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Observation of the transition state of domain wall displacement and GMI effect of FINEMET/graphene composite ribbons†

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In this paper, the morphology, structure, and magnetic properties of FINEMET/graphene composite ribbons are systematically studied by SEM, Raman and static methods (hysteresis loops) and the dynamic method (giant magneto-impedance effect, GMI) respectively. It is revealed that with the increase of the number of graphene layers, the GMI effect of the FINEMET/graphene composite ribbons decreases, and the anisotropy field and the relaxation frequency of domain wall displacement of FINEMET/graphene composite ribbons increases. The result also confirmed that graphene does regulate the magnetic properties of FINEMET ribbon. Therefore, a comprehensive analysis of the influence of graphene on the magnetic properties of FINEMET ribbon is of important guiding significance in industrial applications.

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1. Introduction

The giant magneto-impedance effect and graphene have attracted more and more attention from researchers because of their excellent application prospects. Due to the high sensitivity, low saturation magnetic field, low power loss and small hysteresis of the FINEMET ribbon, it is considered as an attractive candidate for making the magnetic sensors based on the giant magneto-impedance (GMI) effect.^{1–3} When there is a conductive or magnetically material around the soft magnetic material, the properties of the materials will be changed due to the interactions between them. Therefore, magnetic performance of soft magnetic materials is strongly correlated with the surrounding coating layer beside them, which can be precisely identified by the GMI effect. Many researchers have previously studied the effects of nickel-zinc ferrite,⁴ copper oxide,⁵ silicon dioxide⁶ and copper,⁷ carbon materials⁸ on the GMI effect of soft magnetic materials. Recently, some researchers have turned their attention to how to combine graphene with soft magnetic materials to study their structure, magnetic properties, sensitivity and other scientific problems and produce some meaningful research. For example, Zhen Yang *et al.* studied the GMI effect of tortuous-shaped Co-based amorphous ribbon coated with graphene and reported that the 13.1% MI ratio enhancement was observed by spin coating graphene on the Co-based amorphous ribbon.⁹ L. Jamilpanah *et al.* found that deposition of graphene oxide (GO) on the surface of

Co_{68.15}Fe_{4.35}Si_{12.5}B₁₅ ribbon is performed using an EPD method to evaluate the MI response.¹⁰ The results indicated that the MI ratio of Co-based ribbon deposited GO with 4 and 8 min increased 10% and 30% respectively. Yi Zhang studied the magneto-impedance effect of Fe_{75.5}Cu₁Nb₃Si_{13.5}B₇ ribbon covered by *in situ* grown vertical graphene sheets.¹¹ The results indicated that at certain frequency, the magneto impedance effect of a one side graphene covered sample shows enlargement, but the both sides covered sample shows the reverse tendency. The above researchers studied the change of GMI effect of soft magnetic materials from the perspective of thickness and morphology. Besides, magnetic power loss is also an important question for an electronic device in practical applications. Praveena studied the relaxation frequency and lower power losses in Zn²⁺ substituted ferrites.¹² In general, magnetic loss refers to eddy currents, hysteresis, and loss due to magnetization relaxation or magnetic aftereffects that are associated with magnetization or magnetization reversal processes. In the high frequency band, (the frequency is approximately equal to 10⁶–10⁸ Hz), mainly due to the resonance or relaxation of the domain. In the ultra-high frequency band (frequency is approximately equal to 10⁸–10¹⁰ Hz), it mainly belongs to natural resonance. However, few researchers have systematically studied the morphology, impedance ratio, the relaxation frequency of domain wall displacement and sensitivity of the FINEMET ribbon coated with graphene prepared by CVD method. In this paper, the magnetic properties of FINEMET/graphene composite ribbons are studied by static method and the dynamic method. Our experiment results are exactly the opposite of that of other research groups. The results demonstrate the correctness of other groups and enrich the mechanism research of FINEMET/graphene composite ribbons in the field of GMI effect.

