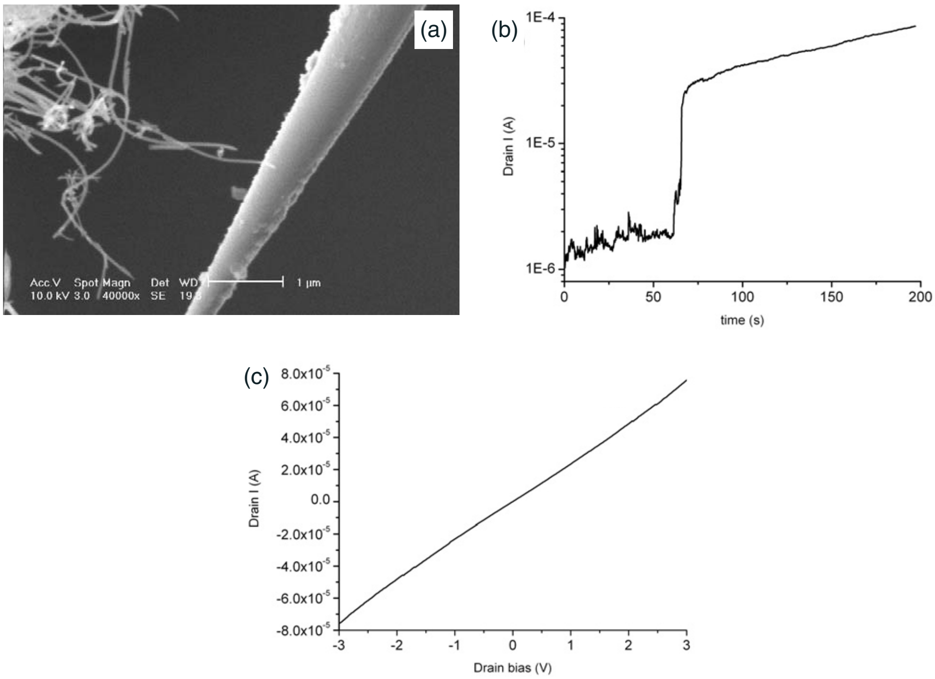

Figure 7. (a) SEM image showing an Au tip connecting to a CNT. The Au tip had been exposed to an electron beam for some time. (b) Current versus time curve measured when the contact area was exposed to an intense electron beam. (c) I–V curve measured after the Ohmic contact was established.

the current went up very slowly in this case. When the curve was stable, we performed I–V measurements. The I–V curves obtained were obviously non-linear (shown in figure 8(c)), indicating the existence of a potential barrier. The resistance at higher bias is calculated to be about 33 kΩ, which is at the same level as that of other CNTs we measured when the contact is Ohmic. The result was found to be repeatable.

We then broke the upper contact and re-established the contact again. The I–V curve obtained after e-beam exposure was found to be also non-linear (figure 8(d)). Furthermore, the resistance at large bias was found to be about 110 kΩ, that is higher than the previous case as shown in figure 8(c). The fact that the resistance is higher in the later case results, among other things, from the fact that the contamination layer was thicker after the last e-beam exposure, so that the potential barrier was thicker and affected the current even at large bias.

We now consider how e-beam irradiation improves the contact. The carbon due to e-beam induced deposition accumulating at the exposed area can strengthen the contact area. However, as such deposited carbon is amorphous with high resistance, the deposited carbon layer was not the reason that the contact resistance dropped dramatically. On the other hand, the electron beam of high energy (10 kV accelerated voltage used in this experiment) may knock out atoms in the contamination layer and cause atoms to rearrange at the contact interface. If the contamination layer is thin enough such rearrangement may produce some small area of less contamination and lead eventually to better contact with the help of the Joule effect. When the contamination layer is relatively thick, the atom rearrangement at the interface is less effective and the thick potential barrier remains. Nevertheless, the deposited carbon layer at the contact area may still strengthen the contact and improve the contact a little but not substantially.

3.2.4. Length dependence of the resistance of an MWCNT. The resistance of the same MWCNT was measured 14 times at different contacting locations. The length of the CNT lying between the source and the drain tips changed from 6.5 to 1.1 μm. All contacts were established with a 30 μA current limit using the Joule heating effect. Figure 9 shows the same CNT was contacted to the same tip at different positions and corresponding I–V curves were measured after the Ohmic contacts were established. The length of the CNT being measured was estimated from corresponding SEM images and the resistance was calculated from the linear I–V curves. The data in the -1 V to -0.8 V range were used for calculating the resistance to avoid the effect of any minor non-linearity. Figure 9(g) and table 1 show the result. It can be seen that the resistance of the CNT changes roughly linearly with the length being measured. The linear fit result is also shown in figure 9(g), giving a resistance of 20 kΩ μm^{-1}.

