Sensitivity of single multiwalled carbon nanotubes to the environment

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Abstract. We report on electrical resistance measurements of single multiwalled carbon nanotubes (MWNTs) in different environments (ambient air, $\text{H}_2$, $\text{O}_2$ and the electrolytes LiClO$_4$, KCl, KMnO$_4$ and $\text{H}_3\text{PO}_4$). The gate dependence is studied using back-gating, electrochemical gating and gates evaporated directly onto the nanotubes (NTs). MWNTs at room temperature are p-doped. Upon changing the environment a change of the doping state of the MWNTs is inferred from the linear resistance. The effect of the environment on the contacts is negligible in our experiments. The p-doping is proposed to originate from the specific adsorption of an oriented dipole layer of water on the nanotube, which is affected by the kind of ions.

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1. Introduction

There is hope that single molecules will function as the active element in electronics [1]. Being composed of a surface only, a strong sensitivity to environmental conditions is expected. In the field of conducting polymers, the sensitivity of conjugated oligomers/polymers to the environment, in particular to oxygen, has been recognized a long time ago [2].

Carbon nanotubes (NTs) are fascinating materials with interesting mechanical and electrical properties [3], but carbon NTs are not true molecules. They are molecules with regard to those properties that arise from the small diameter of the NTs, which for single-walled carbon nanotubes (SWNTs) can be as small as 1 nm. Due to their long length, typically larger than 1 µm, translational invariance is assumed, which gives rise to a continuous band structure. Hence, in this respect, NTs are solid-state-like objects. Common to conjugated molecules, the conduction electrons in carbon NTs arise from delocalized $\pi$-orbitals tightly bound to the surface of the NTs. Because of the mechanical rigidity and the relative ease of contacting single NTs, they are good model systems for studying the environmental effect on conjugated $\pi$-orbitals derived from carbon–carbon bonds.

Carbon NTs are sufficiently large that conventional lithography techniques suffice to contact a single NT. This allows us to go beyond simple two-terminal resistance measurements. For example, four-terminal measurements have been realized [4]. Moreover, a strong electric-field effect has been discovered in semiconducting SWNTs by Tans et al. [5] and Martel et al. [6], which triggered a large body of research on NT-based field-effect transistors (FET). SWNT-FETs were found to be p-type. This is surprising, because an ideal carbon NT should display particle–hole symmetry. Hence, a semiconducting NT should be intrinsic. The characteristic p-type behaviour [7] may suggest that NTs are p-doped. Alternatively, it was suggested that the Fermi level may be pinned at the metal–NT contact in the vicinity of the valence band [5].

That NTs can be intentionally n-doped with potassium as the dopant was demonstrated in mats of NTs [8]–[10] and more recently in single tubes [11]. Furthermore, NTs in a vacuum revealed n-type behaviour, as judged from a negative thermoelectric power [12, 13]. Combining as-grown (p-type) NT segments with vacuum annealed (n-type) ones allowed the realization of p–n junctions [14] and complementary FET devices [15].

In the case of potassium treatment, the n-type characteristics are due to the potassium acting as a donor. But what is the source that breaks particle–hole symmetry in as-grown (if not specified
otherwise, in ambient) and vacuum annealed NTs? Insight into this question comes from studies of NT devices operated in different gases. A remarkable sensitivity to, for example, O\textsubscript{2}, NO\textsubscript{2} and NH\textsubscript{3} was reported, with sub-ppm sensitivity to NO\textsubscript{2} [16, 12, 17].

To make a long story short, oxidizing gases like O\textsubscript{2} and NO\textsubscript{2} were shown to increase the electrical conductance of the NTs, while the reducing gas NH\textsubscript{3} does the opposite. Such a dependence is consistent with ‘intrinsic’ p-type behaviour. If oxidized, the hole carrier density increases and thereby the conductance, while if reduced, by, for example, NH\textsubscript{3}, the net hole–carrier density is decreased. This model of bulk NT doping is appealing, but it is not the only one. One has to recognize that the resistance of a NT-FET has two parts: a bulk resistance and contact resistances due to the NT–metal contacts. It has been argued that the apparent p-type behaviour is due to a workfunction mismatch between the NT and the metal contacts, leading to the formation of Schottky barriers at the contacts [18, 19]. With regard to gas sensitivity a strong threshold shift has been seen in the work of Kong et al [16]. It may very well be that the primary effect of the gas is to change the barrier at the metal–NT contact.