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2. Experimental method

A. Preparation of graphene and FINEMET ribbon

The Fe-based amorphous ribbons were prepared by rapid quenching technique and cut to 20 mm (width 0.6 mm and thickness 33 μm). The wheel linear speed is 20 m s^{-1} . Then, it is annealed for 20 minutes to become FINEMET ribbon at the pressure of 9.8×10^{-5} Pa and 540 $^{\circ}\text{C}$. The FINEMET ribbon has two different surfaces: the free side surface and wheel-side surface with different roughness respectively. Graphene is obtained by traditional CVD method. Specifically, the copper foil was cut into $2 \times 2 \text{ cm}^2$ square sheets, placed in a quartz tube. The hydrogen gas (10 sccm) was injected under low pressure (1 Pa) conditions when the temperature was raised to 1050 $^{\circ}\text{C}$ within 90 minutes. Then, CH_4 (15 sccm) was injected and held for 45 minutes to grow graphene on the copper foil. At last, the graphene is transferred to the free surface of the FINEMET ribbon layer by layer, and then the FINEMET/graphene composite ribbons are annealed for 30 min at 400 $^{\circ}\text{C}$ under low pressure conditions (3 Pa) and hydrogen atmosphere (10 sccm).

B. Sample characterization

The magneto-impedance was measured by using the impedance analyzer (HP4294A). The amplitude value was 10 mA and the frequency of the ac current were 100 Hz to 100 MHz, respectively. The relative change of magneto-impedance, *i.e.* the MI ratio, was defined as.

$$\frac{\Delta Z}{Z} = \frac{Z(H_{\text{ex}}) - Z(H_{\text{max}})}{Z(H_{\text{max}})} \times 100\% \quad (1)$$

$$\xi = \frac{\Delta Z}{\Delta H} \times 100\% \quad (\%/\text{Oe}) \quad (2)$$

where $Z(H_{\text{ex}})$ and $Z(H_{\text{max}})$ are the impedance values of the samples in the external field and in the maximum magnetic field, and $H_{\text{max}} = 90 \text{ Oe}$.

The hysteresis loops of FINEMET/graphene composite ribbons were tested by the Lake-shore 7404 vibrating sample magnetometer (VSM), with the magnetic field parallel to the longitudinal axis. The surface roughness and morphology of FINEMET/graphene composite ribbons were observed using atomic force microscope (AFM) system supported by Workshop Company and S-4800 scanning electron microscope (SEM), respectively. Raman analysis of FINEMET/graphene composite ribbons was obtained with a laser Raman spectrometer, the instrument model number is Renishaw inVia Reflex. All the measurements were performed at room temperature (Table 1).

3. Results and discussion

3.1 Morphology characterization of FINEMET/graphene composite ribbons

Fig. 1(a–h) was the morphology diagram of FINEMET/graphene composite ribbons. In Fig. 1, as the number of graphene layers increased, the surface of FINEMET/graphene composite ribbons became rougher with many defects, such as cracks, wrinkles, and pores. The AFM diagram of the FINEMET/graphene composite ribbons also showed the same feature in Fig. S1.† Since the surface of FINEMET/graphene composite ribbons is getting rougher, the greater obstacle received during the movement, and the greater the energy loss resulted in an increase of resistivity. Besides, the appearance of above phenomenon also confirmed the existence of stress. The eddy-current losses were also affected by stress since the effective resistivity of sample increases with internal stress.¹³

3.2 Raman characterization of graphene–FINEMET ribbon

Fig. 2(a–g) was Raman characterization of the FINEMET ribbon and FINEMET/graphene composite ribbons. As shown in Fig. 2(a), the FINEMET ribbon had three characteristic peaks at the wavenumbers of 300 cm^{-1} , 800 cm^{-1} , and 2450 cm^{-1} position. As shown in Fig. 2(b) that the FINEMET ribbon coated with a graphene layer showed the characteristics peak in A ($A = 300 \text{ cm}^{-1}$, 800 cm^{-1} , and 2450 cm^{-1}) and B ($B = 1350 \text{ cm}^{-1}$,

