Electronic fluctuations in multi-walled carbon nanotubes

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Abstract. We report on the low-frequency electronic noise properties of individual multi-walled carbon nanotubes (CNTs). We present experimental evidence of the key role played by the structural quality of the tube and its gaseous environment on the excess noise level. By tuning the Fermi energy location in the electronic band structure, we also demonstrate the energy dependence of the electronic fluctuations in CNTs. Large random telegraph noise can be activated under specific Fermi energy locations. The role of mobile atomic-sized defects in a coherent transport regime is discussed.
1. Introduction

The physics and application of carbon nanotubes (CNTs) in nano-electronics are currently under intense studies [1, 2]. Most of the striking characteristics of the electronic transport in CNTs have been expressed using the time average of the conductance [3]. However, high-order statistic conductance fluctuations in nano-scale conductors are a powerful experimental tool to unveil the exceptional richness of charge carriers properties in a confined environment. In one-dimensional wire, the discreteness nature of the electrical charge is responsible for the probabilistic current fluctuations (shot noise). Its factor reduction, compare to the Schottky value ($2I|e|$) expected for the low-transmission limit, gives an insight into the strength of the electron–electron interactions [4]. In the diffusive regime, the quantum noise is usually overcome by the presence of active fluctuators coupled to charge carriers. At low temperature, single events like atomic fluctuations of one scatter between two sites or the capture of one electron on an active trap may induce remarkable random resistance switches between two discrete values [5, 6]. The statistical analysis of the so-called random telegraph noise (RTN) versus external perturbations (temperature, electric or magnetic field . . . ) yields detailed information on the electronic scattering processes and their energy dependence [7].

The issue of the electronic noise experiments on CNTs has to be addressed in the very specific context of their unique electronic transport properties. Firstly, it is now well established that the conductivity of individual CNTs exhibits a large sensitivity to gas adsorbate [8, 9], substrate [10] and contact effects [11] due to charge transfers and work function mismatch at the metal/tube interface. The electronic fluctuations measurements, sensitive to localized fluctuating entities strongly coupled to charge carriers, may reveal the microscopic mechanisms responsible for the chemical sensitivity of the CNTs. Secondly, recent works stress the strong energy dependence of the conductivity in CNTs [12, 13]. While, in pristine tubes, the conductivity is proportional to the number of conduction channels, the situation gains in complexity for disordered tubes: both the electronic mean-free path ($l_e$) [14] and the phase coherence length ($l_{\phi}$) [12], are strongly reduced in the vicinity of the van Hove singularities (vHs) and near the energy levels of scatters. The unsolved question of the excess noise level and its physical origins in CNTs requires noise measurements as a function of the Fermi energy location to probe the interplay between the
electronic fluctuations and the band structure. Up to now, the very few studies on individual CNTs display a low-frequency $1/f$-noise whose magnitude ranges over four decades [15]–[20]. The contribution of the tube alone to this noise and its structural quality dependence are open questions.

In this paper, we present the first evidence that gas, mainly oxygen surrounding the tube, significantly contributes to the electronic fluctuations at room temperature. Under controlled atmosphere, the excess noise level and its temperature dependence are also conditional on the conduction regimes of the tubes. We demonstrate that quasi-ballistic multi-walled CNTs (MWCNTs) are low-noise conductors at room temperature. The $1/f$-noise behaves like a background noise independent on the number of conduction channels. In case of diffusive MWCNTs, we infer a distribution of the activation energies responsible for the intrinsic CNT noise. From multi-probe measurements, an estimate of the localized contact noise is proposed. Finally, we give evidence that low-noise MWCNTs at room temperature may exhibit huge RTN at low temperature by tuning the Fermi energy location. From the spectroscopies of the RTN, we sketch the possible contribution of impurity scattering fluctuations enhanced by quantum interferences.