The total resistance of the CNT system results mainly from the two contacts and the CNT. We can write the resistance between the two tips as $R = R_c + B \cdot L$, where R_c is the contact resistance, L is the length of the CNT between the two electrodes, and B is the resistance of the CNT per length. The contact resistance of the system may be obtained by extending the linear fit to zero length, i.e. $L = 0$, giving $R_c = 60$ kΩ. The parameter B may also be obtained from the linear fit and 20 kΩ μm^{-1} is obtained for the MWCNT shown in figure 9.

Using SEM images of figure 9 we estimate the outer diameter of the CNT to be about 30 nm. Assuming the inner diameter of the CNT is 10 nm (see, e.g., figure 1(b), inset), we

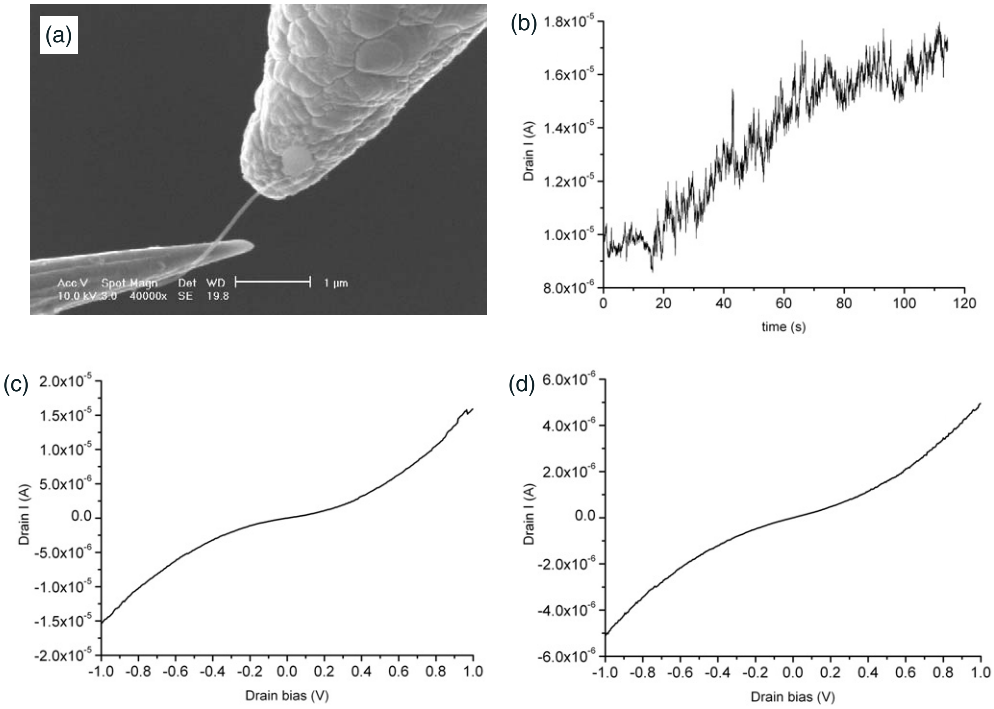

Figure 8. (a) SEM image showing a tip was covered by a thick contamination layer (top tip) and connected to a CNT. (b) Current versus time curve measured when the contact area was irradiated by an intense electron beam. (c) I–V curve measured when the contact became stable. (d) I–V curve measured after the contact was established for the second time.

can then calculate the resistivity of the CNT and obtain 7.5×10^{-4} Ω cm. The resistivities of MWCNTs have been measured previously [25, 26]. Ebbesen *et al* used the focused ion beam technique to make the electrodes and performed two-probe and four-probe measurements. Their four-probe measurements reveal great differences between nanotubes. Their two-probe measurements gave more concentrated results, and the resistivities measured from three CNTs are 7.5×10^{-5}, 1.2×10^{-4} and 9.8×10^{-4} Ω cm [25]. Dai *et al* used lithography to make a macroscopic electrode and used AFM tip as the second electrode and performed two-probe measurements. The smallest resistivity they obtained is 7.8×10^{-4} Ω cm from a CNT with 13.9 nm diameter [26]. Our result (which is 7.5×10^{-4} Ω cm) is comparable with the MWCNT resistivities reported previously. The ballistic transport property was not obtained from our measurements on CNTs that were suspended or on the substrate. The present MWCNTs were grown by the CVD method. It should be noted that the MWCNTs grown by CVD are defective, and it is expected that scattering by these defects will play an important role in the transport of CNTs, especially at low bias when the scattering is dominated by acoustic phonons and defects.

