

Current mapping in Graphene Contacts to AlGa_N/Ga_N Heterostructures

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Abstract: We have exploited conductive atomic force microscopy (CAFM) to characterize the vertical current transport from graphene (Gr) to the 2D electron gas of Al_xGa_{1-x}N/GaN heterostructures considering different kinds of AlGa_N surfaces in terms of roughness and unevenness. The vertical current transport mechanism can radically change depending on these nanometer size superficial fluctuations whereby the nanoscale lateral resolution of CAFM current-voltage (I-V) measurements offers the ideal conditions to distinguish this effect from the average macroscopic behavior. We have characterized bare and Gr-coated high quality AlGa_N surface at first, observing for both a rectifying behavior. In particular the contact on Gr shows a lower Schottky barrier height (SBH) ($\Phi_B = 0.4$ eV) than the bare AlGa_N ($\Phi_B = 0.9$ eV), and a smaller spread between the array of sampled positions. In particular this lateral homogeneity can be explained as an averaging effect of Gr on the AlGa_N surface potential fluctuations over a length scale around the AFM tip in the order of the electron mean free path of a transferred CVD grown Gr (~100 nm). In order to exclude the role of the AFM metal tip force contact to the observed behavior we have performed a force dependent characterization establishing a tip force range in which this effect is negligible. We have also repeated the same characterizations on a Gr/AlGa_N/GaN heterostructure with a high structured AlGa_N surface. In this case a lower SBH ($\Phi_B = 0.6$ eV) and an ohmic behavior have been observed on bare AlGa_N and Gr-coated AlGa_N respectively. This result has been attributed to the presence of preferential current paths in correspondence of the surface voids and the contemporary collection of the AFM morphology and the current map of the bare AlGa_N has confirmed it. In particular, the ohmic behavior through Gr has been imputed to a contemporary lowering of the SBH and a homogenization effect of a certain density of preferential current paths.

Keywords: Graphene, AlGa_N/Ga_N Heterostructures, Atomic Force Microscopy, Conductive Atomic Force Microscopy

1. Introduction

Graphene (Gr)[1] has been the object of many scientific interests in the last ten years. The extraordinary combination of electrical [2], optical [3], thermal [4] and mechanical properties [5], makes Gr a promising keystone for many technological advancements in electronic, optoelectronic, photovoltaic, thermal management and flexible devices.

Obviously, next to its extraordinary properties many limiting factors are also widely known. For instance, although the outstanding in plane mobility (ideally $\mu > 10^5$ cm²V⁻¹s⁻¹ at R.T. [2]) makes this material a potentially superb channel for Field Effect Transistors (FETs) the lack of a band gap at the Dirac point produces an intrinsically poor modulability of the channel and a consequent unacceptably

low current On/Off ratio for switching applications.

Moreover, although the current transport mechanism through the plane is largely studied, the vertical current transport from graphene to another conductive medium is a less emphasized aspect even if this can play a leading role in the final physiology of the device.

Depending on the conductive material, same aspects of interest related to the current transport can be listed. To obtain high quality Gr based electronic devices the nature of the junction between Gr and the metallic contact needs a certain control. Hence, it is of great importance to clarify the factors that determine the contact resistance and the role of the metal work function [6], the field effect, the temperature [7] and so on.

Gr heterostructures with semiconductors paves the way to

an even more fascinating scenario of applications. In this case, the lack of the Gr band-gap can be overcome by creation of a FETs channel composed by a properly fabricated Gr/semiconductor heterostructure in order to establish a Schottky junction. As reported for the first time in the case of Gr transferred over a hydrogen saturated silicon surface, the barrier height established at this heterojunction can be modulated by a field effect, giving a reasonable current On/Off ratio to the final device (current On/Off ratio of 10^5) [8].

