Room-temperature high on/off ratio in suspended graphene nanoribbon field-effect transistors

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Abstract
We have fabricated suspended few-layer (1–3 layers) graphene nanoribbon field-effect transistors from unzipped multi-wall carbon nanotubes. Electrical transport measurements show that current annealing effectively removes the impurities on the suspended graphene nanoribbons, uncovering the intrinsic ambipolar transfer characteristic of graphene. Further increasing the annealing current creates a narrow constriction in the ribbon, leading to the formation of a large bandgap and subsequent high on/off ratio (which can exceed $10^4$). Such fabricated devices are thermally and mechanically stable: repeated thermal cycling has little effect on their electrical properties. This work shows for the first time that ambipolar field-effect characteristics and high on/off ratios at room temperature can be achieved in relatively wide graphene nanoribbons (15–50 nm) by controlled current annealing.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Graphene is a single atomic layer of graphite that exhibits exceptionally high carrier mobility, offering the tantalizing possibility of graphene-based electronics [1]. In the mean time, graphene is a zero-gap semiconductor with finite minimum conductivity, which poses a major problem for mainstream logic applications. One way to circumvent this problem is to slice graphene into nanometer-scale ribbons, where a bandgap can be created by spatial confinement and edge effects [2]. Electron-beam lithography was first used to pattern graphene into nanometer-scale ribbons, where a bandgap can be created by spatial confinement and edge effects [2]. Electron-beam lithography was first used to pattern graphene nanoribbons (GNR) down to a width $\lesssim 20$ nm for field-effect transistor (FET) applications; and a width-dependent transport gap was observed in these devices [3–5]. However, GNRs fabricated by electron-beam lithography and subsequent oxygen plasma etching have relatively rough edges (of the order of a few nanometers) limited by the resolution of electron-beam lithography, which may degrade their electrical properties. Subsequently, several alternative methods have been developed to produce GNRs, including chemical sonication of exfoliated expandable graphite or chemically derived graphene sheets [6, 7], controlled nanocutting with either metal particles or scanning probe tips [8–10], etching with physical masks (e.g. nanowires) [11] and longitudinal unzipping of multi-wall carbon nanotubes [12–14]. In particular, sub-10 nm GNRs with ultrasmooth edges have been produced by sonicating thermally exfoliated expandable graphite in solution [6, 15]. FET devices based on these GNRs have demonstrated an on/off ratio as high as $10^7$ at room temperature, representing a significant breakthrough in the field of graphene-based electronics [6, 15]. High on/off ratio has also been achieved in dual-gate bilayer graphene FETs, where a bandgap is created by applying a perpendicular electric field [16].

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In this paper, we present an alternative method to fabricate GNR-FETs that exhibit the characteristic ambipolar behavior and on/off ratio exceeding $10^4$. We fabricated FET devices consisting of a GNR suspended $\sim 150$ nm above the Si substrate underneath (which is used as the back gate). Subsequently, we used controlled current annealing to create a narrow constriction in the suspended GNR to open a confinement gap, thus to afford a high on/off ratio at room temperature. Atomic force microscopy (AFM) was used to confirm the formation of a narrow constriction in the GNRs. Room-temperature high on/off ratio graphene transistors have also been previously realized by creating nanometer-sized quantum dots (QD) using electron-beam lithography and plasma over-etching [17]. However, these lithographically defined graphene QDs have an on-state conductance at least two orders of magnitude lower than that of our devices, and do not exhibit ambipolar behavior, presumably due to the substantial disorder induced by the adsorbed impurities and/or edge roughness [17].

