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Magnetic scanning gate microscopy of graphene Hall devices (invited)

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We have performed sensitivity mapping of graphene Hall devices with the width of 0.6–15 μm operating in the diffusive regime under non-uniform, local magnetic and electric fields induced by a scanning metallic magnetic probe. The transverse voltage was recorded, while tuning the magnitude and orientation of the bias current, the probe-sample distance, and orientation of the probe magnetization. A strong two-fold symmetry pattern has been observed, as a consequence of capacitive coupling between the probe and the sample. The effect is particularly pronounced in small devices (<1 μm), where the dominating electric field contribution significantly lowers the effective area of the magnetic sensor. We show that implementation of the Kelvin probe feedback loop in the standard scanning gate microscopy setup drastically reduces parasitic electric field effects and improves magnetic sensitivity. © 2014 AIP Publishing LLC. [http://dx.doi.org/10.1063/1.4870587]

I. INTRODUCTION

Miniaturized Hall sensors made of two dimensional electron gas (2DEG) semiconductor materials, and recently of atomically thin graphene, have been increasingly used as high sensitivity devices for detection of localized magnetic fields in biomedical applications1,2 and data storage.3,4 The behavior of such devices depends on many physical parameters, and a significant effort has been made across a number of disciplines on optimization of the Hall sensor performance. This optimization becomes particularly relevant on a submicron scale, where it is often necessary to calibrate the Hall devices locally.5 One of the crucial intrinsic parameters is the distribution of the local carrier density (potential landscape), which determines the electron scattering and total electrical resistance of the sample. The necessity to understand and ideally visualize how this landscape changes when locally perturbed by electrical and magnetic fields has long been recognized.6

Previous research in the area, implementing a scanning gate microscopy (SGM) technique was primarily focused on studies of 2DEG semiconductor devices. Baumgartner et al. used local electric and uniform magnetic fields to demonstrate that GaAs/AlGaAs Hall devices generally give different responses to local electric perturbation in diffusive6 and ballistic7 regimes at low temperatures. This approach was significantly developed in room temperature experiments on AlSb/InAs/AlSb near-surface quantum wells, where contributions of local magnetic and electrical fields to the total transverse voltage signal were first reported and transition from diffusive to quasi-ballistic transport was visualized.8 These experiments demonstrated that proper separation of local electric (scanning probe or geometry induced) and magnetic field contributions is crucial, though often difficult to implement. Electrostatic simulation was performed to study the effect of device geometry and allowed the improvement of device performances in terms of magnetic sensitivity and reduced impact of the electrostatic effects.9

In our earlier works, we employed SGM technique to study the local changes of the carrier concentration in epitaxial graphene devices,10,11 In contrast to conventional 2DEG devices, where the sensing channel is often placed a few tens of nanometers beneath the device surface due to the insertion of cap layers, the carriers in the graphene channel can be easily approached and influenced by the magnetic probe, enabling a higher spatial resolution in scanning gate experiments. Additionally, graphene-based Hall sensors benefit from high sensitivity (Hall coefficient, RH)12 and robustness to large biasing currents (Ibias).13 The possibility to use such devices for efficient and straightforward detection of a single magnetic bead was recently demonstrated.11

In this paper, we report a detailed study into the characterization of graphene based Hall devices to local electric and magnetic fields. It has been observed that these devices suffer from the same inherent problem as their semiconductor prototypes, namely, a superposition of local potential and magnetic responses. Depending on device dimensions, the contribution arising from the electrostatic forces between the current-biased device and metallically coated probe can significantly dominate the magnetic signal, thus decreasing the effectiveness of such devices for sensing applications. Therefore, caution is to be stipulated when interpreting sensor signal obtained in real world applications.

The paper consists of two sections. First, we perform a detailed characterization of a range of epitaxial graphene devices in the classical Hall effect regime using local, non-uniform magnetic and electrical fields applied to the metallic magnetic scanning probe. We study the response of the

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graphene Hall device by varying parameters such as the bias current magnitude and direction, orientation of the probe magnetization, and distance between the probe and sensing surface. In the second part, we implement a combination of SGM with frequency-modulated Kelvin probe force microscopy (FM-KPFM) feedback loop.\textsuperscript{14} This completely eliminates the parasitic electric field contributions, allowing us to characterize the sensor to magnetic fields alone. The SGM-KPFM technique is particularly important for characterizing small graphene devices, which are greatly influenced by the parasitic electric field. For accurate interpretation of results, we demonstrate that it is necessary to perform the non-uniform field characterization to calibrate the sensor prior to real world applications, such as magnetic bead detection. The experimental results are supported with a finite element model that has been developed to simulate the spatial distribution of the electric potential inside the graphene Hall bars in the presence of a probe that locally induces magnetic and electric fields.

