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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 ($B_{\text{probe}}$) for quantitative measurements. We present a straightforward calibration of $B_{\text{probe}}$ 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 $B_{\text{probe}} \sim 70$ mT and $\sim 76$ mT for probes with magnetic moment $\sim 10^{13}$ and $\times 10^{13}$ 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 ($B_{\text{probe}}$) 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 ($I_{\text{bias}}$) [7]. However, the electrostatic forces between the current-biased device and metallically 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 $B_{\text{probe}}$.

We present $B_{\text{probe}}$ 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-µm wide epitaxial graphene Hall sensor, effectively separates the electric field contributions, giving rise to $V_{xy}$ signal solely due to $B_{\text{probe}}$.

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 ~95% one layer graphene (1LG) and ~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-µm 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 $I_{\text{bias}} = 20$ µA.](image-url)
III. EXPERIMENTAL METHOD

Single pass FM-KPFM utilizes tapping mode AFM and AC/DC voltages to map the sample morphology and surface potential, respectively. The difference in potential between the probe and the current biased sample gives rise to the electrostatic force

\[ F = \frac{dC V^2}{dz} \]

where \( C \) and \( z \) are probe-sample capacitance and spacing, \( V_{\text{probe}} \) and \( V_{\text{mod}} \) are the DC and AC components of the voltage applied to the probe and \( V_{\text{CPD}} \) is the contact potential difference [10]. The topography is measured at the mechanical resonance frequency (\( f_0 \sim 300 \) kHz with an oscillation amplitude set point of 10-20 nm), while a low frequency AC voltage (\( V_{\text{mod}} = 5 \) V at \( f_{\text{mod}} = 2 \) kHz) applied simultaneously to the electrically conductive probe. \( V_{\text{mod}} \) gives rise to sideband resonances of frequencies \( f_0 \pm f_{\text{mod}} \) produced by oscillating electrostatic force gradient (\( dF/dz \)), which is expressed as

\[ f_0 \pm f_{\text{mod}} \approx f_0 \left(1 - \frac{1}{2k} \frac{dF}{dz}\right), \]

where \( k \) is the spring constant. The FM-KPFM feedback loop minimises the sidebands by applying \( V_{\text{probe}} \) such that \( V_{\text{probe}} - V_{\text{CPD}} = 0 \), where \( V_{\text{CPD}} \) is the contact potential difference, therefore eliminating the electric field between the probe and sample. Recording \( V_{\text{probe}} \) pixel by pixel provides the mapping of the surface potential (Fig. 1b), which can ultimately be used to determine the work function of the sample [13].

The experimental method consisted of scanning the current biased (\( I_{\text{bias}} = 20 \) µA) Hall sensor with FM-KPFM technique using conductive magnetic scanning probes, while simultaneously measuring \( V_y \) with an external Stanford Research SR830 lock-in amplifier, referenced to the mechanical oscillation of the cantilever (Fig. 2). Due to the finite potential of the biased sensor (~1 V), the grounded probe acts as a local scanning gate that couples capacitively to the sample. The FM-KPFM feedback loop accurately accounts for the surface potential, hence eliminating electrostatic forces between the probe and sensor, resulting in the measurement of only the magnetic contribution of the probe.

Using the described method, \( V_y \) was mapped using two types of magnetic probes with different thickness of Co/Cr coating: MESP and MESP-HM (Bruker) probes with coercivity ~400 Oe and the moment \( m \sim 1 \times 10^{-13} \) and >3x10\(^{-13}\) emu, respectively.

