Single layer graphene Hall sensors for scanning Hall probe microscopy (SHPM) in 3–300 K temperature range

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ARTICLE INFO

Article history:
Received 18 March 2014
Accepted 28 April 2014
Available online xxx

Keywords:
Scanning Hall probe microscopy
Graphene
Graphene Hall sensor
Magnetic imaging

ABSTRACT

Graphene micro-Hall probes were developed for a scanning Hall probe microscope system and used for the direct magnetic imaging domains of demagnetized NdFeB permanent magnet for the first time. The Hall coefficient and minimum magnetic field resolution of graphene Hall probes at 1kHz were found to be 0.18 Ω/G and 0.20 G/√Hz for a drive current of 3 μA at room temperature in vacuum. The magnetic domains in NdFeB demagnetized magnet were observed at 300 K, 126 K and 3 K successfully.

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1. Introduction

Scanning Hall probe microscopy (SHPM) belongs to scanning probe microscope (SPM) family, which provides quantitative and non-invasive local magnetic field imaging of magnetic and superconducting materials [1]. It provides high magnetic field and spatial resolution, simultaneously with the topography of the sample surface [2,3]. The most important element of SHPM is the Hall sensor which is sensitive to the perpendicular component of the magnetic field on the specimen. Hall sensors can be fabricated from various materials such as GaAs/AlGaAs heterostructure, Bi, InSb and graphene. GaAs/AlGaAs two-dimensional electron gas heterostructure is one of the most popular materials for Hall probe production, but the minimum size is limited to ~200 nm with this sensor because of surface depletion effect, which result in low spatial resolution [4]. In Bi Hall probes, the spatial resolution was decreased up to 50 nm, on the other hand minimum detectable magnetic field was increased due to low mobility of Bi film [3]. In order to overcome this problem, InSb has been used as sensing element. It has high mobility (55,500 cm²/Vs) at room temperature and low carrier concentration. It was reported that minimum detectable magnetic field was 6–10 mG/√Hz at 50 μA drive current in InSb Hall sensor with a size of 1.5 μm [5]. After the discovery of high mobility in graphene, it is considered to be a good candidate for Hall probe material [6,7]. Hall sensors have been fabricated by epitaxial growth graphene and it has been demonstrated that minimum detectable magnetic field of epitaxial graphene Hall sensors was 39 mG/√Hz which is comparable with similar sized InSb Hall sensors [8]. Meanwhile, graphene produced by chemical vapor deposition (CVD) method has been used as Hall probes. It has been shown that it is a promising material for Hall probes in SHPM applications thanks to its commensurable characteristics to conventional Hall probes [9]. However, utilization of GHP for magnetic imaging has not been shown in the literature yet.

In this paper we represent fabrication, characterization and performance of graphene based micro–Hall devices for low temperature scanning Hall probe microscopy (LT–SHPM). Graphene produced by CVD method was preferred in order to increase the production yield of the fabricated Hall sensors. In this work, for the first time, we used GHP integrated with Quartz Tuning Fork for imaging localized magnetic field of NdFeB in a wide temperature range of 3–300 K.

2. Experimental

2.1. Device fabrication

Graphene Hall probes have been fabricated using commercially available 1 cm × 1 cm CVD single layer graphene on 285 nm SiO₂/Si
Fabrication process flow of GHPs is described as follows and the optical microscope images of the fabrication steps of device are shown in Fig. 1: (i) optical lithography was used to define Hall cross patterns which were protected by photoresist. (ii) This was followed by oxygen gas plasma reactive ion etching (RIE) (20 sccm O₂ flow, in a 100 W RIE power, 37.5 mTorr, for 9 s) in order to etch the unprotected parts of CVD-graphene sheet (Fig. 1a). (iii) Second optical lithography was used to pattern for contact pads on graphene, and it was followed by thermal evaporation of 10 nm Cr/200 nm Au evaporation and lift-off (Fig. 1b). (iv) The mesa which serves as AFM tip was obtained by etching SiO₂/Si wafer ~830 nm in an inductively coupled plasma reactive ion etching (ICP–RIE) system (Fig. 1c and supplementary material [19]). (v) GHP patterns on the wafer were diced into individual chips and glued with low temperature epoxy on one of the tines of the quartz tuning fork (QTF) which is used as a force sensor in a wide temperature range. QTF was glued to the ceramic plate on non-magnetic printed circuit board (PCB). (vi) Finally, electrical connections of GHP to PCB were established with 25 μm gold wires using wedge wire bonder (Fig. 1d).

3. Results and discussion

3.1. Characterization of graphene Hall probe

We used a Renishaw inVia Reflex Raman Microscope (532 nm excitation wavelength) to determine the number of graphene...
Fig. 3. (a) Hall voltage response of GHP to applied magnetic field at room temperature. (b) $B_{\text{min}}$ for different drive current as a function of frequency at 300 K. (c) $B_{\text{min}}$ for different temperature as a function of frequency for 5 μA drive current.
layers and uniformity of graphene thickness over the GHP surface. Raman spectrum of the center of Hall Cross which is shown in Fig. 2(a) verifies that the GHP was fabricated from single layer graphene and the insert shows optical microscope image of fabricated sensor. Moreover, it was reported that $I_{2D}/I_G \geq 2$ is associated with the presence of monolayer graphene [10–12]. In Fig. 2(b), large scale (14 $\mu$m $\times$ 14 $\mu$m) Raman map of intensity ratio gives further evidence.