II. EXPERIMENTAL SETUP

The one-two layer epitaxial graphene (1LG and 2LG, respectively) was grown on the Si-terminated face of a nominally on-axis 4H-SiC(0001) substrate at 2000°C and 1000 millibars argon gas pressure in a sublimation furnace.\textsuperscript{15–17} The resulting material is n-doped, owing to charge transfer from the interfacial layer, with the measured electron concentration in the range \(n_e = 6 - 20 \times 10^{11} \text{ cm}^{-2}\) and mobility (\(\mu\)) of \(\sim 200 - 3000 \text{ cm}^2 \text{V}^{-1} \text{s}^{-1}\) at room temperatures.\textsuperscript{12,18}

The fabrication process involves various steps of electron beam lithography, oxygen plasma etching, and thermal deposition of Cr/Au (5/100 nm) electrical contact.\textsuperscript{19} Using these processes, symmetrical double-cross devices with width (\(w\)) of 0.6–15 \(\mu\)m were fabricated (Fig. 1). Initial characterization of devices was performed at room temperature using magnetotransport and noise spectral measurements.\textsuperscript{12} Results are summarized in Table I.

Scanning probe microscopy (SPM) measurements were carried out using a Nanotec SPM system controlled by the WSxM software,\textsuperscript{20} using the amplitude modulation mode with a phase-locked loop (PLL) that keeps the oscillation phase constant. Topography height images of the graphene device were recorded simultaneously with tapping phase and scanning gate images, which were compiled either on their own or with implementation of the FM-KPFM feedback. For magnetic gating and calibration, the following probes were used:

| TABLE I. Summary of transport and noise measurements for epitaxial graphene devices with the width \(w\) of 0.6–15 \(\mu\)m, where \(R_4\) is the 4-point resistance, \(R_H\) is the Hall coefficient, \(n_e\) is the electron density, \(\mu\) is electron mobility, and \(B_{\text{min}}\) is the minimal detectable field. |
|——|——|——|——|——|——|——|
| Size (\(\mu\)m) | \(R_4\) (k\(\Omega\)) | \(R_H\) (\(\Omega\)/T) | \(n_e\) (cm\(^{-2}\)) | \(\mu\) (cm\(^2\)/V s) | \(B_{\text{min}}\) at \(I_{\text{bias}} = 60 \mu\)A (\(\mu\)T/Hz) |
| 0.6 | 29.5 | 296 | \(2.1 \times 10^{12}\) | 803 | 4.9 |
| 1  | 33.6 | 838 | \(7.5 \times 10^{11}\) | 998 | 3.7 |
| 10 | 22.7 | 715 | \(8.7 \times 10^{11}\) | 170 | 3.7 |
| 15 | 26.2 | 1022 | \(6.1 \times 10^{11}\) | 3124 | 1.3 |

FIG. 1. (a) Topography and (b) surface potential images of a 0.6-\(\mu\)m wide device.

(i) Commercial probes (Bruker) with two different thicknesses of CoCr coating, i.e., MESP (40 nm) and HM-MESP (>40 nm), with coercivity \(\sim 400 \text{ Oe}\) and moment (\(m\)) of \(\sim 1 \times 10^{-13}\) and \(>3 \times 10^{-13}\) emu, respectively.\textsuperscript{21}

(ii) Customized probes, which were prepared by sputtering cobalt films with thicknesses of 15 and 20 nm onto one side of commercially available non-magnetic probes.

Both types of probes have a resonance frequency of \(f_0 \sim 75\) kHz with a spring constant of \(\sim 3\) N/m.

FM-KPFM is one of the most widely used surface potential mapping techniques that utilize AC/DC voltages simultaneously, while scanning the sample’s topography using dynamic mode AFM. The Kelvin feedback loop eliminates the electrostatic forces by applying a compensating DC voltage to the probe at each pixel such that \(V_{\text{probe}}\) equals local potential at the sample surface.\textsuperscript{19,22} The surface potential of the sample is mapped by recording \(V_{\text{probe}}\) at each pixel.