Table 1 The comparison of sensitivity between GMI materials and other materials

Magnetic sensor type	Materials	Sensitivity (%/Oe)
Superconducting quantum interferometer (SQUID)	GaAs/InAs ⁴³	10^{-10} to 10^{-4}
Giant magnetoresistive sensor (GMR-S)	MnIr/CoFeB/Cu/CoFeB ⁴⁴	10^{-6} to 10^{-2}
	NiO/NiFeCo/Cu/NiFeCo/C ⁴⁵	
Fluxgate sensor (FG-S)	$\text{Co}_{66}\text{Fe}_4\text{Ni}_{14}\text{B}_{14}\text{Si}_{15}$ ⁴⁶	10^{-5} to 10^{-1}
	FeNiCo ⁴⁷	
Hall effect magnetic sensor (HE-S)	NiFe/Cu/IrMn ⁴⁸	$1\text{--}10^6$
	MnBiTe ⁴⁹	
Magneto-optical sensor (MO-S)	LiNbO ₃ ⁵⁰	$1\text{--}10^6$
	Au/NiFe ⁵¹	
	Ag/CeYIG ⁵²	
Inductive sensor	CoFeB ⁵³	10^{-6} to 10^{-2}
Giant magnetoimpedance sensor (GMI-S)	NiFe/Cu ⁵⁴	$10^{-2}\text{--}10^2$
	Py/Ti/Cu/Ti/Py ⁵⁵	
	CoFeSiBCr ⁵⁶	
	Nanocrystalline ribbon ⁵⁷	



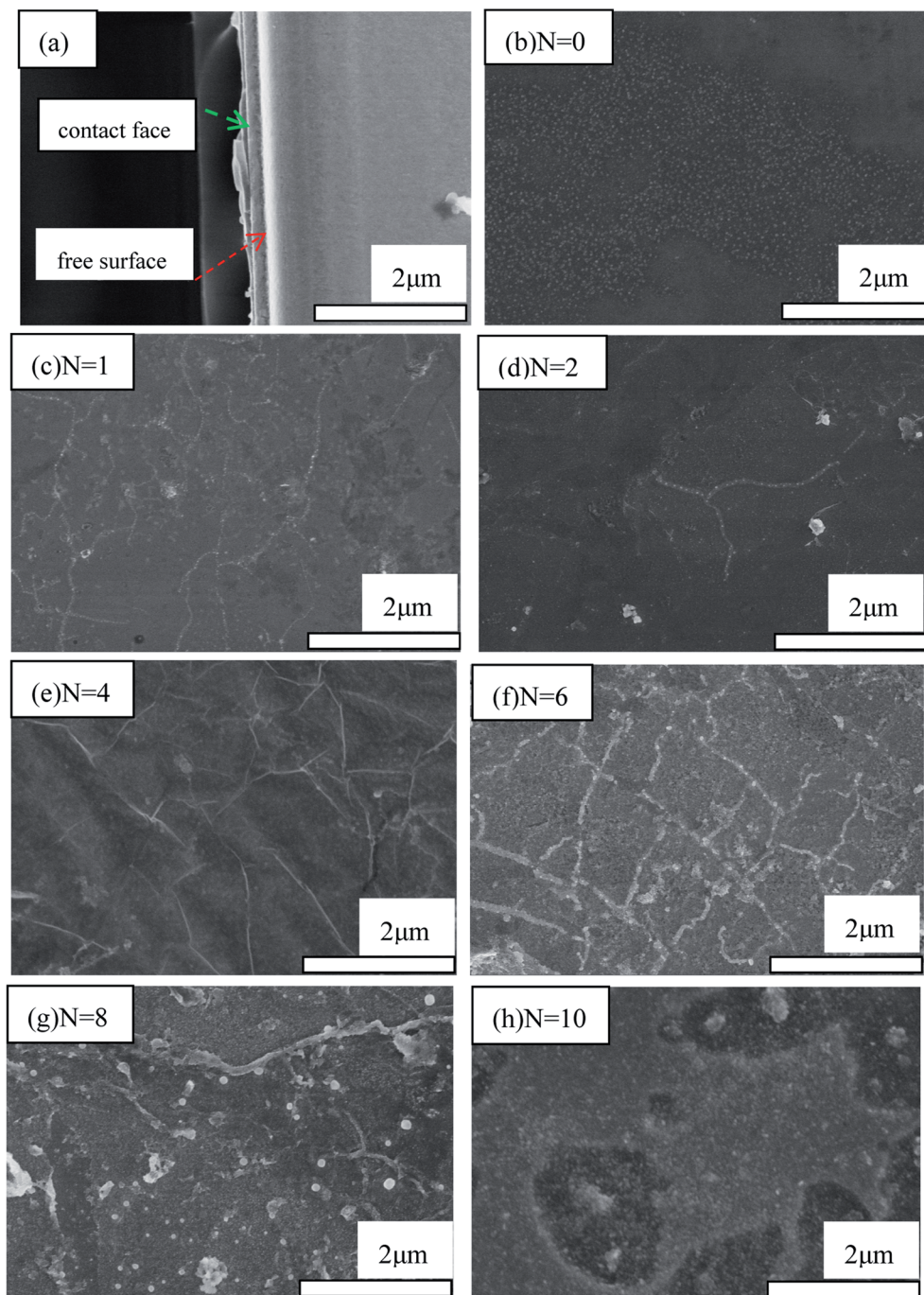


Fig. 1 SEM microscopic images of (a) section image of FINEMET/graphene composite ribbons (b) FINEMET ribbon and (c–h) FINEMET ribbon with N graphene layer ($N = 1, 2, 4, 6, 8, 10$).