2. Experimental procedure and device characterization

Experiments have been performed on both arc-discharge (Arc) and chemical vapour deposition (CVD) produced MWCNTs with diameters ranging from 3 to 15 nm. Tubes are deposited on Si/SiO$_2$ (100 nm) substrates, using the Si as a back-gate and individually connected with palladium electrodes. Low-frequency electronic noise measurements versus temperature, bias voltage and back-gate are based on the standard dc technics. The noise behaviours we present are representative of general tendencies we observed on MWCNTs. For clarity, we mainly depict the electronic fluctuations obtained on quasi-ballistic Arc-MWCNTs. Comparisons are made with the resistance fluctuations we measured on more diffusive CVD-MWCNTs.

In the inset of figure 1(a) is shown the AFM picture of a 15 nm diameter Arc-MWCNT with Pd electrodes spaced by 250 nm. Its electronic transport properties are now well-investigated [12, 21] and representative of high-quality Arc-MWCNTs. Room temperature conductances at two and four probes are $1.1G_0$ and $1.5G_0$ respectively. After cooling down to 10 K under vacuum ($P = 10^{-6}$ torr), the differential conductance versus back-gate voltage exhibits strong modulations between 0.3 and 0.6$G_0$ (figure 1(a)) with linear IV curves (not shown here). In the light of recent transport experiments on similar MWCNTs [12], the well-defined maxima of conductance at −3 and 6.5 V are likely due to the first vHs (dotted lines, figure 1(a)). Their locations versus $V_g$ are consistent with the 160 meV sub-band energy spacing considering the electrostatic coupling of our device$^4$. The smaller modulations between the vHs peaks, around $0.7 \times 10^{-5}$ S, are attributed to universal fluctuation conductance (UCF) in the mesoscopic regime with $l_\psi$ equal to 50 nm. Complementary magneto-transport experiments (details are published elsewhere [21]) give evidence of the co-existence of both quantum interference effects in the weak-localization regime (Altshuler–Aronov–Spivak effect) along with a strong magnetic

$^4$ The electrostatic coupling between the tube and the back-gate is defined by $\Delta E_F \approx e \times C_{NT} l_\psi$ with $C_\psi$, the geometrical back-gate capacitance and $C_{NT}$, the electrochemical capacitance of tube expressed by $C_{NT} = e^2 \cdot DoS(E_F)$. We infer a gate coupling around 16 meV V$^{-1}$ in the metallic bands. The locations of the $dI/dV$ peaks at −3 and 6.5 V correspond to an energy separation of 152 meV. This is favourably compared to the calculated sub-band spacing equal to $2 \gamma_0 \sin(\frac{\pi}{110}) \approx 160$ meV for a (110, 110) CNT with a 15 nm diameter.
Figure 1. (a) Differential conductance versus the back-gate voltage obtained on a 15 nm diameter Arc-MWCNT (AFM picture in inset) at 10 K. The dot lines indicate the first vHs locations and the grey one indicate the CNP (see footnote 4). (b) The normalized power spectral density of the noise $S_{v}/v^2$ versus frequency at 300 K, in ambient air (open circle) and under a $10^{-6}$ torr vacuum. In inset, the normalized 1/f-noise versus the conductance (in units of $G_0$, with $G_0$ the quantum of conductance equal to $2e^2/h$) extracted from different studies: circle (present paper), diamond [15], star [18] and square [20].

field modulation of the DoS (Aharonov–Bohm effect) with $l_\phi$ larger than the circumference ($l_\phi \simeq 60$ nm at 10 K in the metallic bands, in agreement with the UCF magnitude). We conclude on a quasi-ballistic regime with well-defined one-dimensional DoS effects on the electrical transport and some native defects responsible for low-temperature quantum interference.

3. 1/f-noise in MWCNTs

3.1. Gas exposure effects

Firstly, we deal with the excess noise on MWCNTs at room temperature and its sensitivity to the gaseous environment. All samples we measure exhibit standard Gaussian noise proportional
to the square of the bias voltage with a $1/f^{\alpha=1}$ frequency dependence. The magnitude of the power spectral density of the $1/f$-noise is described by the Hooge formula [22], $S_v = \gamma V^2 / \Omega f$, where $\gamma$ is the Hooge parameter, $V$ the bias voltage and $\Omega$, the number of charge carriers in the noisy volume. To avoid any assumption on the number of charge carriers and the physical location of the noise sources, we first deal with the ratio $\gamma / \Omega$. For MWCNTs with room temperature conductance in the range of $G_0$, like the one in figure 1(a), the $1/f$-noise level presents an astonishing dependence on the gaseous environment around the tube (figure 1(b)).