Magnetic SGM is realized by scanning the device and measuring the voltage response to a localized magnetic field (\(B_{\text{probe}}\)) produced by a magnetically coated probe. When scanning a DC current-biased Hall sensor with a magnetic force microscope (MFM) probe oscillating at \(f_0\), the sensor experiences an oscillating magnetic field (\(dB_{\text{probe}}/dz\)), giving rise to an oscillating transverse voltage (\(dV_{\text{probe}}/dz\)). The voltage response is mapped at each pixel by measuring the sensor with an external lock-in amplifier referenced to \(f_0\). It should be noted that \(f_0\) varies when PLL feedback is on, making sure the relevant information on the in-phase channel is recorded and avoiding frequency-shifts induced by probe-sample interactions.

The magnetic coating of an MFM probe is also electrically conductive; therefore, performing SGM on a current-biased device results in the probe-sample potential difference. In this experiment, a unipolar current source was used, which effectively makes the potential of the sample always higher than potential of the probe, i.e., \(V_{\text{sample}} > V_{\text{probe}}\), if the probe is grounded. The response of the Hall sensor to the parasitic electric field masks the magnetic field response, making accurate measurements of the magnetic signal difficult. However, the parasitic electric field can be eliminated by using the FM-KPFM feedback loop, which applies a compensating voltage to the probe, such that the probe-sample potential difference is zero at each pixel of the scan. Thus, performing SGM with \(\text{in situ}\) FM-KPFM feedback (Fig. 2) allows effective separation of the electrostatic from magnetic responses.
III. MODELING OF THE HALL DEVICE RESPONSE TO LOCALIZED MAGNETIC AND ELECTRIC FIELDS

A 2D finite element model has been developed to calculate the electric potential distribution and, thus, the transverse voltage in the graphene Hall device under the assumptions of diffusive transport regime and non-uniform magnetic field $B_{\text{probe}}(r)$. In the electrostatic potential equation, a spatially dependent conductivity tensor is introduced

$$\sigma(r) = \sigma(r) \frac{1}{1 + \left| \mu B_{\text{probe}}(r) \right|^2} \left[ -\frac{1}{\mu B_{\text{probe}}(r)} \right] \mu B_{\text{probe}}(r) \frac{1}{1}, \quad (1)$$

where $\sigma(r) = \mu n(r) e$, with $\mu$ being the electron mobility, $n(r)$ being the local electron density, and $e$ being the electron charge. The local magnetic field $B_{\text{probe}}(r)$ in Eq. (1) is the component of the probe stray field orthogonal to graphene sheet, determined from Green integral formulation. The probe magnetic coating is modeled by introducing a 3D spatial distribution of dipoles with uniform magnetization perpendicular to the sensor surface.

The probe-sample capacitive coupling is handled by describing $n(r)$ as a spatially dependent function

$$n(r) = n_0 + \frac{C_{\text{probe,max}}}{e} \left[ V_{\text{probe}} - V(r) \right] \exp \left[ -|r - r_0|^2/\lambda^2 \right], \quad (2)$$

where $n_0$ is the electron density in the absence of probe induced doping effects, $V(r)$ is the local voltage of the sample, $C_{\text{probe,max}}$ is the maximum local value of the probe-sample capacitance per unit area at the considered lift height, and $\lambda$ is a characteristic length scale describing the spatial decay of probe-sample capacitive coupling.