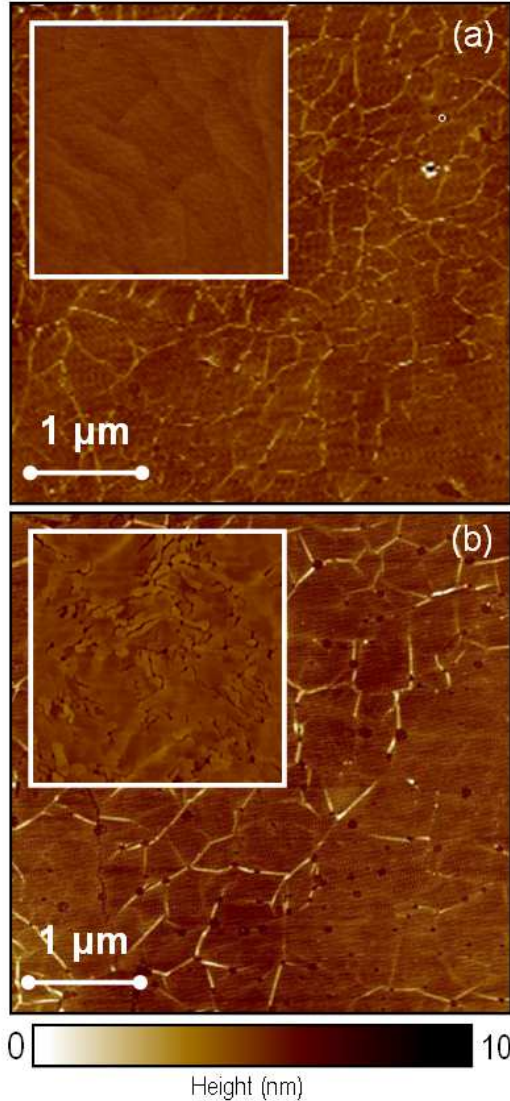


Figure 1. AFM morphologies of Gr transferred on the defect-free (a) and on the defect-rich (b) AlGaIn surfaces. The inserts of (a) and (b) show the morphologies of the correspondent bare AlGaIn/GaN substrates before the Gr transferring, with equal dimension scale.

It is well known that nanometer sized defects or inhomogeneities at the heterointerface of this kind of stack structures can play a leading role on the final macroscopic average behavior. Nevertheless, a macroscopic electrical measurement does not allow one to distinguish the specific contribution of such features. In this sense, nanoscale

resolution electrical characterizations can represent the essential approach to understand the electrical behavior of the heterojunction. Atomic force microscopy (AFM) based electrical characterization techniques have been extensively applied for various kind of investigations on Gr, such as the potential distribution [9], capacitance [10], the conductivity [11] the electron mean free path [12] and the unevenness of the Schottky barrier height at the junction between graphene and a semiconductor [13]. In this sense, we recently reported a nanoscale investigation of the vertical current transport from Gr to the 2D Electron Gas (2DEG) spontaneously formed at the heterointerface of an AlGaIn/GaN heterostructure [14]. In this case, the interface between Gr and the semiconductor surface has a decisive role on the transport mechanisms at the heterojunction determining a radical change of the electric behavior (from Schottky to Ohmic) depending on the superficial microstructures of the AlGaIn layer [15]. For this purpose, nanoscale electrical characterizations techniques, such as the Conductive Atomic Force Microscopy (CAFM), played a prominent role to clarify the interface driving mechanisms related to the surface peculiarities.

CAFM is a scanning probe microscopy technique, which allows one to characterize the local current transport mechanism from the substrate to a nanometer-sized tip with a high lateral resolution and eventually to compare the current map with the correspondent local morphology.

Here it is reported a detailed description of the CAFM measurements performed in order to characterize the current transport mechanisms from Gr to the AlGaIn/GaN 2DEG and the decisive role of the surface unevenness in terms of potential fluctuations and morphological microstructures in the final transport mechanisms.