Whether the doping state of the NT as a whole is affected by its environment can be clarified if multiwalled carbon nanotubes (MWNTs) are used. On the typical length scale of NT devices, SWNTs can be ballistic, even at room temperature [20]. In contrast, it has been demonstrated that in well contacted MWNTs the dominant part of the potential gradually drops along the NT [20]. Hence, contacted MWNTs at room temperature can be regarded as diffusive wires whose bulk properties determine the measured two-terminal resistance. Unfortunately, there is much less work on the environmental sensitivity of MWNTs. In a recent study with CVD-grown MWNTs (macroscopic assembly of tubes) Varghese et al [21] observed a strong sensitivity to water vapour. Water vapour increases the conductance, while NH\textsubscript{3} decreases it. The strong sensitivity to water has been mentioned before in the work of Krüger et al [22] who immersed the NT devices fully into an aqueous solution. A very strong gating efficiency was achieved in an electrolyte through which the NT is electrochemically gated. This work has been extended to SWNTs and established a solid starting point for liquid ion and biosensors that operate in aqueous solutions [23]. Using electrochemical gating (EC gating), Krüger et al showed that MWNTs are p-doped in water. Fermi-level shifts as large as $\Delta E_F = 1$ eV were measured (the positive sign refers to hole doping). $\Delta E_F$ in an ambient environment could be estimated to be $\approx 0.3$ eV. Because the Fermi-level shift is larger than the subband spacing for a typical MWNT, the distinction between semiconducting and metallic tubes is no longer observable in transport.

In this work, we extend the previous work on the EC gating of single MWNTs to different solutions and compare them with gas experiments using ambient air, O\textsubscript{2}, vacuum with or without thermal annealing and forming gas (N\textsubscript{2} (98%):H\textsubscript{2} (2%) mixture). Moreover, the effect of an Al gate, evaporated directly over the NTs, is studied.

2. Experiment

We use arc-discharge grown and purified MWNTs, which were shown to be highly graphitized [24, 25]. Single MWNTs are contacted using a combination of optical and electron-beam lithography. The contact material is Au (50–200 nm in thickness) evaporated over the tubes [26]. Two-terminal and four-terminal devices are used, see figures 1(a)–(c). Contact separations range from 300 nm to 2 µm. As substrates we use degenerately doped Si wafers which are thermally oxidized to a thickness of 400 nm. The conducting substrate serves as a gate electrode (back-gate) in measurements performed in a gaseous environment (figure 1(d)).
Figure 1. (a)–(c) Typical single MWNT devices, electrically contacted with two (b) or four (c) Au electrodes. The electric-field effect is studied using back-gating (d) or electrochemical gating (EC gating) in aqueous electrolytes (e). In the latter, the NT is immersed in an electrolyte. (f) A typical measurement of the linear resistance $R$ as a function of EC gate voltage $U_g$ in aqueous LiClO$_4$.

For EC gating, the NT device is immersed in a small droplet (~200 µm in size) of an electrolyte. The droplet is delivered by a micropipette attached to a micromanipulator. Gating is achieved by applying a voltage $U_g$ to the Pt electrode relative to the NT, set on ground potential (figure 1(e)). At positive $U_g$ cations are attracted to the NT where a thin charge double layer is formed, which is counterbalanced by excess electrons in the NT. Hence, the Fermi energy $E_F$ of the NT increases [22]. Because double layers are thin, the effective capacitance is large. This, together with the complete immersion, results in a very strongly coupled gate [22, 23].

The linear response NT resistance $R$ is determined from the response to a small ac voltage (~1 mV at ~30 Hz) applied over the NT. $R$ ranges between 1 and 10 kΩ at zero gate voltage. Simultaneous to measuring $R$ the current $I_g$ through the gate electrode is monitored. This current is immeasurably small if the back-gate is used. In an electrolyte, the current never exceeded $I_g \approx 100$ nA. The largest part of this ion current does not, however, flow through the NT but originates from the Au contact pattern, which is partially covered by the electrolyte.

A typical EC gating measurement is shown in figure 1(f). With increasing gate voltage $U_g$, the resistance $R$ first increases, runs through a maximum and decreases thereafter. As a solvent,
an 0.01 M LiClO$_4$ aqueous solution was used. This evolution of $R(U_g)$ can be understood by the Fermi energy shift $\Delta E_F$, which to first order is given by the simple relation $\Delta E_F \approx eU_g$ and the corresponding change in density of states \[22\]. The resistance increase with $U_g$ (or Fermi energy) corresponds to a p-type characteristic, while the resistance decrease at $U_g \gtrsim 1.0$ V corresponds to an n-type characteristic. Therefore, the charge-neutrality point (CNP), where the NT should obey particle–hole symmetry, is positioned at $U_g \approx 1$ V. In other words, if we do not intentionally push the Fermi level upwards with an external field, it will be located below the CNP, deep in the valence band.