3.2.5. Three-terminal measurements. To obtain more information on the electronic properties of the CNT, three-terminal measurements are often carried out. We have demonstrated that three-terminal measurements are possible for a CNT sitting on the substrate if we can control the distance between the gate tip and the CNT. We have described how to use two tips to perform I–V measurement on suspended CNTs. Using a third tip we can add a gate voltage to the suspended CNT and study the field effect on it. Figure 10(a) is an SEM image showing the configuration of a three-terminal measurement. The tips are Au tips. The contacts between the CNT and the source and drain tips were Ohmic contacts established using the Joule heating effect. The centre tip used as the gate had been exposed to the electron beam for some time and had amorphous carbon being deposited on the surface to form an insulating layer. The tip was moved to just touch the CNT. Therefore, the distance between the metal gate and the CNT was the thickness of the contamination layer, which was a few nanometres. A linear I–V curve was obtained repeatedly at zero gate voltage (shown in figure 10(b)). The resistance is calculated to be 30 kΩ. Gate voltages of −2, 0 and 2 V were added and I–V curves were measured through the other two tips. The same result was obtained for different gate voltages, indicating the CNT is a metallic one.

We may also use a CNT or a nanowire attached to a conductive tip to form a very sharp tip. As shown in figure 11, such a kind of gate can be used for measuring a very short CNT or nanowire.

In a previous report, Chiu *et al* studied a carbon nanotube T junction [27]. A metallic CNT was used as the conducting channel between two electrodes. A carbon nanotube located roughly perpendicular to the metallic CNT and separated from it by an insulator molecule was used as a local gate. Measurements at 4 K show that the band structure of metallic nanotubes can be dramatically altered by the local electrostatic field. Our present configuration using a third tip or a CNT or a nanowire as a gate may perform a similar study as previously reported. Furthermore, our configuration can study the effect of the position change of the local electrostatic field on the conducting channel. However, so far, we have not been able to perform low temperature measurement yet, and our room