2. Experimental details

The GNRs were produced by sonicating mildly oxidized multi-wall carbon nanotubus (MWNT) in a 1,2-dichloroethane (DCE) solution of poly(m-phenylenevinylene-co 2,5-dioctoxy-p-phenylenevinylene) (PmPV), where the PmPV is used as a surfactant to stabilize the unzipped GNRs in solution [14]. The solution was then centrifuged at 15 000 rpm (Fisher Scientific Marathon 26kmr centrifuge) for 1 h to remove aggregates and some of the remaining MWNTs; and a supernatant containing nanoribbons and the remaining MWCTs was obtained. Next, the GNR samples from the supernatant were deposited on degenerately doped Si substrates with 290 nm of thermal oxide, and subsequently non-contact mode AFM (Park System XE-70) was used to locate individual GNRs with respect to the prefabricated Au alignment marks and to characterize their thickness, width and length. The GNRs produced from this method mostly consist of 1–3 layers [14]. The AFM tip dilation effect (leading to an artificial width increase) is accounted for based on the estimated tip radius provided by the tip manufacturer [18]. Standard electron-beam lithography (EBL) was used to pattern electrodes on selected GNRs followed by thermal metal deposition (a 0.5 nm Cr adhesion layer and 50 nm of Au) and lift-off in acetone. Suspension of the GNRs in FET devices was achieved by placing a small drop of 1:6 buffered hydrofluoric acid (HF) on top of the GNR. Thermal metal deposition (a 0.5 nm Cr adhesion layer and 50 nm of Au) and lift-off in acetone. Suspension of the GNR samples from the supernatant were deposited on degenerately doped Si substrates with 290 nm of thermal oxide, and subsequently non-contact mode AFM (Park System XE-70) was used to locate individual GNRs with respect to the prefabricated Au alignment marks and to characterize their thickness, width and length. The GNRs produced from this method mostly consist of 1–3 layers [14]. The AFM tip dilation effect (leading to an artificial width increase) is accounted for based on the estimated tip radius provided by the tip manufacturer [18]. Standard electron-beam lithography (EBL) was used to pattern electrodes on selected GNRs followed by thermal metal deposition (a 0.5 nm Cr adhesion layer and 50 nm of Au) and lift-off in acetone. Suspension of the GNRs in FET devices was achieved by placing a small drop of 1:6 buffered hydrofluoric acid (HF) on top of the GNR device for 90 s to etch away approximately 150 nm of the SiO$_2$ underneath the ribbons [19, 20]. After wet etching, the device was transferred to hot isopropyl alcohol (which has low surface tension) on a 120 $^\circ$C hot plate. Finally, the device was annealed in vacuum at 600 $^\circ$C to clean the ribbon surfaces and improve electrical contacts. In some of the studied devices, an additional EBL step was used to open a window in the electron-beam resist serving as an etching mask for selectively etching the SiO$_2$ in the active device area. Both methods yielded similar results, with or without an additional EBL step. Surprisingly, about 50% of the over 30 devices with the suspended portion of the ribbon shorter than 800 nm survived the rather harsh fabrication processes.

The electrical transport properties of the suspended GNR devices were measured in high vacuum ($10^{-6}$ Torr in a Lakeshore Cryogenics vacuum probe station) and at room temperature unless stated otherwise. A semiconductor parameter analyzer (Keithley 4200) was used to apply the annealing current and to measure the device characteristics. The degenerately doped Si substrate was used as a back gate. We repeatedly applied gradually increasing annealing current and subsequently carry out the electrical measurements in situ after every consecutive step. To avoid the possible collapse of the suspended GNRs, the back-gate voltage was limited to $-20$ V $< V_g < +20$ V during the electrical measurements.

3. Results and discussion

We have studied over a dozen suspended few-layer GNRs (1–2 nm thick) with a width between 15 and 50 nm. The length of the suspended GNR ranges from 100 to 800 nm. Figures 1(a) and (b) show atomic force microscopy (AFM) images of typical devices before and after etching, respectively. The line profile of the suspended GNR (figure 1(c)) indicates that the ribbon is suspended $\sim 150$ nm above the surface of the remaining SiO$_2$ without substantial slacking (sagging)

