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Induced ferromagnetism in multilayered graphene in proximity with CoFe$_2$O$_4$

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Composites of anisotropic diamagnetic multilayer Graphene (MLG) and ferrimagnetic CoFe$_2$O$_4$ (CFO) nanoparticles (NPs) were synthesized through a one-step sonication process in the presence of a surfactant. The samples were characterized at ambient using x-ray diffraction (XRD), scanning electron microscopy (SEM), atomic force microscopy (AFM), Raman spectroscopy, magnetic force microscopy (MFM) and magnetometry (vibrating sample magnetometer). An induced ferromagnetism was observed in MLG with saturation magnetization $M_S = 18$ emu/g(MLG). This induced ferromagnetism is attributed to ferrimagnetic CFO uniformly distributed on the surface of diamagnetic MLG. SEM images confirm uniform dense distribution of CFO nanoparticles on MLG. MFM images confirm the formation of larger magnetic domains in the composite sample of MLG/CFO. The measured coercivity $H_C = 3.6$ Oe of MLG/CFO composite is much smaller than $H_C = 32.4$ Oe of pristine CFO NPs; this difference in $H_C$ is attributed to the effect of change in surface anisotropy. © 2018 Author(s). All article content, except where otherwise noted, is licensed under a Creative Commons Attribution (CC BY) license (http://creativecommons.org/licenses/by/4.0/). https://doi.org/10.1063/1.5003035

I. INTRODUCTION

Cobalt Ferrite (CoFe$_2$O$_4$) with inverse spinel structure has shown unique magnetic, magneto-optical, and magnetic-resistive properties that can be used in various applications such as magnetic recording media, magnetic fluids, and magnetic resonance imaging.$^{1-3}$ Spinel ferrites have a close-packed fcc structure containing 8 formula units of AB$_2$O$_4$ in the unit cell, where A and B represent the metallic ions located at the 8 tetrahedral A-sites and 16 octahedral sites B-sites respectively in the normal spinel structure. The presence of oxygen helps in the formulation of ionic bonding in all types of oxide ferrites$^{4,5}$ and provides path for superexchange coupling between the metallic ions. Cobalt ferrite (CFO) with the chemical formula of CoFe$_2$O$_4$ has inverse spinel structure in which the unit cell containing 8 formula units of CFO has half of the Fe$^{3+}$ cations are located at the 8 tetrahedral sites whereas 16 octahedral sites are equally occupied with Fe$^{3+}$ and Co$^{2+}$ cations.$^6$ The ferrimagnetism of CFO is due to the ordered Co$^{2+}$ ions on the B-sites since Fe$^{3+}$ moments on the A and B sites are antiferromagnetically aligned and cancel each other below its ordering temperature $T_C = 693$ K. Among the ferrites, bulk CFO is a hard magnetic material with moderate magnetization and high coercivity $H_C$.$^{1,7}$ Such unique magnetic properties along with the physical and chemical stability makes CFO a good candidate for technological applications$^5$ such as magnetic memory storage.$^8$

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During the last six decades, silicon-based devices have dominated the electronics industry. However, their performance improvements are coming to a limit and so the semiconductor industry is looking for suitable replacements. Graphene with a zero band-gap has emerged as a potential replacement.\textsuperscript{9–11} Since its discovery in 2004, graphene has attracted huge attention towards its application in device engineering mainly based on the high carrier mobility observed in this material.\textsuperscript{12–16} There are numerous research activities around the world directed to use graphene as a viable replacement of silicon.\textsuperscript{11,17} Graphene based hybrid materials have shown many advantages such as excellent mechanical and electrical properties.\textsuperscript{18,19}

In this paper, we present magnetic properties of a composite of multilayer graphene (MLG) with ferrimagnetic CFO. Several samples of MLG/CFO nanocomposites were prepared using a unique solvent method to integrate MLG into CFO homogenously through fluids at the nanoscale. In these hybrid structure, enhanced magnetic properties were observed.\textsuperscript{20–23} In this work, MLG/CFO nanocomposite were synthesized using the above one-step solvent method and structurally characterized using X-ray diffraction (XRD), atomic force microscopy (AFM), scanning electron microscopy (SEM), scanning transmission electron microscopy (STEM), Raman spectroscopy and transmission electron microscopy (TEM). For measuring their magnetic properties, vibrating sample magnetometry (VSM) and magnetic force microscopy (MFM) are employed. Details of these procedure, results and their interpretation/discussion are presented below.

