

Origin of Hysteresis in Carbon Nanotube Field-Effect Transistors

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ABSTRACT

Carbon nanotube field effect transistors (CNT FETs) have many possible applications in future nano-electronics due to their excellent electrical properties. However, one of the major challenges regarding their performance is the noticeable gate hysteresis which is often displayed in their transfer characteristics. The hysteresis phenomenon is often attributed to water-mediated charge transfer between the CNT and the dielectric layer or the CNT and the water layer itself. In this study, we implement three different experimental techniques and provide evidence that the hysteresis phenomenon of suspended CNT FETs, as well as of on-surface CNT FETs which operate at low gate voltage regimes ($|V_g| < 3V$), is based on gate-induced, water-assisted redistribution of mobile charge on the SiO₂ surface, and it is not related to charge injection from the CNT itself. Two techniques are based on the current measurements through the CNT and the third utilizes electrostatic force microscopy (EFM) setup. In addition, the applied external gate voltage affect the relaxation time of the current. This change arises from the modification of the amount of water layers which adsorb onto the dielectric surface, which caused by dielectrophoresis attraction between the water molecules and the substrate. It is found that the relaxation time, and hence the surface conductivity, are very sensitive for the first few layers, and saturates above three monolayers of water molecules.

INTRODUCTION

Carbon nanotube field effect transistors (CNT FETs) possess excellent electrical properties which make them promising candidate for future electronics. Their high sensitivity to various analytes have been the subject of many studies, which are mainly focused on device applications. Yet, several unresolved issues need to be clarified before further successful utilization of these devices can take place. One of these issues is the noticeable gate hysteresis that CNT FETs usually exhibit in their transfer characteristics. This phenomenon has previously been explained by several different models [1-6]. Currently, the common belief is that gate hysteresis is been attributed to stationary charge traps within the dielectric, which are filled and emptied by the applied gate voltage with charge transfer to and from the CNT itself [7-11]. These findings suggest that CNTs behave as leaky FETs where charges can flow back and forth between the CNT and the surface, and that the total tube charge is changing with time. The hysteresis phenomenon was observed for suspended CNTs as well [12], and it has been shown that water molecules give a large contribution to the hysteresis. Therefore, it has been suggested that the hysteresis is due to a water layer which is bound to the surface of the CNT, and behaves as charge trap or mediator [12-14].

A simple model for this behavior which assumes linear relation between the depleted charges and the total current, is based on an RC circuit where the tube is presented by a leaky capacitor with resistance R [15]. In this paper, we introduce RC model as well, but invoke that for suspended CNT FETs, as well as for on-surface CNT FETs which operate at low gate potentials ($|V_g| < 3V$),

the resistances as well as the capacitive elements arise mainly from the oxide itself and not from leakage current to or from the CNT. In 2002, Bradley et al. [16] have controllably introduced hysteresis by coating CNT FETs with charged polymers, and proposed that the effect results from ionic motion. Below we present an extensive study of hysteresis in suspended CNTs, and argue that mobile charges are indeed relevant. Using a method to calculate the hysteresis accurate time constants, we provide experimental evidence that the hysteresis is caused by water assisted movement of mobile charges on the SiO₂ surface. This movement, which aims to bring the system to a lower energetic configuration, is a direct result of the gate bias. Furthermore, we provide evidence that the above-mentioned mechanism underlies the hysteresis phenomenon in on-surface CNT FETs as well for low gate operational biases. Our model is examined using a technique which implements the usage of electrostatic force microscopy (EFM) to gain quantitative information on the substrate lateral and temporal charge distribution, CNT currents, and hysteresis time constants, and the results support our hypothesis. Moreover, a dependence of the hysteresis time constants on the applied external gates is found and is attributed to the modification of the surface humidity with electric field. This allows us to detect the amount of water layers with single monolayer sensitivity.