2. Results and Discussion

We performed the following experiment over two different kinds of Gr/AlGaIn/GaN heterostructures fabricated starting from the same Gr film grown by chemical vapor deposition (CVD) over a copper foil and transferred on the final substrates by a highly reproducible unconventional transfer method. The transferring involved an electrolytic delamination of the carbon membrane from the metal foil [16][17] and a subsequent finely controlled thermo-compressive transfer printing on the final target surface. This highly reproducible transfer procedure ensured to obtain Gr membranes with comparable qualities on the two AlGaIn samples with different morphologies. This is essential for the comparison described in the following, since the effect of different Gr qualities can be excluded. Fig. 1 reports AFM morphology of Gr transferred on two different kind of $\text{Al}_{0.25}\text{Ga}_{0.75}\text{N}/\text{GaN}$ heterostructures with a 24 nm thick AlGaIn layer. In both cases, a wrinkled morphology is observed as the consequence of the cooling down step of the CVD growth process during which the difference in the thermal expansion coefficient between Gr and the copper determines these peculiar corrugations inevitably reported on

the final substrate [18]. Conversely, fractures and folds, typically due to the quality of the transfer process, are scarcely observed overall, confirming the high reliability of the transfer procedure. The inserts of fig. 1 (a) and (b) show the morphology of the correspondent bare AlGaIn/GaN substrates before the Gr transferring: an atomically smooth and defect-free surface for sample in fig. 1 (insert of a) and a highly structured and defect-rich morphology for sample in fig. 1 (insert of b).

The measurement apparatus, which allowed us to collect local Current-Voltage (I-V) characteristics is constituted by an Atomic Force Microscope equipped with a high sensitivity current amplifier in series with an Au coated tip, which acts as nanometric metal contact over the sample surface. The bias is applied between the CAFM and a macroscopic ohmic contact fabricated over the bare AlGaIn surface to connect the AlGaIn/GaN 2DEG, as schematically illustrated in fig. 2 (a).

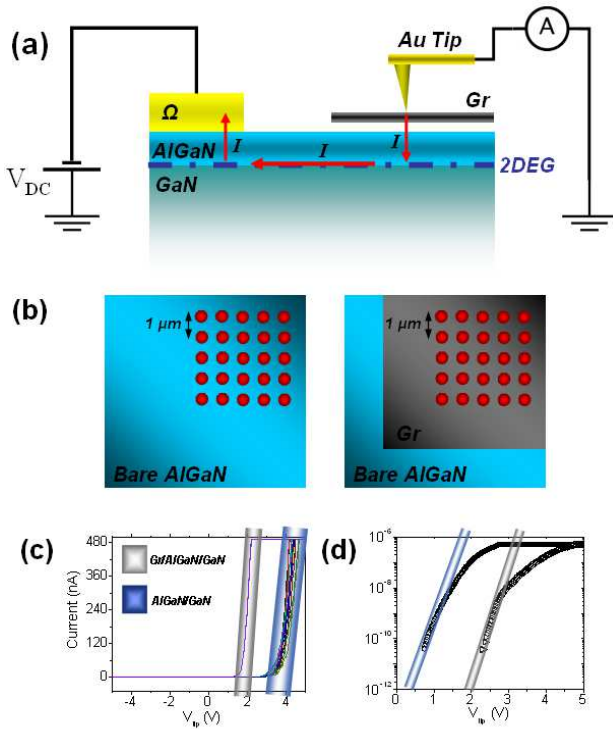


Figure 2. (a) Schematic representation of the experimental setup for local I-V measurements by CAFM and (b) scheme of the square arrays of positions mapped on bare AlGaIn and on Gr. (c) Two different sets of I-V curves collected using an Au coated AFM tip for both Gr coated (highlighted by gray rectangle) and bare (highlighted by blue rectangle) defect-free AlGaIn/GaN heterostructure. (d) Two representative I-V curves for Gr coated (gray rectangle) and for the bare (blue rectangle) AlGaIn, reported in a log I-V plot.

In this configuration, the major contribution to the measured resistance is due to the vertical current path from the tip to the AlGaIn/GaN 2DEG, whereas the path from this 2DEG to the large-area contact adds a negligible series resistance contribution.