\section*{II. SAMPLE PREPARATION}

MLG (Cheap Tubes Inc.), sodium dodecylbenzene sulfonate (SDBS) surfactant, and CFO nanoparticles (Sigma-Aldrich) were used as received. Ultra-sonication was performed using a Branson 450 Digital Sonifier with a \(\frac{1}{2}''\) disrupter horn. Sample preparation was initiated by dispersing 3.75 g of SDBS into deionized (DI) water of resistivity 18 M\textohm\textperiodcentered cm and sonicated for 20 minutes until a clear solution was achieved. Then 0.5 g of MLG was added into this solution and sonicated for additional 20 minutes. Next, 0.25 g of CFO was added to this solution and sonicated further for 40 minutes. The solution was filtered and dried inside a vacuum oven (pressure of 15 inches of mercury) at 80 \(^\circ\)C for 10 hours. The composite sample so obtained has the mass ratio of 15:2:1 for the SDBS/MLG/CFO components and this information is used later in analyzing the magnetic data. The samples of as-received pristine CFO, MLG and the composite sample of SDBS/MLG/CFO were structurally characterized followed by magnetic measurements as described below.

\section*{III. STRUCTURAL CHARACTERIZATION}

X-ray diffraction (XRD) measurements of the samples were performed at room temperature using a Rigaku MiniFlex diffractometer (Cu K\textsubscript{α} source, \(\lambda = 0.154185\) nm). The XRD patterns of samples of pristine CFO and the SDBS/MLG/CFO composite are shown in Figure 1. Major peaks of CFO at 2\(\theta\) = 30.2\(^\circ\), 35.7\(^\circ\), 43.45\(^\circ\), 53.50\(^\circ\), 57.55\(^\circ\), and 63.05\(^\circ\) corresponding to the (220), (311), (400), (422), (511), and (440) reflections respectively are observed in both samples. All the observed peaks of CFO are in agreement with reported values in the literature\textsuperscript{7,24} as well as with JCPDS card#22-1086. The sharp peaks of CFO nanoparticles demonstrate the good crystallinity of CFO cubic structure in both samples indicating that crystallinity has not been destroyed during the preparation of the SDBS/MLG/CFO sample. The prominent carbon peak is also clearly observed in SDBS/MLG/CFO sample around 2\(\theta\) = 26\(^\circ\) which corresponds to the diffraction from (002) plane of MLG. The other less prominent peak of MLG around 2\(\theta\) = 42\(^\circ\) corresponding to the (100)/(101) plane\textsuperscript{25} is interfered by the stronger (400) peak of CFO and so not observed. No Bragg peak from the surfactant SDBS are expected or observed since it is amorphous. Therefore, for reasons of brevity, the SDBS/MLG/CFO composite is simply listed as MLG/CFO in the rest of the paper. Within our experimental uncertainties, no other peaks due to any impurities could be detected suggesting proper dispersion of carbonaceous matrix into CFO nanoparticles.

Surface morphology of the MLG/CFO composite was characterized using scanning electron microscopy (SEM, Hitachi S-5500) and transmission electron microscopy (TEM, JEOL JEM 2100F). For this purpose, the composite was dispersed in isopropanol (IPA) and the solutions were diluted
FIG. 1. XRD patterns of pristine CFO and the SDBS/MLG/CFO composite.

Further using Dimethyl formamide (DMF). SEM image of MLG/CFO nanocomposite in Fig. 2(a) shows a dense and uniform distribution of CFO nanoparticles on MLG. The particle dimensions were measured using SEM taking an average of 10 measurements for CFO and MLG. The average particle size for CFO nanoparticles is 42 nm whereas the average flake size measured for MLG is 74 nm. Figure 2(b) shows the equally spaced lattice fringes separation of the MLG and CFO nanoparticles. The lattice fringe separation is 0.32 nm for MLG and 0.23 nm for CFO nanoparticles corresponding to the inter-planar distances.

For atomic force microscopy (AFM) measurements, MLG/CFO solution sample was drop-cast on a solvent-cleaned and air-dried Si substrate. AFM (AFM-Workshop TT-AFM model) with a single crystal silicon tip (Sensa Probes) was used. The cantilever was coated with Al on the reflex side and was tuned at 184.5 kHz natural frequency before scanning the sample. Figure 3(a) below shows the

FIG. 2. (a) SEM image of MLG/CFO nanocomposite (b) HR-TEM image of lattice fringes of MLG/CFO nanocomposite.

FIG. 3. (a) AFM images of the CFO nanoparticles on a Si substrate (b) AFM images of the MLG-CFO nanocomposites on a Si substrate.
AFM images of CFO nanoparticles and MLG/CFO nanocomposites on a Si substrate. Figure 3(b) further verifies uniform distribution of CFO nanoparticles onto the MLG.