IV. EXPERIMENTAL RESULTS AND DISCUSSION

A. Magnetic scanning gate microscopy

First, we consider the case of standard SGM with a grounded metallic magnetic probe, i.e., FM-KPFM feedback is disabled. We study the response of graphene Hall devices to non-uniform magnetic fields introduced by a scanning custom-made probe with a 20-nm thick magnetic Co coating. A bias current of $I_{\text{bias}} = +70 \mu A$ (from source to drain in Fig. 3(a)) is driven across the 1-μm device and the transverse voltage $V_{xy} = V_B - V_A$ is measured and mapped at each point of the probe position. Fig. 3(a) shows the transverse voltage mapping when the probe is magnetized downwards and scanned at 0 nm lift height with respect to the sensor surface. For a finite electric field between the probe and sample, the dominating features in $V_{xy}$ mapping are peaks at the corners of the active sensing area (Fig. 3(a)). The image shows a two-fold symmetry, characterized by increase/decrease of the transverse voltage when the probe gates at the corners #1–3/2–4, respectively. A maximal positive/negative signal of $\sim$4.5 μV is measured at the respective corners (Figs. 3(a) and 3(e)). At the same time, a non-zero positive signal of $\sim$2 μV is measured at the center of the cross (Figs. 3(b) and 3(e)). The modeling approach described above (i.e., probe-sample capacitive coupling is included) confirms this behavior, as demonstrated by the calculated map of $V_{xy}$ reported in Fig. 3(c). Here, a maximum local area capacitance ($C_{\text{probe,max}} = 9900 \mu F/m^2$) with a spatial decay length ($\lambda$) of $\sim$60 nm was estimated by fitting the experimental scanning gate images.

When the current direction is reversed from $-70 \mu A$ to $+70 \mu A$ (from drain to source and vice versa), the voltage polarity flips at the corners, such that signal at the corners #1 and 3 changes from negative to positive (Figs. 4(a)–4(d)). The signal at the center of the cross also changes its polarity with the bias current (Figs. 4(a) and 4(b)). Fig. 3(f) shows that the transverse voltage at the center of the device (point A in Fig. 3(b)) increases almost linearly with the biased current.

The origin of the strong diagonal contrast can be understood by considering diversion of the flow of electrons...
towards \( V_A \) (\( V_B \)) electrodes when the electrically biased probe is scanned above corners #1 and 3 (#2 and 4) of the sensor, which results in a rise (drop) in \( V_{xy} \) (see Fig. 3(a)). It should be noted that due to the use of a unipolar current source, the surface potential of the sample is always higher than the grounded metallic probe, irrespective of the direction of the current. If we consider the case of \( I_{bias} < 0 \) (as experimentally shown in Fig. 4(a)), the lower potential of the probe (\( V_{probe} < V_{sample} \)) locally gates at corners #1 and 2 at bias current (e) and (f) \( I_{bias} < 0 \) and (g) and (h) \( I_{bias} > 0 \).

Next, we perform magnetic SGM mapping of the device with the FM-KPFM feedback loop turned on. As described in Sec. II, the feedback loop eliminates the electrostatic forces by applying a compensating DC voltage to the probe at each pixel, such that \( V_{probe} = V_{sample} \). When the electric potential between the probe and sample is nullified at every point in the scan, the measured transverse voltage \( V_{xy} \) originates only from the local magnetic field of the probe oscillating at \( f_0 \). The result of the elimination of the electrostatic interaction by enabling the FM-KPFM feedback is shown in Fig. 3(b), which demonstrates a complete suppression of peaks at the corners of the Hall cross. This is also confirmed by the calculated map of \( V_{xy} \), which has been computed neglecting probe-sample capacitive effects. In this case, the map demonstrates a “bell-like behavior” with maximum values at the cross center, indicating that the signal at the center has a prevalent magnetic origin (Fig. 3(d)). It should be noted that the maximum magnitude of the output signal is much lower after switching on the FM-KPFM feedback.

FIG. 4. SGM mapping of \( V_{xy} \) signal in 1-\( \mu m \) wide graphene Hall cross with bias current (\( I_{bias} \)) equal to (a) \(-70 \mu A\) and (b) \(+70 \mu A\). FM-KPFM feedback loop is disabled. White lines outline the contour of the device. (c) and (d) Line profiles obtained along the dashed lines indicated in (a) and (b), respectively. Schematic of the current distribution when the probe (\( V_{probe} < V_{sample} \)) locally gates at corners #1 and 2 at bias current (e) and (f) \( I_{bias} < 0 \) and (g) and (h) \( I_{bias} > 0 \).
compared to the initial experiment (Fig. 3(e)). The largest value of $V_{xy}$ is measured when the probe is at the center of the sensing area, which is a result of its maximum coupling to the probe stray field (Fig. 3(b)). The signal at the cross center is not affected by the inclusion of the FM-KPFM feedback.