IV. RESULTS AND DISCUSSIONS

A. Transport Measurements

Transport and noise measurements were performed to fully characterise the Hall sensor in ambient conditions, which will be used to calibrate the stray field of the magnetic probes. The sensitivity of the sensor was determined by sweeping the DC magnetic field (\( B \)) up to ~0.55 T, while simultaneously measuring the Hall voltage (\( V_H \)) for bias currents of \( I_{\text{bias}} = 10, 30 \) and 50 µA (Fig. 3). The Hall coefficient \( (R_H = V_H/I_{\text{bias}}B) \) was determined giving an average of \( R_H = 1250 \) \( \Omega \) T. The finite \( V_H \) offset at zero fields can be a result of misaligned voltage leads and/or non-uniform flow of the carriers due to material inhomogeneities. The conduction in epitaxial graphene occurs through electrons (n-type). The measured carrier density \( n_e = 5 \times 10^{11} \) cm\(^{-2}\) and mobility \( \mu = 1500 \) cm\(^2\)/Vs are comparable to other published work [6], [14], [15]. The noise spectral density at \( I_{\text{bias}} = 20 \) µA reveals a noise floor of \( S_n \sim 40 \) nV at \( f_0 = 80 \) kHz, leading to a minimum detectable field of \( B_{\text{min}} \sim 1.6 \) µT.

B. Scanning Gate Microscopy

First, we consider the case of standard scanning gate microscopy (SGM) with a metallic magnetic probe, i.e. where FM-KPFM feedback is disabled. For a finite electric field between the probe and sample, the dominating features in mapping of \( V_y \) are peaks at the corners of the active sensing area (Fig. 4). Consider the case where the probe is gating above corner 1 and 4 (corner 2 and 3) of the sensor, the flow of electrons is diverted towards \( V^+ \) (\( V^- \)), resulting in a drop (rise) in \( V_y \). In essence, these features are identical to those observed in SGM experiments and are well documented [9], [16].

C. Magnetic Probe Calibration

Next, we perform SGM mapping of the device with the FM-KPFM feedback loop enabled. In the ideal case of total
nullification of the probe-sample electric field, the response of $V_{xy}$ is a consequence of only the modulated $B_{\text{probe}}$ 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 $B_{\text{probe}}$ 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 (↓) and reverse (↑) polarities of the magnetizations, producing 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, date for MESP-HM ↑ 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 ↑ 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 $B_{\text{probe}}$, 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 $M_s = 1400$ kA/m, exchange stiffness $A =$ 3×$10^{-11}$ 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 $B_{\text{probe}}$ was calculated in the surrounding volume. Fig. 6a shows the $B_{\text{probe}}$ 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 $V_{xy}$ values are proportional to the total $B_{\text{probe}}$, the integral of the $B_{\text{probe}}$, has been calculated. However, since the magnetic field is not homogeneous across the area of the sensor ($A_{\text{cross}}$) [3], it is necessary to estimate the effective area of the probe ($A_{\text{eff}}$) and the effective stray field created by the probe ($B_{\text{effect}}$) to evaluate the $V_{xy}$. The effective area is calculated taking the radius = FWHM/2. The effective stray field can be calculated assuming the $B_{\text{probe}}$ spread homogeneously into the calculated $A_{\text{eff}}$. Notice the exponential decay of the calculated $B_{\text{effect}}$ when the probe-sample distance increases (see Fig. 6b). In order to calculate $V_{xy}$ produced by the probe, we use the equation $V_{xy} = R_H I_{\text{bias}} B_{\text{effect}} A$, where $R_H = 1250$ Ω/T, $I_{\text{bias}} = 20$ μA and $A = A_{\text{eff}}/A_{\text{cross}}$.

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 $A_{\text{osc}} = 10$ and 20 nm and probe-sample distances of 10 and 20 nm.

The micromagnetic simulation corresponding to MESP probes gives $B_{\text{probe}}$ of 153 and 70 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 0.74 and 0.87 μV.

<table>
<thead>
<tr>
<th>Probe</th>
<th>$A_{\text{osc}}$</th>
<th>Experimental $V_{xy}$</th>
<th>Simulated $V_{xy}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>MESP</td>
<td>10/20 nm</td>
<td>-0.38/NA μV</td>
<td>0.74/0.87 μV</td>
</tr>
<tr>
<td>MESP-HM</td>
<td>10/20 nm</td>
<td>0.64/1.08 μV</td>
<td>0.96/1.24 μV</td>
</tr>
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