EXPERIMENTAL DETAILS

Suspended CNT FETs were fabricated using a growth at-the-end method [17, 18] in order to avoid any additional sources for the hysteretic phenomenon from residual process contamination. The processing was performed on 1 μm/500 nm SiO₂ thermally grown on highly p-doped silicon substrate, using photolithography techniques. First, source and drain were patterned with 5/40 nm chrome/platinum electrodes, with gaps of 1.3/1.8 μm between source and drain. Next, the devices were submerged in 1:6 buffer oxide etch (BOE) for two minutes in order to etch trenches of about 200 nm deep between each electrodes pair. Finally, iron nanoparticles were deposited at specific places on top of the electrodes, and the CNTs were grown by chemical vapor deposition (CVD) with methane/hydrogen gas mixture at 900°C. This technique yields extremely clean devices, since the CNT growth is performed at the last step of the process, and all resist residues are burned and disposed of in the CVD process. In order to create on-surface CNT FETs, suspended devices were submerged in deionized water for a couple of seconds and then allowed to dry in ambient air for several hours.

RESULTS AND DISCUSSION

When suspended CNT FETs were exposed to an ambient environment, hysteresis was observed in the current (I_{ds}) versus gate voltage (V_g) characteristics when V_g was swept between -3 V to 3 V [19]. The hysteresis was advancing in nature, and its width (which is defined as the difference in the threshold gate voltages between the down and the up sweep directions) exhibited a strong dependence on ambient humidity, as was previously reported [13, 20]. The hysteresis width has also exhibited exponential time dependence on the scanning time of a single sweep period with time constant of $\tau = 38 \pm 4.2$ sec (see Fig. 1a). This dependence, which is in agreement

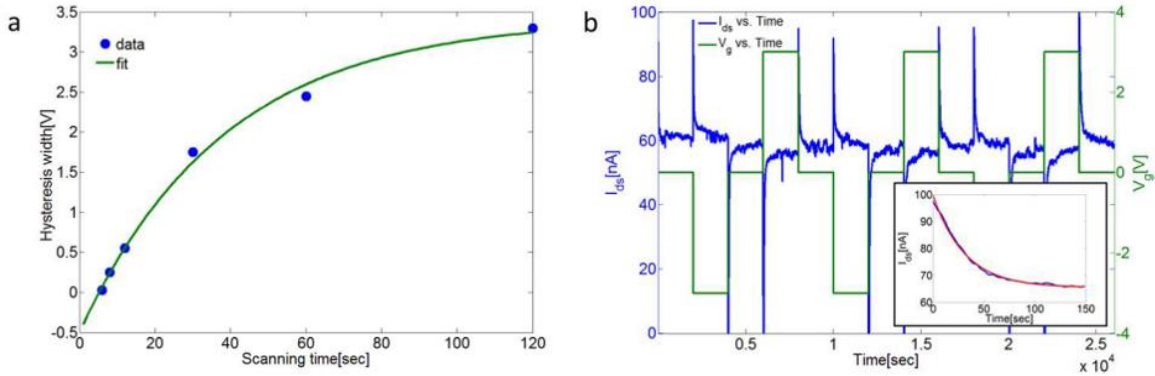


Figure 1. (Color online) (a) The hysteresis width of a suspended CNT exhibiting an exponential dependence on the gate scanning time with time constant $\tau = 38 \pm 4.2$ s. V_g is swept between -3 and 3 V, and $V_{ds} = 10$ mV. Error bars are smaller than the markers. (b) I_{ds} (blue) as a function of time for the same CNT as in (a) while applying a series of gate voltages: $0, -3, 0, 3,$ and 0 V. This series repeats itself three times (green). Inset: zoom-in into single current decay curve, I_{ds} -time, following compliance of $V_g = -3$ V and exponential fit (red) to it.

with earlier observations of hysteresis in CNT FETs [16], suggests that the hysteresis is caused by slow moving species carrying charge. Based on this evidence, we implemented the usage of a more accurate measurement aimed to extract the time constants of the hysteresis effect. As shown in Fig. 1b, we measured I_{ds} as a function of time while applying a series of gate voltages: 0 V -3 V 0 V 3 V 0 V. This series of values repeated itself several times. Two main findings were observed: a) the data can be divided to two pairs of gate voltages, $V_g = 0$ V, 3 V ($V_g = -3$ V, 0 V), on which the current shows a rapid decrease (increase) followed by a slow recovery (decay); b) after a long time on which the gate voltage was kept constant the currents for all the gate voltages tended to the same value. Moreover, for each value of V_g that was kept constant for a fixed period of time, I_{ds} had converged back to its initial value exponentially. The time constants of I_{ds} for each value of V_g were extracted by fitting the data to an exponential function (see inset of Fig. 1b), and an average time constant of $\tau_{av} = 35.3 \pm 8.5$ sec was found. This τ_{av} agrees reasonably well with the time constant which was extracted from Fig. 1a, with a relative error of 8.4%. This result suggests that sweeping the gate voltage with different speeds, or measuring the current versus time for a fixed gate voltage probe the same hysteresis phenomenon, and we will utilize both methods in order to clarify the origin for it.