As discussed above, magnetic and electrostatic forces have similar dependences on a number of experimental parameters, i.e., current, probe oscillation amplitude, and vertical separation. Investigations into the orientation and magnitude of the probe magnetization can provide unambiguous proof that carriers are affected by the Lorentz force. Thus, we perform mapping of the transverse voltage using an MFM probe magnetized down (Fig. 5(a)) and up (Fig. 5(b)) with the FM-KPFM feedback on. The $I_{bias}$ is fixed to $-70 \mu A$ and the distance between the probe apex and the sensor surface is 0 nm, i.e., effectively the probe is above the surface at a distance equal to the amplitude of probe oscillations. The results have been obtained using custom-made magnetic probes with low magnetic moment (20-nm Co coating). The maps in Figs. 5(a) and 5(b) show that the polarity of the signal at the center of the device corners (Figs. 7(a) and 7(b)), i.e., the decay length of the electrostatic signal is much smaller than the device size. Consequently, this result indicates that the 1-μm Hall device is significantly affected by electrostatic effects, which may notably decrease their performance as magnetic sensors. It is noteworthy, however, that in practice any electrically charged object in the vicinity of the device can play the role of a metal probe and affect the carrier flow in the device.

C. Effect of device size

Local distribution of the transverse voltage was mapped for Hall devices of different sizes, i.e., $w = 0.6–15\mu m$. In this case, the FM-KPFM feedback was kept disabled to better illustrate the spatial distribution of the electrostatic signal. Representative SGM images are shown in Fig. 7. It is noteworthy that the images were obtained at different values of $I_{bias}$, as smaller devices are potentially unstable at larger currents, whereas imaging of larger devices with low currents led to a small output signal.

As it can be seen from Figs. 7(c) and 7(d), small devices ($w = 1$ and 0.6 μm, respectively) are most affected by the electrostatic signal, as it becomes increasing difficult to differentiate the electrostatic and magnetic signal, which is a result of the overlapping of two distributions with peaks at the diagonal corners. As it has been previously shown for 1-μm device the maximum response of the magnetic signal is typically a few times weaker than its electrostatic counterpart (Fig. 3(e)). This indicates that small devices are significantly affected by electrostatic effects, which may notably decrease their performance as magnetic sensors. It is noteworthy, however, that in practice any electrically charged object in the vicinity of the device can play the role of a metal probe and affect the carrier flow in the device.

On the other hand, in large devices ($w = 10$ and 15 μm) the electrostatic component is relatively strongly localized at the device corners (Figs. 7(a) and 7(b)), i.e., the decay length of the electrostatic signal is much smaller than the device.
width. However, it is worth mentioning that the magnetic output of these devices is negligible even after FM-KPFM compensation (Fig. 7(e)). These results seem to be counterintuitive, bearing in mind a larger Hall coefficient and correspondingly lower minimum detectable field (Table I). A significant imbalance between the size of the sensor and magnetic object leads to a poor magnetic coupling and substantially decreases the output of the magnetic sensor. Ideal coupling is expected when the areas of the sensor and the object are compatible. 25 Thus, devices in this size range (tens of microns), while ideal for detection of low uniform magnetic fields, are not particularly suitable for measurements of small localized fields, i.e., for single (or small number) magnetic particle detection in biomedical applications.

V. CONCLUSIONS

Using magnetic SGM, we have mapped the distribution of the transverse voltage response in epitaxial graphene devices with width of 0.6–15 μm. The local transverse voltage has been measured with dependence on the magnetic probe position, correlated to the sample topography and studied by varying the magnitude and direction of the bias current as well as the probe-sample distance and orientation of the probe magnetization. We demonstrate that the strong signal, with a two-fold symmetry observed at the device corners, has an electrostatic origin caused by capacitive coupling of the charged probe with a biased device, whereas the weaker response in the middle of the cross is mainly determined by the local magnetic field of the probe. We show that the electrostatic effects are the most pronounced in small devices (<1 μm), where they affect the largest part of the sensor, thus decreasing the effective area of the sensor. Implementation of the FM-KPFM feedback loop in the standard magnetic SGM setup drastically reduces capacitive effects, improves magnetic sensitivity and expands the effective area of the device. The presented techniques provide a straightforward route for characterizing the device performance and evaluating the most suitable for a particular application.

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21See www.bruke.com for data specification about MESP and HM-MESP probes.