IV. MAGNETIC MEASUREMENTS

Magnetic measurements were carried out using a vector magnetometer model 10 VSM system (MicroSense Company) equipped with a 30 kOe electromagnet. Magnetization ($M$) versus applied magnetic field ($H$) hysteresis measurement at room temperature for $H$ ramped up to 15 kOe in steps of 5 Oe, were made on pristine CFO and the MLG/CFO and the results are shown in Figure 4.

The as-synthesized composite sample consists of surfactant (SDBS), CFO, and MLG. The surfactant is only weakly diamagnetic and the pristine sample of as-received MLG is diamagnetic with magnetic susceptibility $\chi = -1.28 \times 10^{-5}$ emu/gm-Oe. Therefore, we make the reasonable assumption that the measured $M$ in the composite is due to MLG and CFO. The $M$ vs. $H$ data in pristine CFO in Fig. 4(a) gives contribution from CFO alone whereas the $M$ vs. $H$ data in Fig. 4(b) has contributions to $M$ from CFO and MLG in the composite. Therefore, the difference between the two plots in Fig. 4 can be used to determine the contribution of MLG in the composite to $M$ after normalizing with the mass of each component of the composite. As noted in Section II, the composite has the mass ratio of 15:2:1 for the SDBS/MLG/CFO components, giving the mass ratio of 1/6 for (MLG + CFO)/(composite) and mass ratio of 1/18 for CFO/composite. In Fig. 4(b), we have plotted $M$ normalized to the mass of (MLG + CFO) in the composite since SDBS has no significant contributions to $M$.

For the pristine CFO, the saturation magnetization $M_S = 63$ emu/gm(CFO) and the coercivity $H_C = 32$ Oe. For the composite, the measured $M_S = 99.4$ emu/gm(CFO+MLG) and $H_C = 3.6$ Oe. Making the reasonable assumption that the magnitude of $M_S$ for CFO in the composite is the same as for pristine CFO, the enhancement of $M_S$ in the composite is attributed to acquired ferromagnetism of MLG because of its proximity to CFO in the composite. This acquired ferromagnetism of MLG leads to $M_S = (99.4-63)/2 = 18.2$ emu/gm(MLG) since the mass ratio of MLG/CFO =2 in the composite. This is the major result of this paper.

The low field portion of the hysteresis loop, inset in Fig. 4(a), show step-like features. This could be due to the nano size of the CFO nanoparticles. However, the inset in Fig. 4(b), the low field portion of the hysteresis loop, shows a smoother increase as compared to that observed in pristine CFO. This smoothening of the low-field portion of the hysteresis loop of the composite compared to pristine CFO is attributed to the uniform distribution of CFO nanoparticles on the MLG.

MFM (AFM-Workshop Company model TT-MFM) with a magnetic tip from AppNano was used for magnetic force measurements. The silicon tip was magnetically coated on both sides with CrCo (Chromium Cobalt) with a resonance frequency ranging between 47–76 kHz. The tip was further magnetized for few minutes before mounting on the MFM head. The MFM scan was performed in non-contact mode. The tip lift for best resolution was found to be 50 nm to scan the magnetic domain images. The interaction between the tip and the sample is detected by retracting the tip position. Figures 5(a) and 5(b) depict MFM images of CFO nanoparticles and MLG/CFO nanocomposites.

FIG. 4. (a) Hysteresis loop of pristine CFO. (b) Hysteresis loop of the MLG/CFO composite. The insets in both graphs show the low field portion of the hysteresis loops.
Raman spectroscopy is a highly sensitive tool to investigate lattice effects such as local cation migration, lattice distortion and spin-lattice coupling. Therefore, Raman spectroscopy here was used to probe these magnetic samples deposited on Si substrate in order to understand the effect of CFO on MLG. This characterization was carried out using a Raman Spectrometer (Horiba LabRAM HR Evolution) which uses a grating with 1800 lines/mm and laser exciton wavelength of 514 nm. Figure 6 shows Raman spectra of the MLG/CFO nanocomposites. As expected, the Si substrate peak was observed at 520 cm$^{-1}$. The three major graphene peaks corresponding to D-band, G-band, and 2D-band are also observed. All the other observed peaks in Fig. 6 are identified with CFO present in the CFO.