In order to find whether suspended and on-surface CNT FETs exhibit similar hysteretic and time constants behavior, we examined the hysteresis phenomena of the same suspended and on-surface CNT FET. From I_{ds} -time measurements an average $\tau_{av} = 19.5 \pm 6.3$ sec was found for a typical suspended device according to the method discussed before. Afterwards, the same device was submerged in deionized water in order to make the CNT adhere to the surface (as was verified by AFM), was dried for several hours, and the hysteresis was examined again. This time an average $\tau_{av} = 19.07 \pm 5.3$ sec was deduced. The I_{ds} reduced roughly by third after the CNT touched the surface, and the device became somewhat noisier; however, the hysteretic characteristics did not change and the time constants remained nearly the same, with a relative error of 2.2%. The same behavior was found for other 5 tubes as well. These results suggest that the same mechanism underlies the hysteresis phenomenon in both types of CNT FETs for small V_g sweep ranges.

Next, we conducted a series of control experiments in order to unveil the mechanism that is responsible for the hysteresis in these CNTs [19]. Exposing the CNTs to different atmospheres may hint which ingredients in air are essential for the hysteresis phenomenon. The hysteresis disappeared almost entirely upon changing the environment from ambient air to vacuum. Also, the current reduced significantly, probably due to the removal of oxygen which is known to modulate the Schottky barriers and alter the p-channel current [21]. We found that the hysteresis did not reappear upon exposure to oxygen, nor upon exposure to nitrogen. However, when water vapors were introduced into the cell, the hysteresis gradually increased once again. This observation is consistent with previous findings, which have reported that humidity affects hysteresis significantly [12, 13, 20].

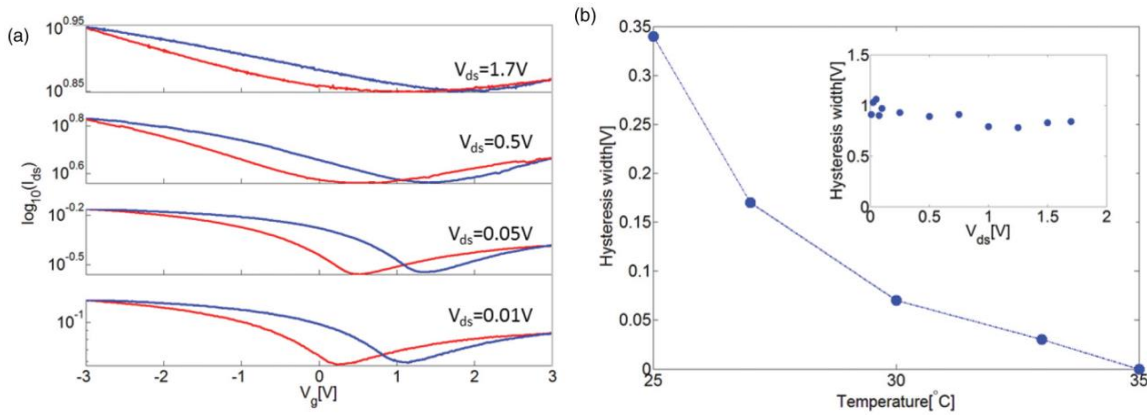


Figure 2. (Color online) (a) Semi-log plots of I_{ds} - V_g of a suspended, metallic CNT for several values of V_{ds} . In all of these curves, the scan rate was 200 mV/s. (b) Hysteresis width for the same CNT as in (a) as a function of the hot plate temperature. $V_{ds} = 10$ mV, and V_g is swept between -3 and 3 V with a scan rate of 200 mV/s. Inset: hysteresis width for the same CNT as in (a) as a function of V_{ds} . Error bars are smaller than the markers.