1580 cm^{-1} and 2700 cm^{-1}) position. The A and B represented the characteristic peaks of FINEMET ribbon and graphene, respectively. At the same time, a characteristic peak of the C (C represent the characteristic peak of Si–H and Si–H₂) appeared around 2000 cm^{-1} . The reason for the emergence of the C peak was hydrogen annealing caused new chemical bonds between graphene layer and the FINEMET ribbon, such as Si–H and Si–H₂ bond.¹⁴ There were three main effects for the FINEMET/graphene composite ribbons during the hydrogen annealing

process:^{15–19} the first was to remove contaminants (PMMA, *etc.*), the second was to saturate the surface metal dangling bonds, and the third was to form hydrogen bonds and Si–H bonds to increase adhesion between the FINEMET ribbon and graphene.

At the same time, the 2D (2700 cm^{-1}) peak of graphene are sensitive to the stress, which referred to the two-phonon Raman resonance peak.^{20–22} As shown in Fig. 2(h), when the graphene subjected to the stress, the 2D peak would change.²³ Specifically, the 2D characteristic peak of graphene had a small low



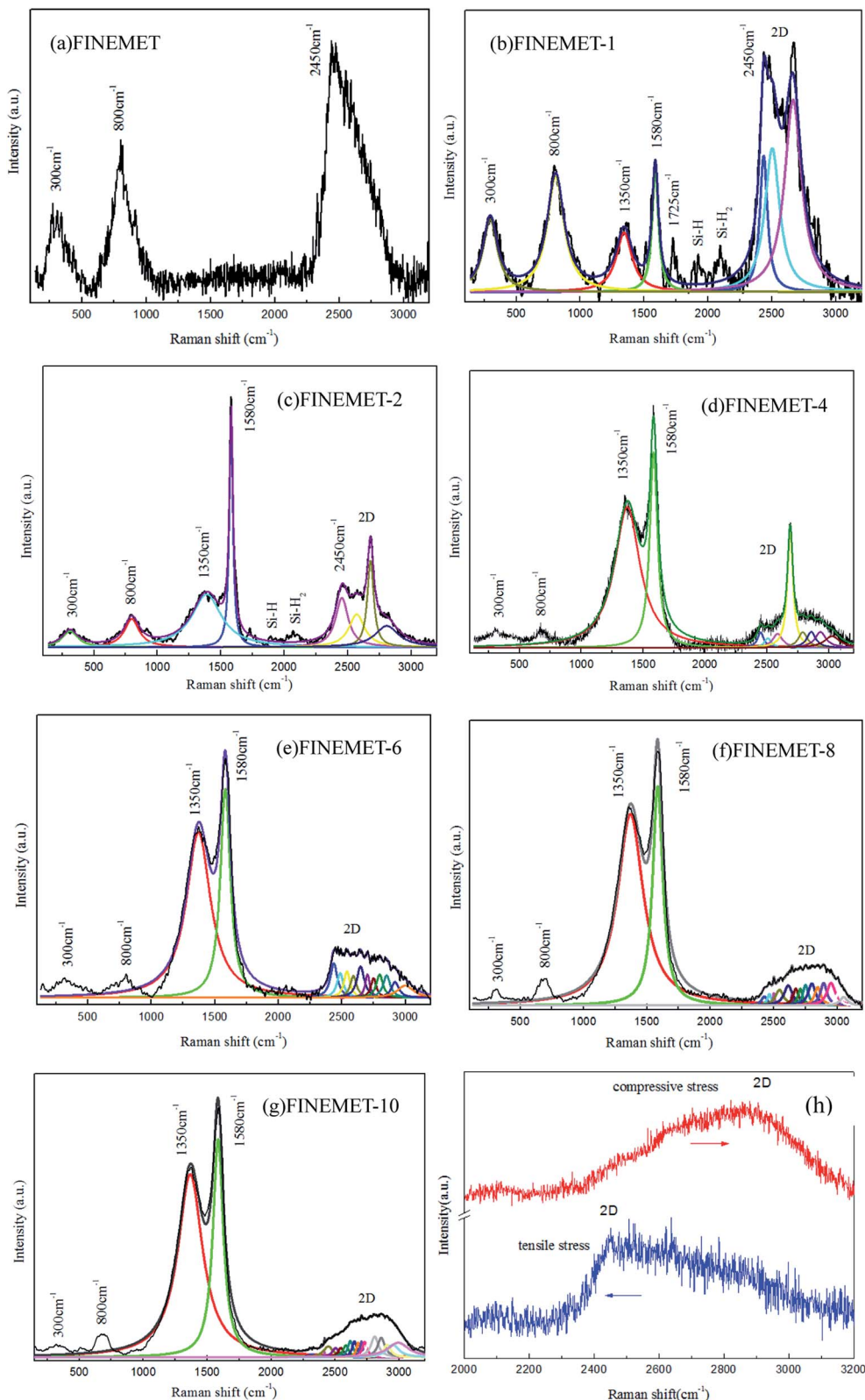


Fig. 2 (a–g) Raman spectrum of FINEMET/graphene composite ribbons and (h) red shift and blue shift of FINEMET/graphene composite ribbons under different stress.

wavenumber displacement and a high wave number displacement when it was received the tensile stress and compressive stress respectively.^{24–26} The reason was attributed to the change

in the carbon–carbon bond. The Lorentz fitting of the characteristic peak of the FINEMET/graphene composite ribbons were shown in Fig. 2(b–g). The 2D peak of the single-layer graphene