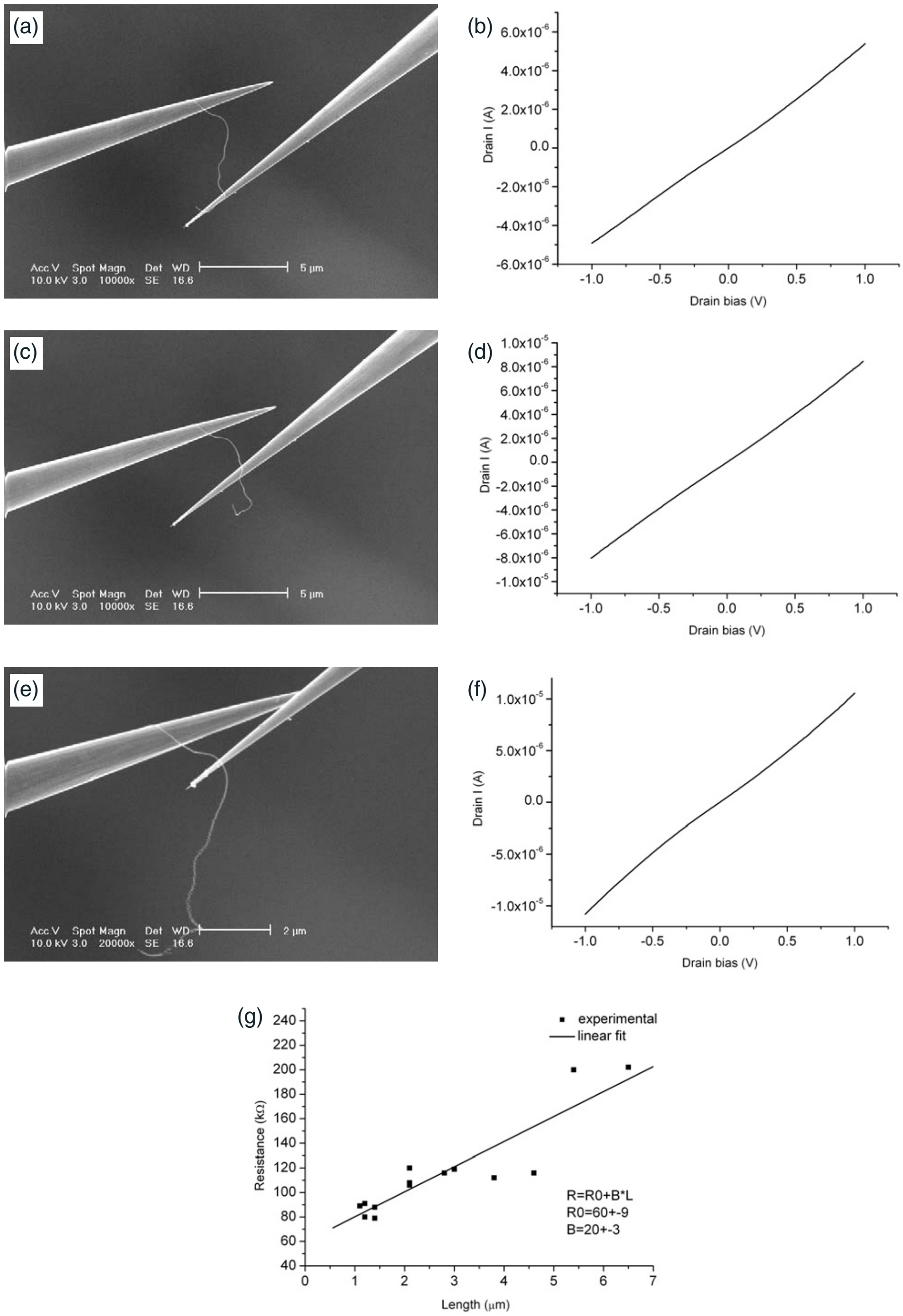

Figure 9. SEM images showing that the lower tip was contacting to the same CNT but at different positions ((a), (c), (e)). (b), (d), and (f) are I–V curves measured after Ohmic contacts were made for the configurations shown in (a), (c), and (e) respectively. (g) Resistance plot of 14 measurements on the same CNT.

temperature measurement on metallic CNTs so far has not shown any effect of the local gate.

4. Discussion

Our experiments show that the geometry of the tip may also affect the contact. When the CNT is perpendicular to the tip axis, the contact is relatively easy to make. When the CNT is parallel to the tip axis, due to the round shape of the tip, the CNT cannot stay steadily on the tip by the Van der Waals force. In such a case, using the Joule heating effect to establish good contact is not always successful.

We found that almost all the I–V curves we measured, with or without contact barrier, are symmetric or almost symmetric for positive and negative bias. In our experiments, most of the time the CNT was connected first to one tip on transferring the CNT from the Pt probe to the tip, and the contact was usually Ohmic. However a contact barrier usually existed for the other end. The symmetric or almost

Table 1. Resistances of the same CNT measured with different lengths between the two electrodes.

No.	01	05	06	07	08	09	10	12	13	15	16	17	18	19
Length (μm)	5.4	3.8	2.8	2.1	2.1	1.4	1.2	1.4	1.2	1.1	2.1	4.6	6.5	3.0
R (kΩ)	200	112	116	106	108	79	91	88	80	89	120	116	202	119

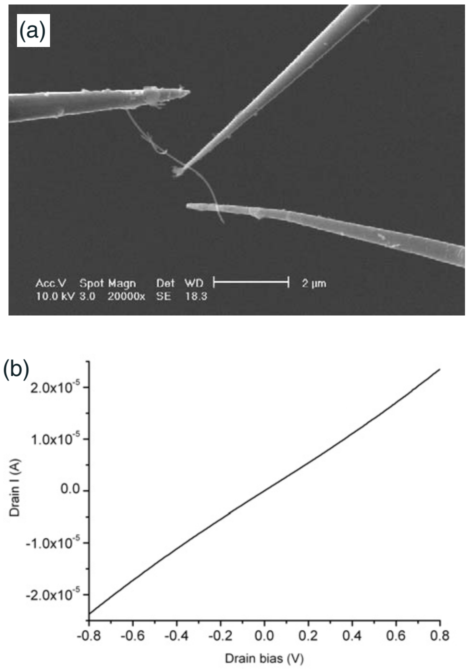

Figure 10. (a) SEM image showing a three-terminal measurement configuration. (b) I–V curve measured using the configuration shown in (a).