The same hysteretic behavior and atmospheric dependence was found for small band gap CNTs as well. These are metallic CNTs with a small band gap that is usually formed as a consequence of tube deformation [22, 23]. One can utilize these metallic tubes in order to find the location of the water layer on the device. For that purpose, we continuously measured I_{ds} versus V_g for a suspended, metallic CNT FET (see Fig. 2a), while increasing the bias voltage V_{ds} gradually at the end of each sweep. The width of the hysteresis for each sweep is shown in the inset of Fig. 2b as a function of V_{ds} . Previously, it has been shown that under high source/drain biases suspended metallic CNTs are heated to several hundred degrees centigrade, since the heat dissipates mainly through the metallic contacts and the surrounding air which is a poor thermal conductor [24]. A signature for such heat-enhanced phonon scattering is typically observed in the negative differential conductance at high V_{ds} , as found for our device as well. At such elevated temperatures any water layer that might be adhered to the tube surface would probably be evaporated and disappeared. Nonetheless, the hysteresis did not decrease and remained nearly constant throughout the experiment (inset of Fig. 2b). However, when the same CNT FET was placed on a hot plate and its I_{ds} versus V_g characteristics were continuously recorded while the hot plate temperature gradually increased (V_g was swept between -3 V and 3 V, and V_{ds} was kept constant at 10 mV), the results were completely different. Fig. 2b shows the width of the hysteresis that was obtained in each sweep as a function of the hot plate temperature. In this case, the hysteresis is rapidly reduced and reaches near zero at about 35° C. This process was carried out on more than 10

suspended metallic and semiconducting CNTs and all showed the same hysteresis behavior versus source-drain bias, V_{ds} , and hot plate temperature.

These results suggest that as opposed to common belief, the hysteresis is caused by water molecules that adhere to the oxide and not to the CNT itself. However, since our experiments were conducted using CNTs which are suspended relatively far above the SiO_2 surface on which the water molecules are located, charge transfer between the water layer and the CNT becomes impossible. Thus, the water layer cannot function as charge trap or mediator for charge transfer between the CNT and the oxide as was previously believed [12-14]. Furthermore, because on-surface CNTs exhibit similar hysteretic behavior with roughly the same time constants as suspended CNTs for low operational gate voltages ($|V_g| < 3\text{V}$), we argue that for these conditions charge injection is not the dominant cause for hysteresis of on-surface CNTs as well. We propose that water assisted mobility of surface charge on the SiO_2 surface is the underlying mechanism for the hysteresis phenomenon observed in suspended CNTs for all V_g regimes (up to $\pm 10\text{ V}$) and for on-surface CNTs for low gate voltages (up to $\pm 3\text{ V}$). The screening process can be described as follows: Applying non zero gate bias results with lateral electric field lines in the vicinity of metallic electrodes. Under these lateral electric fields, opposite signed mobile charge drifts across the SiO_2 surface and gradually screens the gate. This movement of the charges is facilitated by the water layer which exists on the sample surface at ambient conditions, and results in a lower energetic configuration of the electric field lines in the system. After some time the external gate is screened and the I_{ds} converges to its initial value, equal to the value obtained at $V_g = 0\text{ V}$. On the other hand, when the device is under deep vacuum, the absence of water layer prevents the charge from drifting across the SiO_2 surface, and the I_{ds} remains constant and does not change with time [19, 25].

A simplified model for the temporal charging and de-charging phenomena can be described as follows: a thin slab on top of the sample surface between the two electrodes behaves as two leaky capacitors. For each capacitor one plate is near one of the electrode, and the other plate is in the middle between the two electrodes. If $\rho(t)$ is the charge density per unit area on one of the plates, then using continuity, Ohm, and Gauss equations one can write the following expression

$$\frac{d\rho(t)}{dt} = -J = -\sigma E_x = -\sigma \frac{\rho}{\epsilon_0 \epsilon_{ox}}, \quad (1)$$

where J , σ and E_x are the current density, conductivity and lateral component of the electric field in the slab, respectively. The solution for this equation has an exponential behavior which is consistent with the experimental results exhibited so far, with time constant, $\tau = \epsilon_0 \epsilon_{ox} / \sigma$. A more accurate model was performed by finite element analysis software (COMSOL Multiphysics 4.2a). The obtained results confirm our model very well. Fitting the simulated data to the experimental one (performed between the two electrodes with 65% humidity), yields the conductivity of the SiO_2 , $\sigma = 4.1 \cdot 10^{-12} \pm 3.5 \cdot 10^{-13} (\text{S/m})$ with σ as a single fitting parameter for the exponential time dependence. The reported range of SiO_2 conductivity $\sigma = 10^{-12} - 10^{-16} (\text{S/m})$ is in good agreement with our result. We suggest that the relatively improved conductivity in our system is caused by the water layer which exists on the surface of SiO_2 in ambient conditions.