After a 2 h pumping at 300 K to reach a vacuum around $10^{-6}$ torr within the sample chamber, we observe a large decrease by a factor of 7 of the $1/f$-noise level along with a 5% decrease in the conductance. This first evidence of gas effect on the electronic fluctuations also reveals an unconventional behaviour: while the normalized noise level usually scales with the resistance (an increase in scattering means an increase in the number of fluctuators), here, the situation is opposite. A gain of resistance comes along with a less noisy conductor. It is well established that active molecules on CNTs present in ambient air are O$_2$ [8, 9] and H$_2$O [23]. Even if the microscopic mechanisms are still under debate [24], these molecules are thought to be directly physisorbed on pristine tubes, chemisorbed on topological defects on the tube [25] and, finally, adsorbed at the contacts [26]. It induces an increase in the density of states at $E_F$ [25, 27] or an increase in the metal-work function [28]. In both cases, it results to a slight increase in the conductance. The slightness of the effect comes from the metallic nature of the tube. In the same time, the large enhancement of the electronic fluctuations likely reveals the effects on charge carriers of the adsorbate molecules fluctuations between different energetically equivalent sites.

3.2. $1/f$-noise level at 300 K

Interestingly, under controlled atmosphere (a vacuum of $10^{-6}$ torr), the normalized $1/f$-noise and its temperature variation seem structural quality dependent of the tube. We measure different $\gamma / \Omega$ noise levels ranging from $3 \times 10^{-7}$ for quasi-ballistic MWCNTs with $G(300 \text{ K}) \simeq 1.2G_0$ to $10^{-5}$ for diffusive CNTs with $G(300 \text{ K}) \simeq 0.2G_0$. Postma et al [18] obtained $\gamma / \Omega \simeq 4 \times 10^{-5}$ for SWCNTs with $G(300 \text{ K}) \simeq 0.43G_0$; Collins et al [15] measured $\gamma / \Omega \simeq 2 \times 10^{-5}$ for CNTs with $G(300 \text{ K}) \simeq 0.065G_0$ and Roche et al [20] achieved an extremely low $1/f$-noise on suspended and soldered ballistic SWCNTs, $\gamma / \Omega \simeq 10^{-9}$ (two orders of magnitude smaller than our best results obtained on quasi-ballistic MWCNTs). For clarity, these values are summarized in the
inset of figure 1(b). Comments are certainly not straightforward since results on SWCNTs and MWCNTs experiments with different lengths, from 250 nm to 1 µm, are gathered. Nevertheless, the general tendency is a gradual 1/f-noise level decrease for CNTs approaching the expected $2G_0$ value for pristine devices. This dependence, which both involves the structural quality of the tubes and the contact transparency, certainly contributes to the dispersion of the CNTs noise levels in the literature. To compare with other materials, we normalize the excess noise by the number of mobile carriers \[\frac{\gamma}{\Omega} = \frac{4L}{\pi\gamma_0} \sqrt{2m(E_F - E_c)}\] where $E_c$ is the minimum of the conduction band. Assuming $E_F$ close to the CNP, \((E_F - E_c) \approx \gamma_0\), $\gamma_0$ being the energy overlap integral between carbon atoms of the order of 2.9 eV. We finally find a number of electrons per unit of length in the metallic bands of the order of $11 \text{ nm}^{-1}$. We infer a Hooge parameter for quasi-ballistic MWCNTs: $\gamma \approx 9 \times 10^{-4}$. This value is favourably compared to the empirical Hooge constant ($\gamma_H \approx 2 \times 10^{-3}$) usually found for metallic and semi-conductor bulk materials [30]. So, we point that CNTs, despite their large surface sensitivity, behave like a low-noise conductor once its purity and the experimental environment are under control. This is of practical interest for CNT potentiality in nano-circuitry.