Electrical and magnetic characterization of GHP was performed in Helium exchange gas and vacuum conditions by using LT–SHPM

![Image](image_url)

**Fig. 4.** (a) Magnetic image of NdFeB demagnetized magnet by using GHP at 300 K. (b) Magnetic field variation along the line drawn on image.

![Image](image_url)

**Fig. 5.** (a) Topographic image of NdFeB. (b) 50 $\mu$m  $\times$ 50 $\mu$m LT–SHPM magnetic image of NdFeB at 126 K. 14 $\mu$m  $\times$ 14 $\mu$m LT–SHPM magnetic image of the same sample for. (c) $I_{\text{Hall}} = +2$ $\mu$A. (d) $I_{\text{Hall}} = -2$ $\mu$A at 3 K.
initially at room temperature. Graphene exhibits p-type behavior due to environmental effects, water absorption when it is exposed to air and resist residues from fabrication process [13–16]. This results in a high charge carrier density and low Hall coefficient. Thereby, we tried to overcome this problem by performing magnetic imaging and characterization in vacuum condition as we did not have a back gate contact. A uniform external magnetic field ($B_{ext}$) was applied to GHP up to 5000 G and the generated Hall voltage ($V_{Hall}$) was simultaneously measured under constant driving current. Fig. 3(a) shows the linear relationship between $V_{Hall}$ and $B_{ext}$ for a 2 μA driving current at room temperature. Room temperature series resistance and Hall coefficient of GHP were measured to be 82.20kΩ and 0.18 ΩG, respectively in vacuum. A spectrum analyzer was used to obtain a noise spectrum of the single layer graphene Hall probe. We determined minimum detectable magnetic field ($B_{min}$) from voltage noise spectrum, which is defined as

$$B_{min} = \frac{V_{Noise}}{R \cdot I_H \cdot G}$$

where $V_{Noise}$, $R_H$, $I_H$ and G are the total measured voltage noise, Hall coefficient, Hall current, and Hall probe pre-amplifier gain respectively [17]. The Hall probe pre-amplifier gain was 1001 for the system used in this experiment. Fig. 3(b) represents $B_{min}$ as a function of frequency for different Hall currents in zero magnetic field and zero back gate voltage. As seen from this figure, $B_{min}$ decreases with increasing drive current and $B_{min}$ is found to be 0.20G/√Hz for a 3 μA drive current at 1 kHz. We also measured the magnetic field noise of GHP at 5 K, 77 K and 300 K for $I_H = 5 \mu$A and calculated Johnson noise levels for each temperature for the same current value (Fig. 3(c)). Smaller magnetic fields can be detected at lower temperatures by GHP, as shown in Fig. 3(c).

3.2. Imaging NdFeB demagnetized magnet

We operated a LT–SHPM system manufactured by NanoMagnetics Instruments Ltd. in AFM tracking mode, in order to acquire topography and magnetic image of a NdFeB demagnetized magnet surface in a wide range of temperature (3–300 K). Resonance frequency of QTF used in this experiment was 32.768 Hz when one prong is free and the other is fixed to the ceramic plate on top of sensor holder PCB. After GHP was glued on top of QTF, this resonance frequency decreased to approximately 17 kHz due to mass of chip and glue (supplementary material) [19]. The sample is tilted ~1° with respect to GHP to ensure that the corner of Hall sensor mesa is the closest point to the sample surface, which is used as an AFM tip. The sample was brought in to close proximity of GHP by means of slip–stick coarse approach mechanism. The tip–sample interaction results in a shift in resonance frequency of the QTF which is measured by a Phase Locked Loop (PLL) for AFM feedback [18]. Fig. 4(a) shows the SHPM image of magnetic domains of NdFeB demagnetized magnet at room temperature. The magnetic field variation along a horizontal line is also shown in Fig. 4(b).

We have also investigated the performance of GHP at cryogenic temperatures. Surface topography and magnetic image of NdFeB sample for $I_{Hall} = 2 \mu$A at 126 K are shown in Fig. 5(a) and (b), respectively. Fig. 5(c) and (d) shows the SHPM image of the same sample at 3 K for $I_{Hall} = 2 \mu$A and $I_{Hall} = -2 \mu$A.

4. Conclusions

In this work, we have microfabricated CVD grown single layer graphene Hall sensors with the Hall coefficient and field sensitivity of 0.18 ΩG and 0.20 G/√Hz, respectively, for a 3 μA drive current at room temperature. We have successfully used the graphene Hall probes for magnetic imaging in 3–300 K range for the first time in SHPM. This study has demonstrated that graphene is an alternative material to be used for magnetic. Additionally, it is also possible to decrease Hall cross dimensions to a few tens of nanometer by employing electron beam lithography, so that spatial resolution of the sensor can potentially reach sub-100 nm resolution. Currently, we are working on the improvement of our results by reducing Hall cross area and removing residues arising from the fabrication process for both higher magnetic field and spatial resolution.

Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.apusc.2014.04.191.

References

[19] See supplementary material for further information for SiO2/Si etching and resonance frequency of QTF.