In order to examine this model, we used a technique [25] which employs the use of EFM to gain quantitative information on the substrate charge distribution. This method overcomes the difficulty of having accurate estimation of the tip-charge mutual capacitance. Using this technique, we measured the amount of charge in a close proximity to an on-surface CNT as a function of time, while measuring the CNT's current under the following gate voltage compliances: 0 V -2 V

0 V 2 V 0V. The results are shown in Fig. 3 [19]. The surface charge density declines/inclines in an exponential decay/recovery manner which is very similar to that of I_{ds} , with equivalent time constants for all V_g regimes (maximum relative error 8.5%). Moreover, the amount of total charge that moves on the substrate during the measurement is roughly the same for all gate voltages ($4.2 \cdot 10^{-9} \pm 0.2 \cdot 10^{-9} \text{ C/cm}^2$), and its sign in each regime matches the expected one. Far from the tube ($\sim 20 \mu\text{m}$), similar behavior is observed with similar time constants which confirms that charge transport on the oxide surface has nothing to do with the leakage current to or from the tube itself.

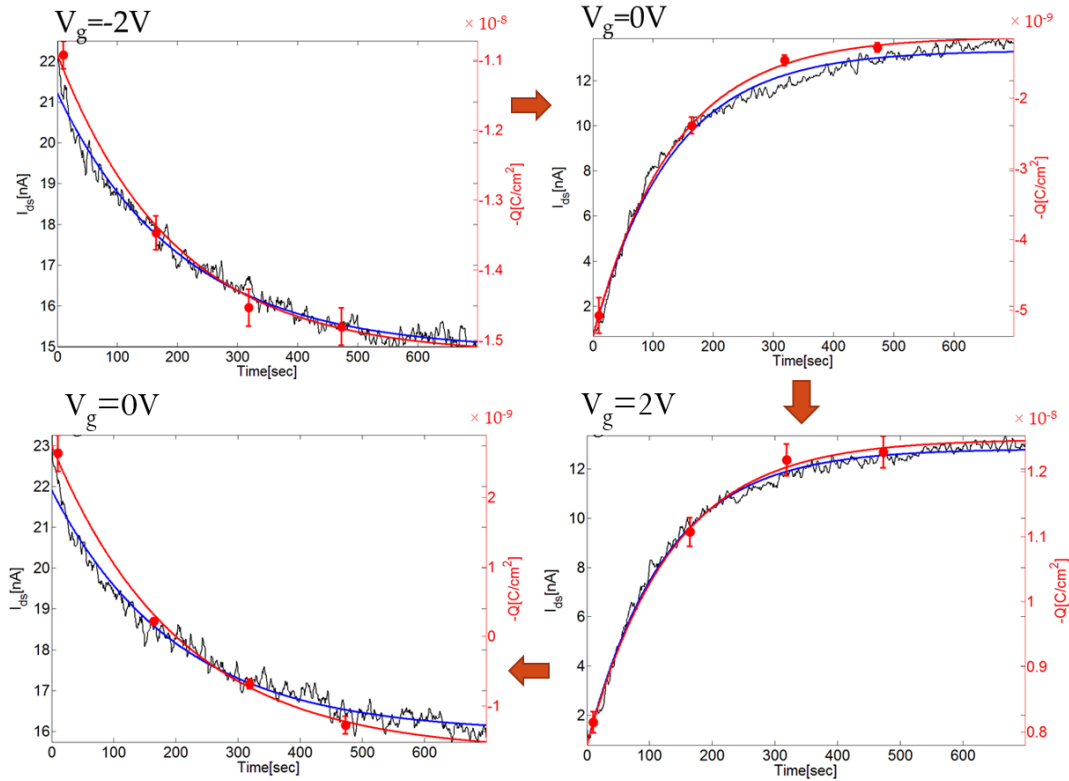


Figure 3. (Color online) EFM-based measurements of the temporal charge distribution on the SiO_2 surface in close proximity to an on-surface CNT (red circles). Red lines are best fit to exponential behavior. Simultaneously, I_{ds} vs time was measured (black lines) with a bias voltage of 10 mV. Blue lines are best fit to exponential time dependence. The measurements were conducted for four values of V_g , initiate at $V_g = -2 \text{ V}$ and continue clockwise.