Most of the suspended GNR devices used in this study showed ambipolar transfer characteristics with a charge neutrality point within a few volts from $V_g = 0$ V after sufficient current annealing in vacuum (at a current density $\sim 10^8$ A cm$^{-2}$). No obvious layer number dependence was observed in the transfer characteristics of our few-layer (1–3 layers) GNRs likely due to the lack of AB (Bernal) stacking in these GNRs [21]. Upon further increasing the
annealing current, about 30% of the suspended GNRs showed a dramatic increase of the on/off ratio in conductance (or drain–source current) as defined by the value measured at $V_g = -15$ V divided by the value at the charge neutrality point, while the rest of the GNRs were destroyed during the annealing processes likely due to localized overheating or electromigration [22]. Figure 2(a) shows the room-temperature conductance ($G$) versus gate voltage ($V_g$) for a representative suspended GNR device after sweeping the annealing bias voltage from 0 to a predefined set point of 2.9 V, and then decreasing the voltage back to 0 V, demonstrating the characteristic ambipolar behavior arising from the electron–hole symmetry of graphene. Figure 2(b) shows that the current versus bias voltage ($I$–$V$) of the device is linear at low bias voltages, indicating near-ohmic electrical contacts. Further current annealing (by slightly raising the predefined voltage level) dramatically reduces the minimum conductance, while the on-state conductance is essentially unchanged (within a factor of two) as shown in figure 2(c). After the device is current-annealed to 3.05 V, the on/off conductance ratio measured at $V_{ds} = 10$ mV approaches $10^5$. Figure 2(d) shows the current versus gate voltage ($I$ versus $V_g$) of the same device measured at different bias voltages after current annealing to 3.05 V, revealing an on/off ratio $>10^4$ for $10$ mV $\leq V_{ds} \leq 200$ mV and $10^3$–$10^4$ for $V_{ds} = 500$ mV. Such high on/off ratios at room temperature have been previously observed in sub-10 nm GNRs, which were attributed to the opening of an effective bandgap of the order of hundreds of meV primarily due to the confinement effect [6]. Since the bandgap decreases as the ribbon width increases and our ribbon is approximately 20 nm wide, the confinement-induced bandgap in our ribbon is expected to be of the order of $\sim 10$ meV [4, 23, 24].

To elucidate the origin of the large on/off ratio, we measured the $G$ versus $V_g$ curve of the suspended GNR device in figure 2 at various temperatures ($T$) after it was annealed to 3.05 V. Figure 3(a) shows a semi-logarithmic plot of the minimum current ($I_{\text{min}}$) at the charge neutrality point versus $1/T$ of the device. The data fit well to the thermal activation law: $I_{\text{min}} \sim \exp(-E_g/2k_B T)$ (where $k_B$ is Boltzmann’s constant), yielding a bandgap of $E_g \approx 0.6$ eV. A likely cause for such a large value of the bandgap and the consequent high on/off ratio is that further annealing beyond 2.9 V may have caused structural changes in the ribbon, such as creating a sub-10 nm constriction in the ribbon. In our suspended GNR devices, Joule annealing removes the impurities on the ribbon surfaces [25], leading to the ambipolar behavior with the charge neutrality point occurring at $V_g \approx 0$ V (figure 2(a)). Further annealing (increasing the predefined voltage level) facilitates structural reconstruction in the suspended ribbon, especially near the edges [26]. To confirm that annealing
beyond 2.9 V created a narrow constriction in the ribbon, it is necessary to characterize the structure of the suspended GNRs especially at the end of the current annealing. Raman spectroscopy has proven to be a powerful and noninvasive tool to characterize the structures of graphene [27–30]. However, it lacks the spatial resolution that is needed to confirm the formation of a nanoscale constriction. While high resolution transmission electron microscopy (TEM) is capable of visualizing individual carbon atoms [31], it is difficult to directly characterize the structure of our GNR devices using TEM as they are fabricated on Si/SiO2 substrates. In this study, we used AFM to characterize the suspended GNR device along with another suspended GNR device fabricated from an adjacent section of the same ribbon as shown in figure 3(b). The two suspended sections are thus expected to have the same width, thickness and comparable length, except that no further current annealing was carried out in the lower section as soon as the characteristic ambipolar behavior with a low on/off ratio (<10) was observed. Figures 3(c) and (d) show the high resolution AFM images of two sections of the same ribbon with high and low on/off ratios, respectively, clearly indicating that (i) both sections of the GNR are suspended without sagging (bowing) and (ii) there is a notch near the middle of the section of the ribbon with high on/off ratio (figure 3(c)) while the GNR section with low on/off ratio is highly uniform (figure 3(d)). The notch in the AFM image is a clear indication that current annealing beyond 2.9 V created a narrow constriction in the suspended GNR, resulting in a large on/off ratio. It is also worth pointing out that the AFM images in figure 3 were taken after electrical measurements with a gate voltage swept between −15 and 15 V and several thermal cycles between a cryogenic temperature (4.3 or 77 K) and room temperature, demonstrating that our suspended GNRs are thermally and mechanically stable (even those GNRs with a narrow constriction).