The nanometric I-V characterization was performed both over the bare AlGaIn and over Gr and replied over square arrays of 25 positions with a precise constant path, as

schematically illustrated in fig. 2 (b). The path length of the square array was fixed at 1 μm in both x and y directions, as a reasonably high value in order to collect completely independent curves considering that the electron mean free path typically observed in substrate supported Gr is in the order of hundred nanometers [12]. The importance of this detail will be clarified in the following.

We considered the defect-free sample (fig. 1 (a)) at first, as the closest example to the ideal morphological interface between Gr and AlGaIn. As reported by the I-V characteristics in fig. 2 (c), the vertical current transport trough Gr or trough the direct contact of the metal tip over the bare AlGaIn, show a rectifying behavior with a negligible current at reverse bias and a rapidly increasing current at forward bias higher than a certain onset voltage. It is immediately evident that the set of I-V characteristics collected on Gr show a lower onset voltage and a definitely smaller spread (fig. 2 (c) gray rectangle) if compared with the set collected on the bare AlGaIn (fig. 2 (c) blue rectangle).

As discussed in ref. [14], in order to extract the Schottky barrier heights (SBH) Φ_B in both cases, it is possible to apply the thermionic emission model. In fig. 2 (d) two representative I-V curves for Gr and for the bare AlGaIn are reported in a log I-V plot. In the low forward bias regime, the curves exhibit a linear behavior, which can be fitted with the thermionic emission relation:

$$I \approx SA^*T^2 \exp\left(-\frac{q\Phi_B}{kT}\right) \exp\left(\frac{qV}{nkT}\right) \quad (1)$$

Being S the tip contact area, A^* the Richardson constant for AlGaIn, k the Boltzman constant, T the temperature, q the electron charge, V the bias and n the ideality factor.

By Gaussian fitting of the SBH extracted for both sets of curves we reported $\Phi_B = 0.41 \pm 0.04$ eV and $\Phi_B = 0.95 \pm 0.12$ eV, for Gr and bare AlGaIn respectively, and coherently with what previously discussed.

What observed on bare AlGaIn is completely consistent with the expectations. It is well known that the Schottky behavior is always observed on AlGaIn/GaN substrates unless specific metal alloys (Ti/Al based alloy) and proper thermal treatments are exploited [19][20]. Regarding the spread between the local I-V curves measured on bare AlGaIn, the phenomena can be associated with surface potential fluctuations, which are typical sources of the laterally inhomogeneous Schottky barrier between metals and GaN or its alloys [21].

What observed on Gr deserves a different attention. According to the Schottky-Mott rule [22][23] SBH expected for an ideal Schottky contact between intrinsic Gr (with W_{Gr} 4.5 eV) and an $\text{Al}_{0.25}\text{Ga}_{0.75}\text{N}$ alloy (with χ 2.7 eV) is $\Phi_B = W_{Gr} - \chi = 1.8$ eV.

The measured SBH is much lower than this theoretical value indicating that the Gr/AlGaIn Schottky contact formation is ruled by a pinning of the Gr Fermi level due to the presence of interface states. Considering that Gr is only weakly (van der Waals) bound to the AlGaIn substrate

structural modifications occurring in Gr during the transfer procedure can be excluded as the pinning source. Rather, these can be related to the surface states of pristine AlGaIn [14][24]. Regarding the extremely low spread between curves it can be argued that differently from classical metal electrodes, consisting of polycrystalline films composed by grains with different sizes and crystalline orientations, a Gr membrane represents a uniform, completely conformal and atomically thin electrode over the AlGaIn surface. The sharp spread between curves collected on Gr most probably results from an averaging effect on the AlGaIn surface potential fluctuations over a length scale in the order of the Gr electron mean free path l_{Gr} , which is ~ 100 nm for substrate supported Gr [12]. It is a much larger value than the electron mean free path of common metal electrodes (typically 1-10 nm). In this case, the area between Gr and the AlGaIn surface concretely involved in the current transport mechanism is extended for at list ~ 100 nm around the tip contact, as schematically illustrated in fig. 3. As mentioned before, the path length of the square array for the collected I-V curves fixed at $1 \mu\text{m}$ is reasonably high to consider every single measure independent involving Gr contact areas not partially overlapped to the neighboring.