The applied pulses were either 0 V or 2 V, and for both values the time constants were similar. One may ask does the time constant, which depends on surface conductivity, depend on gate voltage. The experiment was performed for a series of gate voltages, which were each applied at $t=0 \text{ sec}$: -0.4 V, -0.6 V, -0.8 V, -1 V, -2 V, -3 V [26]. Before applying each voltage, the system was given a sufficient recovery time, after which the surface charge density was verified to be zero. In order to avoid changes in ambient humidity, all of the measurements were performed on the same day. The obtained results are summarized in Fig. 4a. One can see that while τ does not depend on V_g for high gate voltages, a strong dependence is observed for low gate voltages ($V_g < 1 \text{ V}$). Since τ and the surface conductivity are inversely proportional [25], this observation implies that (a) σ is indeed dependent on V_g for the low voltages regime, and (b) this dependence is non-linear. The same behavior was observed from the time constants of the

exponential fits to the CNT's current decay for each value of V_g , as summarized in Fig. 4b. The inset of Fig. 4b depicts the normalized $\tau(V_g)/\tau(V_g = -0.4 \text{ V})$ for the two sets of measurements (EFM-based in blue and CNT based in red), and one can see that the obtained patterns are very similar. The same behavior was found for other 4 tubes as well, supporting our conclusion regarding the dependence of the surface conductivity on V_g .

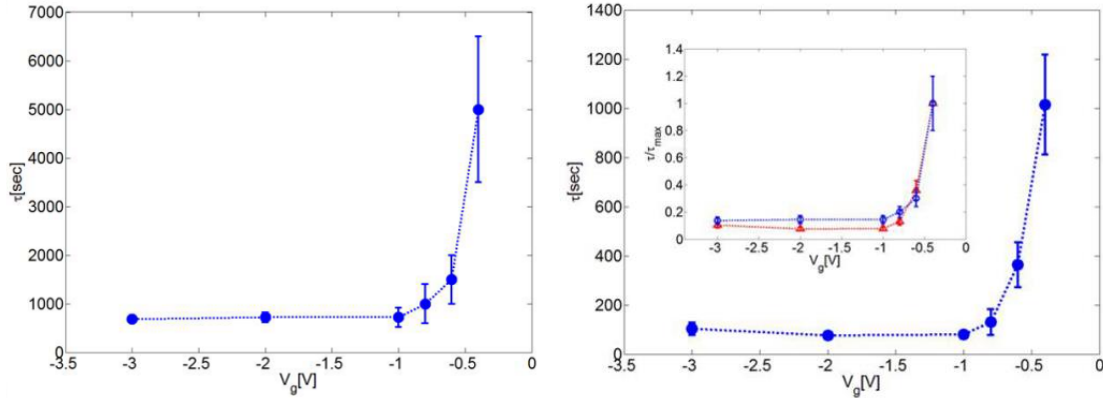


Figure 4. (Color online) (a) The time constants of the charge redistribution process ($Q_s(t)$) in between two electrodes as a function of V_g , as probed by EFM technique. The gap between the electrodes is 10 μm . (b) The time constants of CNT's current decay as a function of V_g . The CNT is suspended over a 1.8 μm gap. The experiments were conducted at 55 % humidity. Inset: normalized $\tau(V_g)/\tau(V_g = -0.4 \text{ V})$ vs. gate voltage for EFM-based data (blue) and CNT-based data (red).

Since humidity is essential for surface charge redistribution, and hence for the hysteresis phenomena, we have studied the dependence of σ on humidity. For that purpose, we have used CNTs-based measurements, and monitored the CNT's current as a function of time upon applying $V_g = -0.8 \text{ V}$ at $t=0 \text{ sec}$ and keeping the voltage constant. This process was repeated several times, and under different ambient humidity (%h) conditions. The normalized time constants ($\tau(\%h)/\tau(\%h = 45 \%)$) of the exponential fits to the CNT's current decay for each humidity percentage are summarized in Fig. 5. As expected, τ exhibits a strong dependence on ambient humidity, and the saturation range is around 60% humidity and above. The same behavior was observed for other CNTs as well.