3.3. Contacts contribution

The contact contribution to the excess noise is clarified by combining two-, three- and four-probe noise experiments. Three Pd-probes were available on a diffusive CVD-MWCNT (AFM picture in the inset figure 2, $G(300 \text{ K}) \approx 0.2G_0$ for a 3 nm diameter with 400 nm between Pd-electrodes). Two-probe measurement on the external electrodes (1–3) gives $\gamma/\Omega \approx 4 \times 10^{-6}$, while three-probe measurements with the middle electrode as a voltage probe yields to $7 \times 10^{-6}$ for the upper (1–2) and the lower part (2–3) of the tube, respectively (figure 2).

Uncorrelated noise sources induce additive fluctuations. So, in a first approach, considering invasive Pd-contacts, we define: $S_v/V^2|_{2\text{probes}} = 4\gamma_c + \gamma/\Omega$ and $S_v/V^2|_{3\text{probes}} = 2\gamma_c + \gamma/\Omega/2$, where $\gamma_c$ is the noise source localized at the contacts. We infer a localized contact noise $\gamma_c$ around $1.5 \times 10^{-7}$, at least one order of magnitude smaller than the two-probe noise level. So, in the diffusive regime, the noise we measure with two Pd-probes is of intrinsic origin and roughly scales with the tube’s length. On the other hand, for the Arc-MWCNTs depicted in figure 1(a), our preliminary attempts to estimate the four-probe noise strongly suggest that voltage fluctuations across the inner electrodes are much smaller than those measured in two probes configuration\(^5\). So, for high-purity CNTs, the two-probe electronic fluctuations, of the order of $3 \times 10^{-7}$ under vacuum (figure 1(b)), mostly unveil an excess noise of extrinsic origin, presumably localized at the CNT–electrode interfaces. Further experiments are needed to check if the contact noise in the range of few $10^{-7}$ is a common feature of Pd/MWCNT interface.

3.4. Temperature dependence of the 1/f-noise

Very instructive is the temperature dependence of the 1/f-noise level. In figure 3(a) are plotted the $\frac{\gamma}{\Omega}(T)$ curves obtained on CNTs with different conduction regimes. For quasi-ballistic MWCNTs, with a very little temperature dependence of the conductance, the 1/f-excess noise is almost

\(^5\) No quantitative data are currently available since a damage of the tube occurs when large currents were applied to unveil the excess noise above the experimental background noise.
Figure 2. Two- and three-probe measurements of the excess noise at 300 K obtained on a diffusive MWCNT of 3 nm diameter and 400 nm between Pd-electrodes (in the upper-inset, the AFM picture). In lower-inset, the temperature dependence of the resistance of this MWCNT below 100 K.

Temperature independent (filled squares). This supports the scenario of a predominant contact noise of $3 \times 10^{-7}$ which remains constant versus temperature. A strong contrast is observed with the temperature dependence of the fluctuations we measure on diffusive MWCNTs dominated by the weak-localization regime at low temperature (inset, figure 2). The excess noise is severely decreased from $7 \times 10^{-6}$ at 300 K to $10^{-7}$ at 4 K (filled circles, figure 3(a)). These features confirm the temperature tendencies already mentioned separately for ballistic [20] and diffusive [18] CNTs (open squares and open circles, respectively in figure 3(a)). The large $1/f$-noise decrease versus temperature is analysed in a standard way for diffusive conductors. For thermally activated fluctuations, an almost linear variation of $S_{v}(T)$ comes from an increase of the number of active fluctuators versus $kT$ in the experimental energy window. An unique distribution of activation energies $D(E)$ both accounts for the frequency dependence $\alpha(T)$ of the noise and its magnitude $S_{v}(T)$. Correlated departures from $\alpha(T) = 1$ and $S_{v}(T) \propto kT$ are signatures of bumps or depletions in the $D(E)$ shape. Following the Dutta et al model [31], we infer a very broad energy distribution with a bump around 0.4 eV and a surprising distribution increase below 50 meV (figure 3(b)). The validity of this approach is supported by the concordance between the experimental $\alpha(T)$ values and the ones deduced from the $S_{v}(T)$ dependence [31] (inset figure 3(b)). As we experience that the large temperature variation of $S_{v}(T)$ appears along with the diffusive regime, the energy distribution $D(E)$ responsible for the excess noise has to be somehow related to the presence of atomic defects in the tube. In metals, the broad energy
Figure 3. (a) Temperature dependence of the normalized $1/f$-noise for a quasi-ballistic Arc-MWCNT (■), a diffusive CVD-MWCNT (●), a diffusive SWCNT [18] (○) and a ballistic, suspended and soldered SWCNT [20] (□). (b) Distribution of the activation energies responsible for the $1/f$-noise in a diffusive MWCNT. In the inset, comparison between the experimental $\alpha(T)$ values (●) and the calculated ones (○) following the Dutta et al model [31].