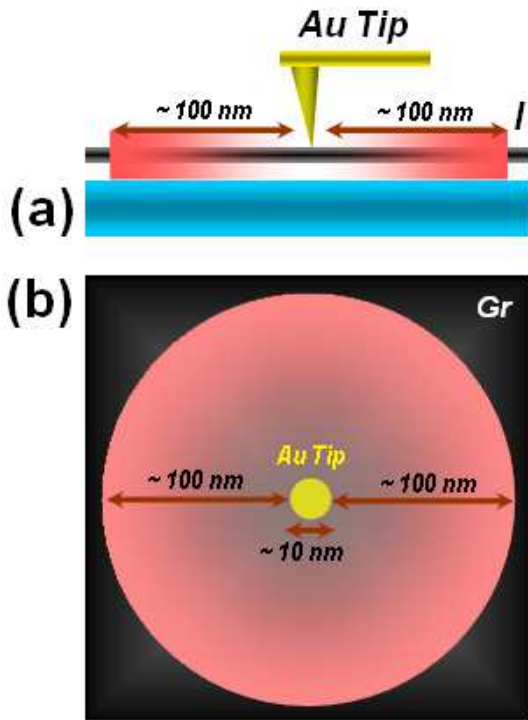


Figure 3. Schematic representation in front view (a) and top view (b) of the Gr area around the AFM tip, involved in the current transport mechanisms with the underlying AlGaIn.

It could be argued that establishing a metallic contact by a metal-coated AFM tip could be considered not equivalent to the formation of a metallic contact by a deposition approach. Assuming that the deposited metal contact does not involve any chemical modification or chemisorptions to the underlying surface, it is possible to consider in both cases an

adhesive force component. Nevertheless, while for a deposited metal the distance at the interface is established by equilibrium of the attraction and the repulsion between the two materials, an AFM tip approaches the surface applying vertical force and undergoing to a surface attraction. In this sense while the tip engage the surface to establish a contact mode, with an increasing applied vertical force, the cantilever is previously positively deflected from surface attraction (pull-on force) and then increasingly negatively deflected by the applied force. In the second stage, the higher is the applied force the higher is

the cantilever deflection and the tip compression over the sample. In order to exclude the effect of the tip-sample force, which could be particularly relevant in the case of Gr as a flexible and deformation sensitive material, we repeated the same measurements for various normal forces within a reasonable range of values, properly setting the cantilever deflection set point (DSP). The DSP and the normal loading force are linearly correlated by the Hooke's law. Fig. 4 reports Φ_B , extracted as previously described, for both the bare AlGaIn (red curve) and the Gr coated AlGaIn (black curve) for various normal forces. It was observed an extremely stable behavior giving essentially identical Φ_B values, with standard deviations of $\pm 0,04$ eV and $\pm 0,002$ eV for bare AlGaIn and Gr coated AlGaIn respectively, and very similar error bars (dispersion between positions of the square array of measures) in a range of normal forces between 380 and 1520 nN. After this value a very slight deviation and small increase of the error bar is observed (one point reported in fig. 4 for 1900 nN).

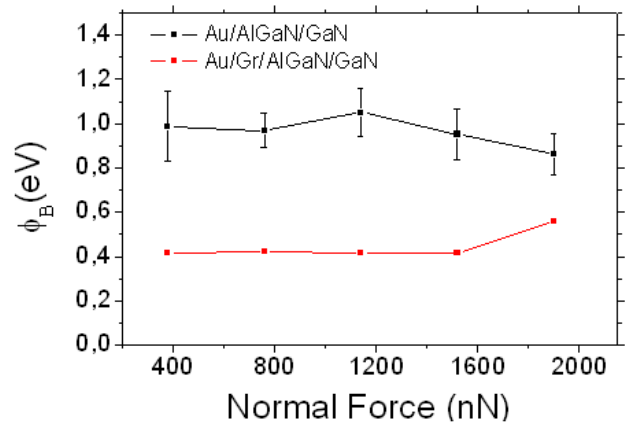


Figure 4. Φ_B vs. the normal force for the bare (in black) and the Gr coated (in red) defect-free AlGaIn/GaN heterostructure.