Similar transfer characteristics and high on/off ratios have been observed in three other suspended GNR devices with varying width, length and thickness. Figure 4(a) shows the transfer characteristics of a device consisting of a suspended GNR ~450 nm long, ~45 nm wide and ~1.6 nm thick. We note that, before current annealing, the suspended ribbon was p-doped with a charge neutrality point beyond +15 V, which can be partially attributed to the adsorption of air or water molecules, or PMMA residue [32, 33]. After intermediate current annealing, the GNR exhibits ambipolar behavior with the minimum conductance associated with the charge neutrality point shifting to \( V_g \approx 0 \) V, indicating that the adsorbed charge impurities have been largely removed by current annealing. Further annealing decreases the minimum conductance by three orders of magnitude, while the overall transfer characteristics and the on-state conductance remain essentially unchanged. Figure 4(b) shows that the transfer characteristics of the device remain virtually the same after a number of thermal cycles, although the unintentional doping level changes after each thermal cycle, as indicated by the shift of the minimum conductance along the horizontal axis. When the conductance \( G \) is plotted versus \( (V_g-V_{G-min}) \) in figure 4(c), all the three curves collapse into a single one, indicating again that the electrical properties of our devices are robust. To further demonstrate the good thermal and mechanical stability of our high on/off ratio devices, we measured the electrical properties of a third device (consisting of a GNR ~19 nm wide and ~1.2 nm thick) before and after an additional \textit{ex situ} annealing step at 600 °C in a vacuum furnace. Figure 4(d) shows that the transfer characteristics of the device remain essentially the same after the additional \textit{ex situ} annealing step.

High on/off ratios have been observed in GNRs of comparable width (tens of nanometers wide) but usually at cryogenic temperatures and have been attributed to the opening of a transport gap [4, 3, 34–36]. Several possible mechanisms have been proposed to explain the large transport gap observed in GNR-FETs at low temperatures, ranging from renormalized lateral confinement due to localized edge states [4, 3], to a percolation-driven metal–insulator transition caused by charged impurities [34], to quasi-one-dimensional Anderson localization [37], and to Coulomb blockade due to edge roughness [38]. More recent experimental studies on disordered GNRs further indicate that charge transport in the
Figure 4. (a)–(c) Transfer characteristics of a second suspended GNR-FET device measured at room temperature; the suspended GNR is 45 ± 3 nm wide, ~1.6 nm thick and ~450 nm long. (a) Transfer characteristics measured after various degrees of annealing. (b) Conductance versus gate voltage measured at room temperature after the final current annealing stage and after different numbers of thermal cycles; #1, #2 and #3 next to the corresponding $G(V_g)$ curves represent measurements after 1, 2 and 3 thermal cycles, respectively. (c) Gate-dependent conductance data in (b) plotted as a function of $V_g - V_{G_{\text{min}}}$. (d) Room-temperature transfer characteristics of a third suspended GNR device (19 ± 3 nm wide, ~1.2 nm thick and ~300 nm long) measured before (blue solid squares) and after (red solid dots) ex situ annealing at 600°C for 10 min in a vacuum furnace.

Figure 5. Schematic density of states (DOS) for GNRs. (a) and (c) represent two infinite GNRs of different widths; the corresponding energy bandgaps, observed in the DOS panels (d) and (f), follow the known inverse relation with ribbon width. (b) GNR with a constriction. (e) DOS projected on the region of the constriction; the quantized energy levels of quantum dots QD1 and QD2 are shown as narrow horizontal bars.

