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Magnetic scanning probe calibration using graphene Hall sensor

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Magnetic force microscopy (MFM) offers a unique insight into the nanoscopic scale domain structures of magnetic materials. However, MFM is generally regarded as a qualitative technique and, therefore, requires meticulous calibration of the magnetic scanning probe stray field (B_{probe}) for quantitative measurements. We present a straightforward calibration of B_{probe} using scanning gate microscopy on epitaxial graphene Hall sensor in conjunction with Kelvin probe force microscopy feedback loop to eliminate sample-probe parasitic electric field interactions. Using this technique, we determined $B_{probe} \sim 70$ mT and ~ 76 mT for probes with magnetic moment $\sim 1 \times 10^{-13}$ and $> 3 \times 10^{-13}$ emu, respectively, at a probe-sample distance of 20 nm.

Index Terms— Epitaxial graphene, Hall sensor, Kelvin probe force microscopy, magnetic probe calibration.

I. INTRODUCTION

MAGNETIC force microscopy (MFM) is a well-established modification of atomic force microscope (AFM) technique for imaging of magnetic domains, allowing for effective mapping with nanoscopic spatial resolution. However, the technique is generally qualitative and further requires meticulous calibration of the stray magnetic field (B_{probe}) of the scanning probe for calibrated quantitative measurements [1], [2]. Microscopic Hall sensors are ideal for such probe calibration [3–5], in particular graphene-based Hall sensors benefit from high sensitivity (Hall coefficient, R_H) [6], and robustness to large biasing currents (I_{bias}) [7]. However, the electrostatic forces between the current-biased device and metal-coated probe gives rise to parasitic electric field [8], [9]. The resulting measurement of the transverse voltage (V_{xy}) is the superposition of the electric and magnetic field contributions, making it difficult to accurately determine B_{probe} .

We present B_{probe} calibration method that eliminates the parasitic electric field with the use of frequency-modulated Kelvin probe force microscopy (FM-KPFM) feedback loop [10]. This technique, performed in ambient conditions with a 1- μ m wide epitaxial graphene Hall sensor, effectively separates the electric field contributions, giving rise to V_{xy} signal solely due to B_{probe} .

II. SAMPLE FABRICATION

The epitaxial graphene was grown by sublimation of Si and subsequent graphene formation on the Si-terminated face of 4H-SiC(0001) substrate at 2000°C and 1 bar argon gas pressure. Details of the fabrication and structural characterization are reported elsewhere [11]. The high temperature annealing process results in a substantial number of atomic scale terraces on SiC (Fig. 1a). In the sample studied

here, the terraces are around a micron wide and do not affect the continuity of the graphene layer. The sample consists of $\sim 95\%$ one layer graphene (1LG) and $\sim 5\%$ double layer graphene (2LG); as revealed by large scale FM-KPFM mapping.

The electrodes and the Hall bars were defined by electron beam lithography in three independent steps. Oxygen plasma etching was used to pattern the Hall bars. Using this method, double Hall sensors with symmetric crosses of width ranging from 500 to 1000 nm were formed and studied at room temperature using magnetotransport and noise spectral measurements. Contact mode AFM was also used to clean the device of resist residues, as the resist is known to significantly affect the transport properties [12]. In the present study, 1- μ m wide cross epitaxial graphene device was used (Fig. 1a).

Fig. 1: (a) Sample morphology largely dominated by SiC terraces. The wavy lines are atomic scale steps in the SiC substrate occurring during the high temperature graphene growth. (b) Surface potential mapping using the FM-KPFM technique with device biased at $I_{bias} = 20$ μ A.

Fig. 4: Transverse voltage mapping of the sensor (at $I_{bias} = 20 \mu A$) showing significant parasitic electric field contribution when FM-KPFM feedback loop is disabled. MESP-HM probe was used.

nullification of the probe-sample electric field, the response of V_{xy} is a consequence of only the modulated B_{probe} at f_0 . Then the largest V_{xy} is measured when the probe is at the centre of the sensing area, which is a result of its maximum coupling to the probe stray field (Fig. 5). However, we still observe small peaks at the corners of the sensor (see e.g. Fig. 5c-d). These peaks are inevitably a result of only partial nullification of the probe-sample electric field. Nevertheless, signal from B_{probe} is generally unaffected and these parasitic signals have only a negligible effect on the data analysis.