The measures collected in the range of forces between 380 and 1520 nN give a standard deviation one order of magnitude lower than the deviation due to the surface potential fluctuations statistically observed mapping the surfaces for every fixed DSP. It means that in this range of DSPs we are reasonably confident that the effect of the tip-surface force is not influential on the SBH and its fluctuations.

Moreover, the 20 time lower standard deviation observed

on Gr coated AlGa_N than on bare AlGa_N confirms what previously argued about the effective Gr contact area around the AFM tip. In fact, in this case, even if different tip-surface forces are applied, the compression effect is expected just in a fraction of the total contact area, immediately under the tip contact, contributing marginally to a SBH deviation (scheme in fig. 3, yellow dot).

The same method described until now was identically adopted to characterize the defect-rich Al_{0.25}Ga_{0.75}N/GaN heterostructure (fig. 1(b)). For bare AlGa_N a lower SBH and a higher spread between curves is observed (fig. 5(a)) while the Gr coated AlGa_N shows an ohmic behavior with a very sharp spread between positions (fig. 5(b)).

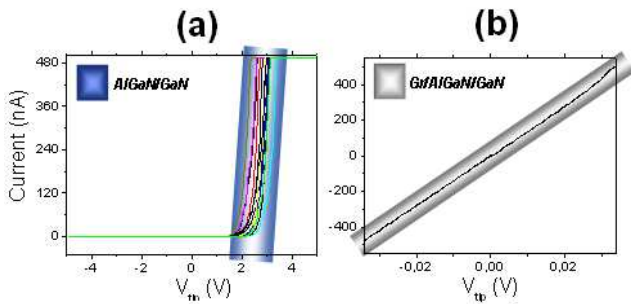


Figure 5. two sets of *I-V* curves collected using an Au coated AFM tip on bare defect-rich AlGa_N/Ga_N (a) and on Gr coated defect-rich AlGa_N/Ga_N (b).

The SBH extracted on the bare AlGa_N is $\Phi_B = 0.63 \pm 0.17$ eV [15]. This result compared with the one observed on defect-free bare AlGa_N could be explained considering the high density of microstructured voids shown in the insert of fig. 1(b). A contemporary collection of the AFM morphology (fig. 6(a)) and the correspondent current map (fig. 6(b)) of the AlGa_N surface at a fixed bias of -2 V allowed us to confirm that the superficial voids act as preferential current paths.

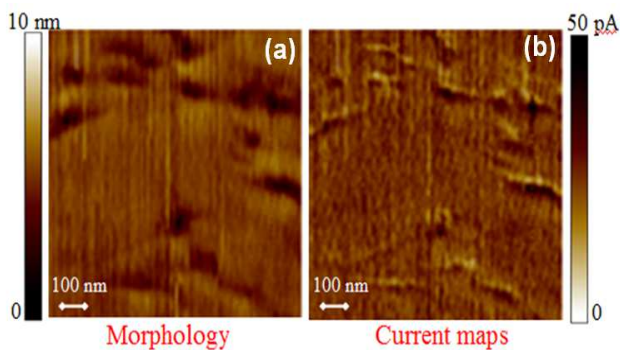


Figure 6. (a) AFM Morphology and (b) correspondent current map collected on the defect-rich AlGa_N substrate.