distribution centred on 1 eV refers to the activation energies of atomic motions between equivalent sites but with different scattering cross-sections [31, 32]. For covalent C–C bonds, the creation of atomic defects like vacancies or pentagon–heptagon defects and their motion require much higher energies, around a few eV [33, 34], incompatible with the energy range we infer. However, these defects are known to be privileged sites for atomic adsorption with cohesion energies inferior to 1 eV. We may expect that thermally activated fluctuations of adsorbate atoms on defects contribute to the $1/f$-noise in the diffusive regime. Extended experiments in the lower temperature regime are required to investigate the contribution of the low-activation energies.
Figure 4. (a) RTN active around \( V_g = -3 \) V versus the back-gate voltage measured on the Arc-MWCNT depicted in figure 1(a). (b) The corresponding occupation times for the high (●) and low (○) resistive states. The dashed lines are a linear fit (see text).

4. Electronic fluctuations versus energy

The energy dependence of the electronic fluctuations in MWCNTs is probed at 10 K by varying the back-gate voltage. For Arc-MWCNT presented in figure 1(a), we first observe that the 1/f-noise remains remarkably constant as a function of the Fermi level location (not shown here). The shift of \( E_F \) from the metallic to the diffusive bands (with an entering into the first vHzs at \( V_g = -3 \) V) does not significantly affect the 1/f-noise: it behaves like a background noise of the device. Once again, this is consistent with a Gaussian excess noise of extrinsic origin. However, superimposed to this Gaussian noise, large non-Gaussian (NG) fluctuations can be activated under specific values of \( V_g \). By varying \( V_g \) from 0 to \(-5\) V, the noise successively alternates from Gaussian noise (of 1/f-type) of constant magnitude to RTN with a large timescale distribution ranging from ms to s. Active RTN is apparently not specific to the presence of the first vHzs. In figure 4(a), we present the gate dependence of a well-defined two-level fluctuation active around \(-3.1 \pm 0.1\) V. The plot of the occupation times \( \tau_{\text{up}} \) and \( \tau_{\text{down}} \) for the high and low resistive states shows a striking dependence on \( V_g \) (figure 4(b)). Over a narrow range of \( V_g \) (\(-0.2\) V), the lifetimes vary by more than one order of magnitude: the low resistive state is stabilized to the detriment of the high resistive state. In the same time, the magnitude of the resistance switch \( \Delta R/R \) continuously increases from 1 to 5%. This is a very unusual dependence for RTN. We mention that this behaviour is reversible and special care has been taken to ensure that we track a very unique fluctuator. The logarithmic plot of the occupation times versus \( V_g \) shows a linear dependence. In the frame of a double-wells model, \( \tau_{\text{up}} \) and \( \tau_{\text{down}} \) can be simply expressed by:

\[
\tau_{\text{up(down)}} = \tau_0 \exp \left[ E_{\text{up(down)}}(V_g)/kT \right] \quad \text{with} \quad E_{\text{up(down)}}(V_g) = E_{0,\text{up(down)}} \pm \xi \times V_g.
\]

Here, \( \tau_0^{-1} \) is the attempt frequency, \( E_{0,\text{up(down)}} \), the activation energy for the high (low) resistive state at \( V_g = 0 \) V.
and $\xi$ expresses the gate voltage dependence of the activation energies. An identical $\xi$ value is obtained for $E_{up(down)}$, of the order of 10 meV V$^{-1}$. At $V_g = -3.02$ V corresponding to the equiprobability between the two states, we infer an activation energy of 30 meV.