The pronounced resistance peak, which is positioned at a relatively large gate voltage $U_g \approx 1$ V in aqueous LiClO$_4$, has been observed in many samples with different resistances, in two-terminal as well as four-terminal configurations. We are therefore convinced that this phenomenon cannot be assigned to the NT–Au contacts, but is intrinsic to the NT. Hence, NTs are strongly p-doped in water, more precisely in aqueous LiClO$_4$ (further discussion follows in section 4). All measured curves show hysteresis of varying degrees (e.g. figures 1(f) and 2(a)), although sweeping is done fairly slowly (cycle time $\approx 10$ min). The origin of the hysteresis is not known at present.

3. Electrochemical versus back-gating

Figure 2(a) shows a four-terminal resistance measurement over a large EC gate voltage range in 0.4 M LiClO$_4$ in H$_2$O. In agreement with figure 1(f) a resistance peak is observed close to $U_g = 1.0$ V. The maximum voltage swing, i.e. $|U_g| \approx 1.5$ V, is limited by the gate current $I_g$, which starts to increase markedly for large gate voltages, in particular for $U_g < -1.0$ V. This is most likely due to the onset of electrochemical reactions, leading to hydrogen and oxygen evolution. There are, however, no bubbles observable under the optical microscope, which is used to position the micropipette, as long as $|U_g| \lesssim 1.5$ V. If we sweep to negative voltages, the resistance starts to increase sharply at $U_g \approx -0.5$ V. If one is not careful the device may be destroyed (i.e. infinite resistance results) beyond some threshold. If one is lucky and stops before device failure, the initially measured curve cannot be re-traced. While the characteristic peak at $U_g \approx 1.0$ V still exists, the total resistance has irreversibly increased. This added resistance may stem from the NT–Au contact which has been changed by an electrochemical reaction. However, electrochemical reactions on the NT itself, such as the reduction of oxygen, cannot readily be excluded \[27\].

In figure 2(b) we compare the gating characteristic for back-gating (inset) with the one for EC gating (again in aqueous LiClO$_4$ at 0.01 M). The $R(V_g)$ dependence (back-gate) was measured over a large window of $V_g = -50 \ldots 50$ V under ambient conditions immediately before the electrolyte droplet was applied and $R(U_g)$ was measured (main figure). Around zero gate voltage, $R$ increases with gate voltage in both cases, but the magnitude of change is much larger if the electrolyte is used. There is a change of 0.16% V$^{-1}$ with back-gating and of 200% V$^{-1}$ with EC gating. Hence, the difference is larger than 1000 \[22\]. Important for the present discussion is the fact that $R$ increases with gate voltage in both situations, showing that the doping state is the same. The NT is p-doped in the aqueous solution and in air. Recently, Zahab et al \[28\] suggested that NT (in their case SWNT) can be changed from p-type to n-type by the adsorption of water. Their conclusion is based on a small resistance increase following the injection of water vapour in a set-up in which the NT device is kept in a chamber. Contrary
to their observation, the resistance always decreases by 10–20% if the water droplet is applied in our experiment. This observation is in agreement with measurements in a gas chamber which we report below.

The decrease in resistance, following the change of environment from air to water, suggests an increase in the doping level. The CNP moves deeper into the valence band, resulting in a right shift of the characteristic resistance peak. Based on figures 1 and 2 we conclude that MWNT devices show p-type behaviour in air. If immersed in water, more precisely into an aqueous LiClO₄ electrolyte at 0.01–0.1 M concentration, the level of p-doping is increased. To gain an insight into the origin of the doping, different aqueous electrolytes have been tested.

4. Different electrolytes

Figure 3 compares the measured normalized resistance as a function of the EC gate voltage $U_g$ for three equally concentrated (0.01 M) aqueous solutions of LiClO₄ (■), KMnO₄ (○) and H₃PO₃$^{Note}$ (□) at pH = 6. All three examples show a clear resistance peak (arrows). Relative to LiClO₄, the peak position is shifted to higher voltages for KMnO₄ ($\approx$1.2 V) and to lower

$^{Note}$ KOH was added to H₃PO₃ to adjust the pH to 6.