The ohmic behavior of the Gr coated AlGa_N can be explained considering the combination of several effects. In this case, the previously described low value of the Gr/AlGa_N SBH is associated to the local increase of the current at the AlGa_N superficial voids. The higher current is probably related to a local reduced thickness, which

generates low resistance conduction paths through a direct or a Fowler Nordheim tunneling mechanisms. Considering that the defects separation is equal or lower than the electron mean free path in supported Gr, it can be assumed that Gr connects several defects in series over a certain area around the tip. This net-effect allows a preferential ohmic behavior and explains how a so limited spread between curves is observed even on this sample where a high density of defects underlies the Gr contact.

3. Conclusions

CAFM is the ideal technique to characterize the local current transport mechanism from Gr to a semiconductor substrate due to the nanometric contact established by the metal-coated tip with the surface. In particular, we analyzed the vertical current transport on a high quality AlGa_N/Ga_N heterostructure, comparing the bare AlGa_N and the Gr coated AlGa_N surface. We observed a lower SBH in the presence of Gr attributed to a Fermi level pinning driven by the AlGa_N surface states. CAFM allowed us to repeat the *I-V* characterization on an array of independent positions in order to establish the lateral homogeneity of the sampled substrate. In this sense, Gr demonstrated a highly repeatable behavior, attributed to a homogenization effect of the AlGa_N surface potential fluctuations over a length scale of the Gr electron mean free path. In order to exclude a contribution of the tip force in the result we repeated the measure for different DSP, observing a negligible effect within a reasonable range of force values.

The nanoscale investigation allowed us to evaluate current transport between Gr and a defect-rich AlGa_N surface demonstrating an ohmic behavior through the heterostructure. By a direct comparison between the defect-rich AlGa_N AFM morphology and the AFM current map, preferential current paths were observed in correspondence of the surface voids. It allowed us to assert that Gr not only reduces the SBH but also connects in series as a net a certain density of preferential current paths to form an ohmic contact.

4. Experimental Section

4.1. Heterostructure Fabrication

Al_{0.25}Ga_{0.75}N/GaN heterostructures were grown by metal organic chemical vapor deposition (MOCVD) on Si (111) wafers. Gr was grown by chemical vapour deposition (CVD) on a ~25 mm thick polycrystalline copper foil at 1000°C under CH₄/H₂ flux as precursors. Gr on copper foil was spin coated with poly(methyl-methacrylate) (PMMA) b for 60 seconds at 1000 rounds per minutes and subsequently baked at 150°C for 10 minutes. Gr/PMMA stack was electrochemically delaminated 0.2 M KOH water solution with an applied voltage of 5 V between the copper foil (cathode) a gold counter electrode [17]. Gr/PMMA stack was printed on the target substrate and PMMA was removed in acetone at room temperature.

4.2. AFM Morphological Analyses

Tapping mode AFM measurements were carried out with Veeco DI3100 atomic force microscope with Nanoscope V controller. Commercial silicon probes with spring constant $k = 20$ to 80 Nm^{-1} and oscillation frequency from 332 to 375 kHz were employed.

4.3. Nanoscale Electrical Characterizations

Local current measurements were carried out at room temperature by conductive atomic force microscopy (CAFM), using a DI3100 AFM with Nanoscope V electronics. These nanoscale resolution electrical analyses were performed both on Gr-coated and bare AlGaIn regions (reference) using AFM Au coated tips obtained by thin film sputtering. During the scan, a DC bias was applied between the tip and a large-area contact on bare AlGaIn, and the current flowing between these two contacts was collected by a high sensitivity current amplifier in series. The I-V characteristics have been collected sweeping the DC bias from -5 to 5 V with a ramp rate of about 5 V/s .

4.4. AFM Current Maps

The contemporary collection of the morphology and of the electrical current map was carried out by Torsional Resonance Tunneling AFM (Torsion TUNA) using a DI3100 AFM with Nanoscope V electronics. Commercial Pt coated silicon probes with spring constant $k = 21$ to 98 Nm^{-1} and resonance frequency from 146 to 236 kHz were employed. The measurements were carried out adopting a scan frequency per line of $0,5 \text{ Hz}$.

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