The sensitivity of the two-level fluctuator to the back-gate voltage reveals either an energy dependence of the coupling between charge carriers and the fluctuator or the existence of a permanent dipolar momentum associated to the fluctuating entity and sensitive to the transversal electrical field. The $\xi$ value is of the order of the calculated coupling between $V_g$ and the Fermi energy shift (see footnote 4), which tends to validate the first scenario. On the other hand, if one converts $\xi$ in terms of an electrical field effect on a dipolar momentum $p$, it yields $p \approx 1.4 \times 10^{-18}$ C·m, an unrealistically large momentum for an unique fluctuating molecule. Furthermore, the magnitude of $\Delta R/R$ is extremely high to be consistent with the picture of a single-electron trapping and de-trapping on a site in the vicinity of the conducting channel [6]. In the case of metallic nano-wires in the diffusive regime at low temperature, individual atomic-sized defect motion between sites of distinct scattering cross-sections $\sigma$ is responsible for discrete resistance switches. For a tubular geometry, one expects a $\Delta R/R$ switch in the range of $\Delta\sigma/2\pi C_h$, where $\Delta\sigma$ is the scattering cross-section change and $C_h$, the circumference of the tube. Atomic dimensions for $\Delta\sigma$ ($\sim 1–2$ Å) yield a maximum $\Delta R/R \sim 0.2\%$, more than one order of magnitude smaller than the measured RTN. However, the coupling between a single atomic motion and the resistance changes can be greatly enhanced in the coherent regime ($l_e < l_p$)[35].

The motion of a single-scattering centre drastically modifies the quantum interference pattern of the coherent electronic paths over a distance $l_p$. In one dimension and for one active defect, the conductance fluctuations are expressed by [36]: $|\delta G| \approx \left(\frac{\hbar}{L}\right)^2 \frac{e^2}{h}$. The experimental conductance switches in the range of $0.1–0.410^{-5}$ S imply a phase coherence length of the order of 50–70 nm, in full agreement with the values extracted at 10 K from the UFC on the $dI/dV(V_g)$ curves and the magneto-transport experiments. Therefore, our analysis supports the idea that the large RTN and its energy dependence present at low temperature on quasi-ballistic MWCNTs is due to atomic-sized defect fluctuations which scatter the charge carriers once their corresponding energies match the Fermi level. The magnitude is driven by $l_p$ and the change of the quantum interference pattern for the two atomic configurations of the fluctuating defect. We underline that electrons at 10 K statistically scatter on very few defects along the tube. The electronic mean-free path is found to be larger than 50 nm in the metallic bands [21]. This corresponds to a very reduced number of events. So the large RTN at low temperature cannot give rise to $1/f$-noise at larger temperatures. Besides, the absence of experimental observation of RTN in the high-temperature regime makes sense with the $T^{-1/3}$ temperature dependence of $l_p$ [13]. These comments are consistent with the $1/f$-noise of contact origin (only) we measure at room temperature on quasi-ballistic CNTs, while huge energy dependent RTN can be active at low temperature.

5. Conclusion

In conclusion, we demonstrate that the electronic fluctuations of MWCNTs exhibit a large sensitivity to gaseous molecules. It opens new perspectives on CNTs based molecular sensors operating with the noise measurement. Further works are in progress to see in which respect the electronic noise can be used as a fingerprint of distinct molecules adsorbed on the tube. On the other hand, we present evidence that high-quality MWCNTs, under controlled atmosphere,
are promising low-noise conductors for the nano-connection between active devices. Finally, our low-temperature results suggest that in the mesoscopic regime, huge RTN can be active by tuning the Fermi energy location in the electronic band structure even if the tube is a quiet conductor at room temperature. We strongly suspect the key role played by one mobile atomic-sized defect, whose the strength of the electronic scattering is enhanced by a drastic change of the quantum interference pattern. This first evidence of the energy dependence of the electronic noise in CNTs should stimulate further experimental works.

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