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Figure 3. Comparison of EC gating in different aqueous solutions (LiClO$_4$: ■, KMnO$_4$: ○ and H$_3$PO$_3$: □), all 0.01 M at pH = 6. Note that the peak is shifted to the right (left) for KMnO$_4$ (H$_3$PO$_3$) relative to the position in LiClO$_4$. Also shown is a measurement in a non-aqueous electrolyte, i.e. in 0.3 M LiClO$_4$ in PC.

voltages for H$_3$PO$_3$ (∼0.2 V). In KMnO$_4$ solutions the peak position is often even larger and can exceed 1.5 V in higher concentrated solutions (∼0.1 M). This comparison shows that water alone is not the only factor which determines the doping state of the NTs; the electrochemical environment matters. It also shows that a relatively strong oxidizing agent (KMnO$_4$) shifts the Fermi level deeper into the valence band, hence oxidizes the NT. In contrast, a strong reducing agent (H$_3$PO$_3$) does the opposite. $R(U_g)$ was also measured in aqueous KCl (not shown). The resistance peak resembles the dependence in LiClO$_4$, both in magnitude and peak position.

We have emphasized that the electrochemical environment matters, in addition to water. But water is crucial, as is strikingly demonstrated by a measurement in a non-aqueous electrolyte, i.e. in LiClO$_4$ in propylene carbonate (PC). The relevant $R(U_g)$ is shown in figure 3 (△). The shape of the curve is similar to the LiClO$_4$:H$_2$O case, but the peak position differs markedly: the resistance maximum is close to $U_g = 0$ V in PC, as compared to $U_g \approx 1$ V in H$_2$O. Hence, in the non-aqueous solvent the NT is undoped, demonstrating that water is a prerequisite for the p-doped state of NTs.

Because no microscopic picture has yet been proposed, we briefly discuss a possible scenario. First, we emphasize that, if the applied gate voltage is always kept small enough ($U_g \lesssim 1$ V), the measured samples can be recycled, i.e. flushed in water, dried and re-immersed, even in a different solvent. Hence, the oxidized state of the NTs (p-doped state) cannot originate from the oxidation of the carbon–carbon bonds with a corresponding loss of the graphitic network, which would be irreversible and associated with a permanent resistance increase. We rather think that a polarized or charged layer is specifically adsorbed on the NT surface. Since water is a prerequisite for the p-state in electrolytes, H$_2$O may adsorb in the form of an oriented dipole layer, inducing a partial charge transfer. In addition to H$_2$O, anions, e.g. OH$^-$, Cl$^-$, ClO$_4^-$ and MnO$_4^{2-}$, may adsorb specifically and increase the effective p-doping. If this hypothesis is true, the observed ‘generic’ p-character of NTs is of an electrochemical nature. Moreover, pure oxygen (O$_2$) is predicted to have a minor effect as compared to water or ambient (humid) air. Before discussing our gas experiments, we briefly mention a solid-state approach to close-proximity gating.
Strong gating allows us to move the Fermi level over a large energy window of the order of 1 eV, as was demonstrated with the electrolyte. For fundamental transport studies one would like to explore $R$ as a function of temperature, which, however, is not possible with an electrolyte. In an attempt to realize a solid-state strong coupled gate, Al has been evaporated directly over the MWNT in an evaporation chamber at a back pressure of $10^{-7}$ mbar. Surprisingly, this procedure does not lead to an electrical short between NT and the Al contact. In contrast, the Al–NT resistance is immeasurably large. Aluminium is a good getter and easily oxidized. We suspect that the first few monolayers are rather Al oxide than metallic Al.

A picture of a device is shown in figure 4(a). Note that only 50% of the NT is covered by the Al gate. In figure 4(b) the NT resistance as a function of gate voltage applied to the Al gate is shown. This curve has been obtained in ambient. Similar to the EC gating experiments, a resistance maximum is observed and there is hysteresis. Unlike the electrochemical case, the magnitude of the resistance change is small and amounts to less than 20% for a sweep range of $-1.5$ to $1.5$ V. Moreover, the hump is very much wider. The position of maximum resistance is always located near $U_g = 0$ V with a variation of $\pm 0.25$ V, suggesting that the NT is close to charge neutral, similar to the H$_3$PO$_3$ case. This observation supports our finding, discussed above, because Al is a strong reducing ‘agent’ which neutralizes the species adsorbed on the NT upon which the Al is oxidized. Since it is very unlikely that Al diffuses into the NT at room temperature, the NT dopants must be located at the surface. To understand the relatively small magnitude of the resistance change and the wide hump, one has to recognize that half of the NT is not covered by the gate and is still in contact with the ambient environment. Hence, the uncovered part is relatively strongly p-doped, while the part underneath the gate is nearly intrinsic or even n-type. It is therefore likely that Schottky barriers have formed. The configuration then corresponds to two diodes opposing each other. This yields the relatively small resistance change. The wide hump may be due to the additional side gating from the Al contact to the uncovered parts of the NT.
6. Gas exposure experiments