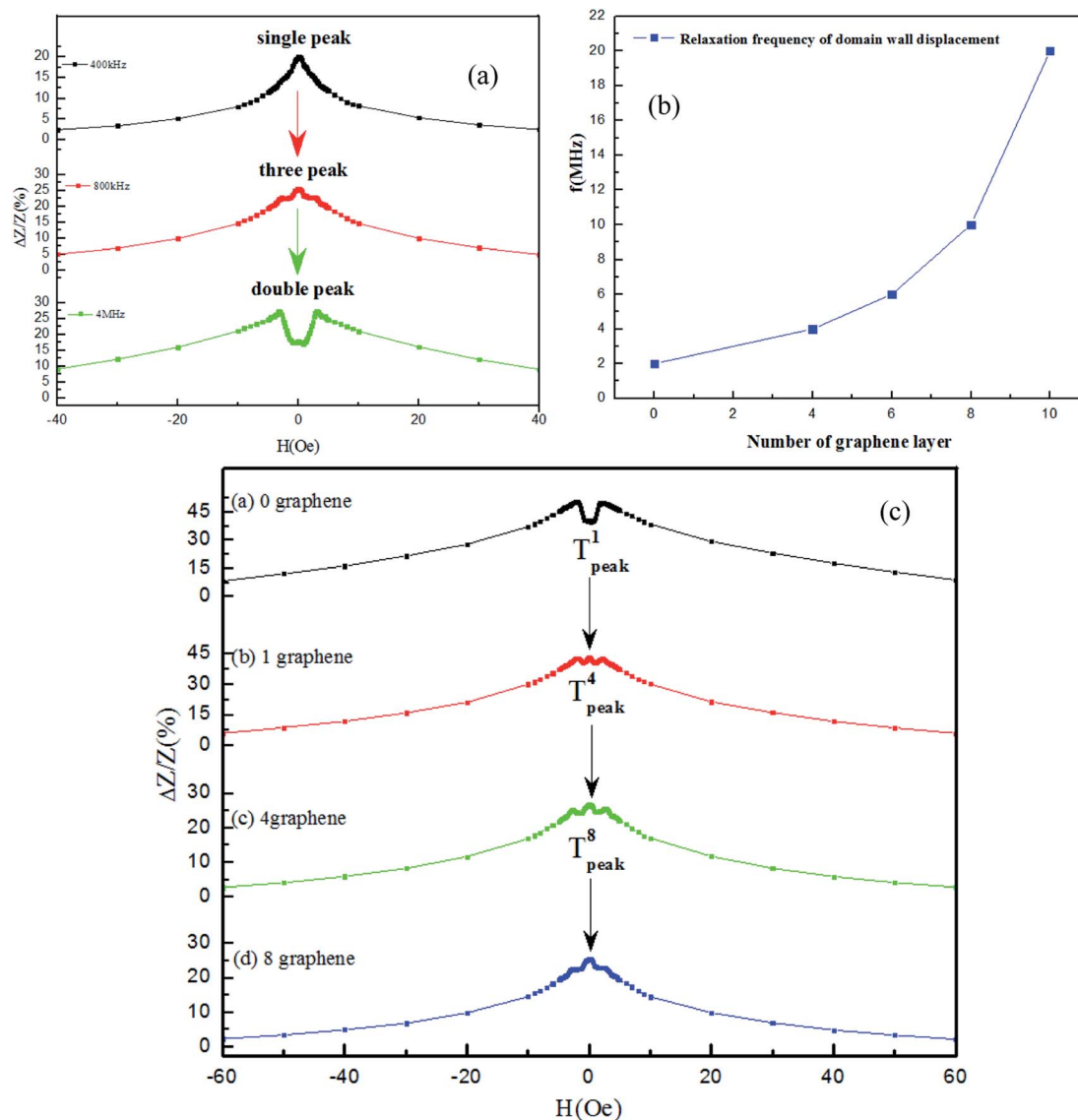


Fig. 3 (a) Three different state of FINEMET/graphene composite ribbon (b) the relaxation frequency of domain wall displacement dependence graphene layers ($N = 0, 4, 6, 8, 10$) (c) the transition state of relaxation frequency of domain wall displacement for the FINEMET/graphene composite ribbons at 0.8 MHz N ($N = 0, 1, 4, 8$).

has a perfect single the Lorentz peak type, which can be divided into two sub-peaks. Similarly, the 2D peak of two layers of graphene has four Lorentz sub-peaks, and so on. By analogy, the 4, 6, 8, and 10 layers of graphene contained 2D peaks with 4, 8, 12, 16, and 20 Lorentz sub-peaks, respectively.

3.3 Relaxation frequency of FINEMET/graphene composite ribbons

Fig. 3(a) was an impedance diagram of FINEMET/graphene composite ribbons. From the Fig. 3(a), the impedance curve was a single peak at low frequencies (400 kHz). This was due to the domain wall displacement of FINEMET/graphene ribbon. At intermediate frequencies (800 kHz), the impedance curve presented three peaks, because of the intermediate process from the domain wall displacement to the magnetic moment