Both types of magnetic probes were calibrated with forward ($\downarrow$) and reverse ($\uparrow$) polarities of the magnetizations, producing

Fig. 5: (a)-(d) Transverse voltage mapping of the sensor (at $I_{bias} = 20 \mu A$ with FM-KPFM feedback enabled) showing largely the magnetic contribution of B_{probe} for MESP and MESP-HM probes with forward ($\downarrow$) and reverse ($\uparrow$) magnetizations. (e) Line profiles for the configurations (a)-(d) obtained along the indicated dashed line in (a). Oscillation amplitude of $A_{osc} = 10$ -20 nm at f_0 .

a positive and negative response of V_{xy} , respectively. The maximum measured response of MESP probe was $V_{xy}(\downarrow) = 0.39 \mu V$ and $V_{xy}(\uparrow) = -0.37 \mu V$, while MESP-HM showed a larger response of $V_{xy}(\downarrow) = 1.08 \mu V$ and $V_{xy}(\uparrow) = -0.64 \mu V$ (Table I). The resulting line profiles (see Fig. 5e) across the centre of the device, as shown in Fig. 5a by the dashed black line, demonstrate a strong signal with a Lorentzian response for data sets in Fig. 5a, 5c and 5d. However, data for MESP-HM $\uparrow$ shows signs of V_{xy} saturation between the positions of 0.7 and 2 μm of the line profile (Fig. 5b). Saturation for MESP-HM $\uparrow$ cannot be explained by total encompassing of the stray field as it not observed in MESP line profiles, where the probe stray field is smaller than that of MESP-HM.

D. Modelling

In order to evaluate B_{probe} , micromagnetic simulations using OOMMF software were carried out for a Co coating MFM probe. The parameters used to simulate the 40 nm-thick polycrystalline cobalt layer are the following: saturation magnetization $M_s = 1400 \text{ kA/m}$, exchange stiffness $A = 3 \times 10^{-11} \text{ J/m}$, magnetocrystalline anisotropy is negligible and a cell size of 1 nm. The geometry of the probe has been modelled following the real shape of the MESP probes with a probe radius of 30 nm. After saturating the probe along the z direction, the equilibrium magnetization distribution was simulated in a remnant state. Then, the emerging B_{probe} was calculated in the surrounding volume. Fig. 6a shows the B_{probe} profiles produced by the probe at different probe-sample distances and fitted to a Lorentzian function. The FWHM values of the fitted curves are shown in inset Fig. 6b. Since the measured V_H values are proportional to the total B_{probe} , the integral of the B_{probe} has been calculated. However, since the magnetic field is not homogeneous across the area of the sensor (A_{cross}) [3], it is necessary to estimate the effective area of the probe (A_{eff}) and the effective stray field created by the probe (B_{effect}) to evaluate the V_H . The effective area is calculated taking the radius = FWHM/2. The effective stray field can be calculated assuming the B_{probe} spread homogeneously into the calculated A_{eff} . Notice the exponential decay of the calculated B_{effect} when the probe-sample distance increases (see Fig. 6b). In order to calculate V_H produced by the probe, we use the equation $V_H = R_H I_{bias} B_{effect} A$, where $R_H = 1250 \Omega/T$, $I_{bias} = 20 \mu A$ and $A = A_{eff} A_{cross}$.

Notice that the V_{xy} data measured experimentally corresponds to an AC value induced by the oscillation of the cantilever near the sensor. These calculated values have been evaluated for the experimental parameters, i.e. with the amplitude of oscillation of $A_{osc} = 10$ and 20 nm and probe-sample distances of 10 and 20 nm.

The micromagnetic simulation corresponding to MESP probes gives B_{probe} of 153 and 70 mT for probe-sample distances of 10 and 20 nm, respectively. The expected V_{xy} values for $A_{osc} = 10$ and 20 nm are 0.74 and 0.87 μV ,

TABLE I
SUMMARY OF PARAMETERS FOR MFM PROBES

Probe	A_{osc}	Experimental V_{xy}	Simulated V_{xy}
MESP	10/20 nm	$\sim 0.38/\text{NA} \mu V$	0.74/0.87 μV
MESP-HM	10/20 nm	0.64/1.08 μV	0.96/1.24 μV

Fig. 6: Micromagnetic simulations for a Co probe of (a) stray field distribution versus the probe-sample distance. (b) Evolution of the B_{effect} and V_H produced by the probe in the sensor with the distance. Inset: FWHM and B_{effect} versus the probe-sample distance.

respectively. Moreover, the simulations corresponding to the higher moment MESP-HM probes give a B_{probe} of 183 and 76 mT for probe-sample distances of 10 and 20 nm, respectively. The expected V_{xy} values for $A_{\text{osc}} = 10$ and 20 nm are 0.96 and 1.24 μV , respectively (Table I). The simulated responses of V_{xy} are in good agreement with the experimental results.

V. CONCLUSION

We successfully demonstrated calibration of B_{probe} using FM-KPFM feedback loop to separate the magnetic and parasitic electric field contributions. Using novel epitaxial graphene sensors allows us to improve coupling between the magnetic probe and sensor as well as to exploit the high stability of the graphene devices to larger biasing currents, which could be attributed to the unique properties of the material, i.e. carrier energy relaxation rate (or the energy loss rate for hot carriers). This is significantly higher in graphene due to much more effective electron-lattice interactions leading to emission of acoustic phonons. The described B_{probe} calibration technique is a simple first step towards calibration of the probe stray field and its gradient, at the same time the method is capable of estimating the effective probe radius. Using this technique, the experimentally determined V_{xy} for MESP and MESP-HM probes are in good agreement with the simulations.

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