We have also studied the sensitivity of single MWNT devices to the exposure of oxygen, air and forming gas (N$_2$ (98%):H$_2$ (2%) mixture). In particular oxygen is interesting, because it was suggested to be responsible for the observed generic p-doped state of NTs in air [16]. The devices were mounted in an annealing oven. A vacuum at the level of a roughening pump can be obtained, i.e. $\approx$1 mbar. Temperature ramps can be programmed and gas can be flushed through the chamber.

Figure 5 shows the result (one of several). First, the chamber is evacuated. Since the chamber is small, the base pressure is reached very quickly. We have monitored the electrical resistance $R$ over a long period of time with the device in vacuum. This measurement is shown in the inset of figure 5(a). $R$ increases from 1.85 to 2.25 k$\Omega$, suggesting a decrease in carrier density and hence a decrease in doping level. Then, the NT device is heated, first to 50°C (not shown) and in a second cycle to 220°C (figure 5(a), □: first heating, ■: cooling and further heating cycles). The temperature ramps are done slowly and typically last 1 h. The resistance evolves nearly reversibly within the low temperature cycle. In contrast, there is an irreversible change which sets in at $T \approx 80$–100°C. This irreversible change is visible in $R(T)$ as an increase (up-pointing
Having annealed the NTs to a temperature of $\gtrsim 150^\circ$C, $R(T)$ becomes reversible again. Importantly, if cooled down to room temperature, there is a permanent resistance increase, 23% in the example of figure 5(a).

This finding further substantiates our picture that adsorbates are responsible for the p-doping state of NTs. As has been shown before water has a large influence, it is tempting to associate the annealing effect with the desorption of water. We have also measured the gate dependence $R(V_g)$ (back-gate) before and after annealing within the vacuum chamber. The slope $dR/dV_g$ is positive before and after annealing, but with a decreased magnitude afterwards. Unlike annealed SWNTs, which revealed n-type behaviour, as judged from a negative thermoelectric power [12, 13], our MWNTs display p-type characteristics before and after annealing. However, the doping level has been reduced during the annealing cycle.

Figure 5(b) shows the result of gas exposure on two different MWNTs. Before gassing, the devices were annealed in vacuum at 50 °C for 24 h. Thereafter, the devices were maintained at 50 °C while gases were added. The curves denoted by I (□) and II (■) were measured in O$_2$ (99.995% purity) and air at 250 mbar, respectively. In the second experiment (curve II), the gas was pumped off at $t_1$ and, at $t_2$, forming gas was added. This figure clearly demonstrates that the effect of oxygen (I) is moderate as compared to air (II). Because a large effect has been seen in water, we suspect that the origin of the resistance change in the air dosing experiment is water vapour. Though we have not studied water vapour alone, Varghese et al. [21] have observed a strong sensitivity to water vapour in macroscopic assemblies of CVD-grown MWNTs, which supports this interpretation. Figure 5(b) also demonstrates that adding forming gas, which contains only 2% of hydrogen, initiates an additional pronounced resistance increase (lowering the p-doping level), even after an extended period of vacuum annealing. This is in agreement with the EC gating experiment where adding the reducing agent H$_3$PO$_3$ substantially lowered the p-level, hence reducing the concentration of dopants.

7. Summary

Our experiments demonstrate that single MWNTs are very sensitive to the environment. They are p-doped if kept in ambient. The dopants are most likely adsorbates that can be (partially) removed if annealed at $T \gtrsim 100^\circ$C. Oxygen (O$_2$) plays only a minor role as compared to water (H$_2$O). In addition to H$_2$O, electrochemical gating experiments using different aqueous electrolytes demonstrate that the type of ions has an effect on the doping state. Adsorbed anions, for example OH$^-$, are a likely source of p-doping. The sensitivity to water and ions in water can be exploited in environmental sensing applications, as proposed by Krüger et al. [22]. Among the many possibilities we mention humidity [29] and pH sensing [30].

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