rotation of FINEMET/graphene ribbon.²⁷ At high frequencies (4 MHz), the impedance curve was a double peak, which was related to the moment rotation process of FINEMET/graphene composite ribbons. Fig. 3(b) was a diagram of the relaxation frequency of the domain wall displacement of the FINEMET/graphene composite ribbon with the increase of the number of graphene layers. From the Fig. 3(b), it was observed that with the increase of the number of graphene layers, the relaxation frequency of the domain wall displacement of the FINEMET/graphene composite ribbon gradually increased. The reason was explained by the equation of the dynamics of the domain wall.²⁸

$$\beta \frac{dx}{dt} + ax = 2\mu_0 M_s H(t) \quad (3)$$



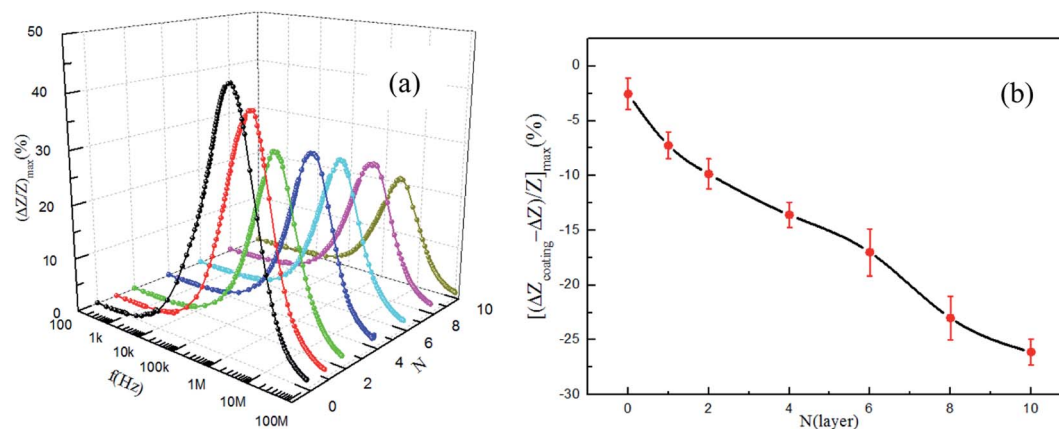


Fig. 4 (a) The frequency of the GMI ratio $(\Delta Z/Z)_{\max}$ for the FINEMET/graphene composite ribbons (b) the impedance ratio of FINEMET/graphene composite ribbons at characteristic frequency ($N = 0, 1, 2, 4, 6, 8, 10$).