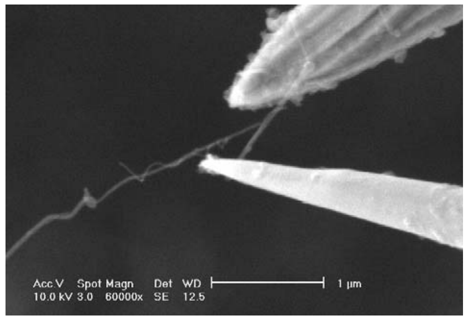

Figure 11. SEM image showing that a CNT can act as a gate probe.

symmetric I–V curves indicate that the contact barrier shape is not significantly affected by the bias direction. This is different from the Schottky barrier described in [3]. Our observations show that the contact barrier height is mainly affected by the thickness of the contamination layer but does not show a clear relationship with the tip materials. We believe that such a contact barrier was caused mainly by the interfacial insulating layer having a roughly squared shape.

Another phenomenon we found is that regardless of the quality of the contact the current change is always linear with the voltage at very low bias. Theoretical analysis of electrodes separated by a thin insulating film shows that the current goes linearly with the voltage when the bias is nearly equal to zero [28]. This analysis also shows that when the two electrodes have different work functions, e.g. the barrier height at the interface of the electrode and the insulator is asymmetric, for small bias ($V < \varphi 1$, where $\varphi 1$ is the barrier height at the interface of electrode 1 and the insulator, the smaller one) the ρ–V characteristic is only slightly asymmetric, while for the ideal trapezoidal barrier it is symmetric [29]. In our experiments, two electrodes connect to the two ends of one CNT with interface layers. When the interface layers are insulating, the structure can be modelled as composed of two electrode–thin insulator film–CNT structures. The bias at around 0 V is smaller than the work function of the electrode and the CNT. The ρ–V and the I–V are symmetric for both ends of the CNT, and are therefore symmetric. This is consistent with our assumption that the interface is not a Schottky barrier; rather, it is an insulating layer.

So far, we have observed mainly metallic behaviour rather than semiconducting behaviour for the MWCNTs we measured. This is because the CNTs we measured are typically 30 nm or larger. The bandgap associated with a semiconducting CNT of such a diameter may be estimated to be less than 30 meV, that is comparable with thermal agitation energy of room temperature energy (24 meV). Therefore, all CNTs with diameter larger than 30 nm behave basically as metallic, regardless of their structure at room temperature.

5. Conclusions

Good contacts have been established using a nanoprobe system inside the SEM for CNTs sitting on the substrate as well as for suspended CNTs, and corresponding I–V curves have been measured. It was found that the CNTs we measured have resistance ranging from 14 to 200 kΩ.

When the CNTs are sitting on the substrate, good contact can be made by directly pressing a freshly made W tip onto the CNT.

For suspended CNTs, Ohmic contact between the freshly made W tip or Au tip and Pt tip and the CNT can be established using the Joule heating effect. E-beam exposure on the contact area can improve the contact between the CNT and the tips that was covered by a thin layer of carbonaceous materials. When the contamination layer is very thin, Ohmic contact can be established using e-beam irradiation. When the contamination layer is thick, a contact barrier exists and cannot be eliminated even after long e-beam exposure.

The resistance of a CNT about 30 nm in diameter is found to change roughly linearly with the length at a ratio of 20 kΩ μm^{-1}. A resistivity of 7.5×10^{-4} Ω cm is obtained, which is comparable with that reported in the literature.

Field effect measurements can be carried out inside the SEM with a movable gate. A third tip or a CNT or a nanowire connected to a tip have been used as the gate. Such measurements can be made either for CNTs sitting on the substrate or for suspended CNTs.

Acknowledgments

We thank Dr X L Liang, Dr S Gao and Mr M S Wang for useful discussions. The first author thanks Mr X L Wei for helping make the tips. This work was supported by the MOST (973 grant No 001CB610502; 863 grant No 2004AA302G11), NSF of China (grant No 60271004), the Key Project and the Program for New Century Excellent Talents in the University of the Chinese Ministry of Education. We would also like to thank the National Centre for Nanoscience and Nanotechnology of China and Peking University.

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