The conduction gap of GNRs is likely dominated by localized states [35] and/or isolated charge puddles acting as quantum dots [36]. These mechanisms may partially contribute to the high on/off ratio in our devices. However, they are unlikely to be the primary cause, since the over $10^4$ on/off ratio in our devices was observed at room temperature and only after sufficient current annealing. For instance, when the device in figure 2 is current-annealed up to 2.9 V, the on/off ratio is less than 2. The large on/off ratio in the device was obtained only after it was current-annealed by applying a bias voltage beyond 2.9 V.

Based on the results of AFM characterization of the suspended GNRs (figure 3), we suggest that the formation of a constriction, at the critical annealing current, as the most likely origin of the high on/off ratio. Figures 5(a)–(c) schematically show a relatively wide uniform GNR (GNR0), the same GNR with a narrow constriction, which effectively forms a finite ribbon (GNR1), and a narrow uniform GNR (GNR2) with a decreasing width (GNR0 > GNR1 > GNR2). Because of the stronger confinement in the narrow constriction (GNR1) compared to the uniformly wide GNR (GNR0), the bandgap for GNR1 (figure 5(e)) is expected to be larger than that for GNR0 (figure 5(d)). In the same fashion, the bandgap of GNR2 (figure 5(f)) is larger than that of GNR0 (figure 5(d)) due to its narrower width [3–5]. In addition to the lateral confinement, region GNR1 is also longitudinally confined by regions QD1 and QD2. The longitudinal confinement
further increases the bandgap of the otherwise infinite ribbon GNR$_1$ to a value that can be possibly larger than that for the narrower ribbon GNR$_2$, thus resulting in higher on/off ratios. Therefore, the double-confinement picture seems to be responsible for the high on/off ratios which are comparable to those of possibly much narrower (sub-5 nm) ribbons [39]. More quantitative understanding of the large bandgap requires detailed information on the dimensions and even the edge structures of the nanoconstrictions, which is beyond the scope of this work. Recently, bandgaps of $\sim 50$ meV have been observed in relatively wide ($\sim 100$ nm) annealed nanotube-derived GNRs [40], indicating that it is possible to obtain a bandgap $\sim 500$ meV in GNRs of $\sim 10$ nm wide. However, the bandgap of these GNRs was substantially larger than that of much narrower GNRs synthesized using a similar approach and the difference in edge structures was suggested as the primary cause of the discrepancy [14, 40]. Unfortunately, the lack of edge information on these GNRs prevents the definitive identification of the true origin of the discrepancy.

Discrete conductance peaks, superimposed on the main $G$ versus $V_g$ curve, were observed at low temperatures in some annealed samples (figure 6). These conductance oscillations can be attributed to the quantized energy levels of regions QD$_1$ and QD$_2$ in figure 5(b), which, because of the lack of any periodicity or quasi-periodicity, act as two quantum dots in series. Therefore, these conductance peaks are due to resonant tunneling through the quantized levels of the dots (shown as narrow horizontal bars in figure 5(e)) tuned by the applied gate voltage. The spacing and periodicity of such peaks depend on the size and symmetry of the quantum dots. Asymmetry between the discrete energy levels of QD$_1$ and QD$_2$ leads to a lack of resonance and random cancellation of the transmission through some levels [17].

During the course of this work, we became aware that room-temperature high on/off ratios were also observed in graphene nanoconstrictions created by first forming a constriction in the gold etch mask covering the graphene channel (using electromigration) and subsequent plasma etching of the graphene underneath [41]. However, the nanoconstrictions presented in this work are created without a mask, which may lead to much cleaner graphene nanoconstrictions with substantially lower disorder.

4. Conclusion

We have fabricated suspended GNR-FET devices from GNRs derived from high quality MWNTs. By controlled current annealing of the suspended GNRs, a large bandgap of the order of hundreds of meV can be created in GNRs with varying width and thickness, leading to an orders of magnitude increase of the on/off ratio at room temperature. The formation of such a large bandgap can be largely attributed to the creation of a narrow constriction in the suspended GNR as confirmed by AFM. Furthermore, the suspended GNRs and their narrow constrictions are structurally robust, and the electrical properties of the devices remain unchanged after a number of thermal cycles. The mask-free fabrication method presented in this study also creates opportunities for studying ultraclean graphene quantum dots.

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