In light of this, we propose the following hypothesis: the amount of water molecules on the SiO_2 surface, which usually depends on ambient humidity alone (for a given temperature), is increased upon applying V_g , and as a result, the surface conductivity is increased as well. One possible mechanism through which water molecules from the surrounding environment may be attracted and adsorbed onto the dielectric surface is due to their net dipole moment. Since oxygen is an electronegative element, it attracts electrons much strongly than hydrogen, resulting in a net dipole moment of $p = 6.2 \cdot 10^{-30} \text{ C}\cdot\text{m}$ for each water molecule. When such a polarized object is found in a nonuniform electric field (such as the one induced upon applying voltage to the gate at $t=0 \text{ sec}$), a force which is known as dielectrophoresis, or DEP, acts upon it. This DEP force can change the partial pressure of water molecules on the SiO_2 relative to their partial pressure high above the surface, and as a result to modify the relaxation time of the hysteresis phenomena. In Ref. [26] we developed a theoretical model based on this assumption, and examine the agreement between the model and the experimental results. It turns out that the experimental data and the theoretical model support our hypothesis, according to which external gate biases modify the surface humidity, which can be very different from the average humidity in the lab.

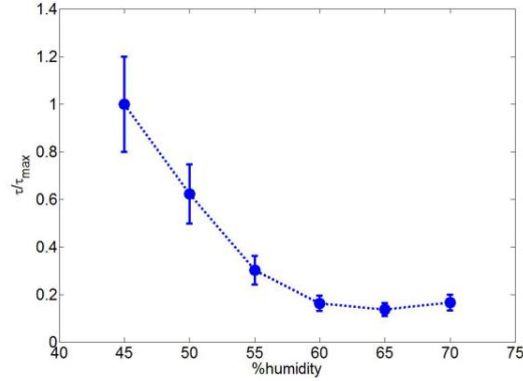


Figure 5. (Color online) Normalized time constants, $\tau(\%h)/\tau(\%h=45\%)$, of the CNT's current decay upon applying $V_g = -0.8$ V at $t=0$ sec as a function of humidity.

Moreover, these results imply that surface conductivity depends on the number of water layers adsorbed on it up to approximately 3 layers. Above it, the conductance saturates and the dependence becomes much weaker if any [26].

CONCLUSIONS

In conclusion, in the current study we have conducted a thorough investigation of the origin of hysteresis of suspended and on-surface CNTs. Using I_{ds} -time measurements in addition to the prevalent I_{ds} versus V_g characteristics measurements, we were able to extract the accurate time constants of the hysteresis phenomenon. To find out which component of the ambient environment is responsible for the effect, we conducted a series of control experiments which confirmed that humidity is the major cause of hysteresis. However, we found that the location of the water layer is on the SiO_2 surface rather than on the CNT itself. On the basis of these results, it was concluded that water-assisted mobility of charge on the SiO_2 surface is the underlying mechanism for the hysteresis phenomenon in suspended CNTs. By comparing the time constants of suspended CNTs with the time constants of on-surface CNTs in which the CNTs were adhered to the surface, we proved that the above-mentioned mechanism is also responsible for the hysteresis of on-surface CNTs operating at low gate voltages ($|V_g| < 3\text{V}$). At higher gate values, additional mechanisms start to contribute to the hysteresis phenomenon of on-surface CNTs, and are the subject of further studies. A model for the hysteresis involving surface charge redistribution aimed to screen the applied gate voltage was proposed and supported by quantitative temporal EFM charge distribution measurements. This model explains not only the temporal dependence of the screening phenomenon, but also the final steady-state value of the current for any gate voltage which is found regularly for these devices.

In addition, it was shown that the gate voltages affect the time constant for the hysteresis phenomena, as humidity does. It was suggested that the applied field, which creates gradient in the electric field, modify the amount of water layer on the surface, and as a results alter the time dependence of the hysteresis. Based on microscopic model, it was argued that the charge redistribution on the substrate is sensitive to three layers of water or less, which paves the way to ultra-sensitive humidity sensors, and electro-chemical switches.

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