### V. DISCUSSION

The XRD spectra of the CFO nanoparticle and the MLG/CFO nanocomposites show all the prominent peaks for CFO in both the samples and two carbon peaks in the MLG/CFO nanocomposite. The positions of these peaks are in good agreement with JCPDS data. The sharpness of CFO peaks indicates good crystallinity of CFO in the samples. The SEM image in Figure 2(b) is consistent of the inter-planar distance between two (220) planes of cobalt ferrite. The saturation magnetization for the pristine CoFe$_2$O$_4$ ($M_s = 63$ emu/g) reported here is consistent with the values reported in literature. For the MLG/CFO composite, $M_S = 99.4$ emu/g (CFO+MLG) is determined which leads to $M_S = 18$ emu/g (MLG) using $M_s = 63$ emu/g (CFO) and 2:1 weight ratio of the MLG:
CFO in the composite. This significant value of $M_S$ for MLG in the composite is attributed to proximity-induced ferromagnetism from the CFO nanoparticles uniformly spread out on MLG in the MLG/CFO nanocomposite. The MFM image shown in Figure 5(a) show different magnetic domains indicated by two different colors, yellow and black for pristine CFO nanoparticles. Figure 5(b) show larger domains compared to pristine CFO because of uniform site distribution of CFO in the composite.

The G-band observed in the Raman spectra in Figure 6, is the outcome of doubly generated phonon mode at the Brillouin zone center from the first order Raman scattering. D-band involves one phonon and one defect whereas 2D-band involves two phonons, both D-bands occur near the $K$- point out of second order Raman scattering. Surprisingly, the intensity of 2D-band is relatively larger compared to the intensity of G-band, indicating the absence of multi-layer graphene in the nanocomposite sample. The disorder in graphene sheets can be calculated by the ratio ($I_D/I_G$) of intensities of D-band and G-band. The $I_D/I_G$ ratio for the nanocomposite sample is 0.6 whereas pristine graphene is 0.99. The degree of disorder in the graphene sheets is caused by the additional defects in sp$^2$ bonds of graphene during the nanocomposite sonication process. However, these defects are a favorable site for attaching CFO nanoparticles to the graphene sheet. The extended duration of sonication time will further break sp$^2$ bonds which cause the loss of anisotropic properties in graphene sheets in the defect free graphene sheets. The duration of sonication time will also cause breaking of graphene layers which is evident here in the absence of multi-layer graphene. The location of D-band, G-band, and 2D-band are at 1363 cm$^{-1}$, 1607 cm$^{-1}$, and 2883 cm$^{-1}$ respectively whereas the original position as predicted in the theory for these bands are 1350 cm$^{-1}$, 1582 cm$^{-1}$, and 2700 cm$^{-1}$. The shift of these bands is the likely result of additional breaking of carbon-carbon bonds during the sonication process.

Coated cobalt ferrite has a cubic mixed ferrite structure with $O^{7_h} (Fd\bar{3}m)$ space group which gives rise to 39 vibrational modes. Out of these vibrational modes, five modes viz. $1A_1g$, $1E_g$, $3T_2g$ modes are Raman active. The five active Raman modes for the cubic inverse spinel ferrite are located at 186, 312, 477, 1330, 1580 cm$^{-1}$. In Figure 6, four peaks are observed which have randomly shifted from their original location. The observed peaks are located at 279, 486, 711, 990, and 1086 cm$^{-1}$. The shifting of Raman peaks for CFO nanoparticles can be attributed to the redistribution of Co$^{2+}$ ions in the cubic inverse spinel structure. However, laser exciton energy and substrate used can also cause peak shift in Raman spectra. In pristine CFO annealed under the same condition, these shifts were not observed.

VI. CONCLUSIONS

In conclusion, we report here enhanced magnetic property for MLG when uniformly coated with CFO nanoparticles. Various characterization tools such as SEM, AFM, HR-TEM, Raman, and XRD were used to explore the surface and morphological properties of MLG/CFO nanocomposites and VSM and MFM were used to explore the magnetic properties of the pristine CFO and the MLG/CFO composite. The MLG/CFO composite shows enhanced magnetic behavior, similar to recent reports of MLG composites with nanoparticles of Fe$_3$O$_4$ Fe$_2$O$_3$, and Co$_3$O$_4$. The results presented here in MLG/CFO composite represent further proof that MLG acquires ferromagnetic moment due to proximity effect when coated with such magnetic oxides. In pristine CFO annealed under the same condition no changes were observed in their magnetic moment. The AFM and SEM images confirm the uniform distribution of CFO nanoparticles on MLG. The likely mechanism for this proximity-induced ferromagnetism in MLG was discussed in some detail in our recent paper on the MLG/Fe$_3$O$_4$ system and it may involve defects created in MLG from the sonication process and possibly charge transfer. How this proximity induced ferromagnetism in MLG can be exploited for device applications needs to be explored in future investigations.

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