where a represents the response coefficient of elastic force, β represents damping coefficient,

$$\beta = \frac{8\mu_0^2 M_s^2}{9\rho} \quad (4)$$

μ_0 represents vacuum permeability. M_s represents saturated magnetization intensity, ρ represents the resistivity of materials. When the magnetic field is $H(t) = H_0 \exp(j\omega t)$, solving the eqn (3) and (4) to obtain the expression of the relaxation frequency of domain wall displacement.

$$\omega = \frac{a}{\beta} \quad (5)$$

When one more graphene layer was coated on the FINEMET ribbon, as the effective resistivity of FINEMET/graphene composite ribbons increased, the damping coefficient decreased. Hence, the relaxation frequency of domain wall displacement increased. This experimental phenomenon was consistent with the theory of domain wall displacement. Fig. 3(c) was the impedance curves of FINEMET/graphene ($N = 0, 1, 4, 8$) composite ribbons at the frequency of 0.8 MHz. The

impedance curves of FINEMET/graphene composite ribbons at different frequency were shown in Fig. S2.† In Fig. 3(c), the FINEMET/graphene ($N = 0$) composite ribbons presented double peak in the impedance curve at 0.8 MHz. However, the FINEMET/graphene ($N = 1, 4, 8$) composite ribbons had a phenomenon of three peaks at same frequency. And also as the number of graphene layers increased, the peaks of the transition state ($T_{\text{peak}}^1 \sim T_{\text{peak}}^4 \sim T_{\text{peak}}^8$) of domain wall displacement of FINEMET/graphene composite ribbons become more and more obvious and sharp. This confirmed that the relaxation frequency of the domain wall displacement had obviously changed.

3.4 Frequency dependence of GMI for FINEMET/graphene composite ribbons

Fig. 4(a) reflected the dependency between the impedance ratio of FINEMET/graphene composite ribbons and frequency as the number of graphene layers increased. Fig. 4(b) showed the change ratio of impedance of FINEMET/graphene composite ribbons at the characteristic frequency. As shown in Fig. 4(a), with increasing frequency, the GMI ratio increased firstly,

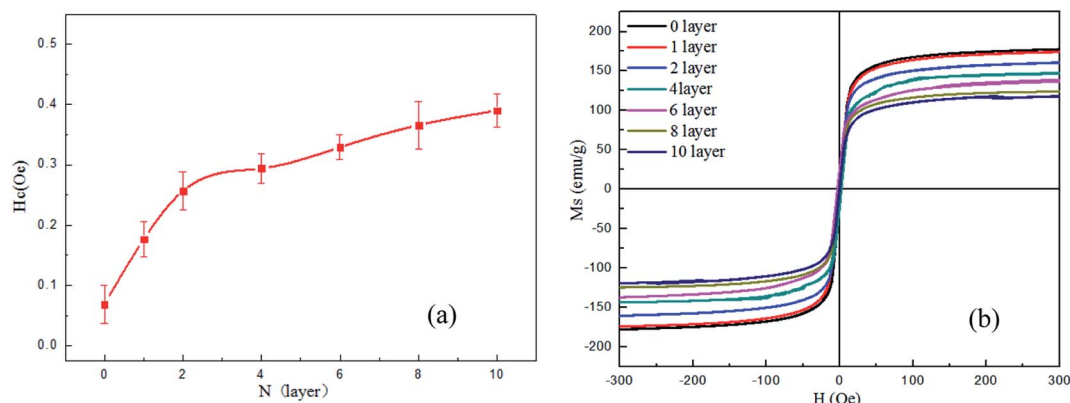


Fig. 5 (a) The H_c of FINEMET/graphene composite ribbons. (b) Hysteresis loops diagram of FINEMET/graphene composite ribbons. N ($=0, 1, 2, 4, 6, 8, 10$).



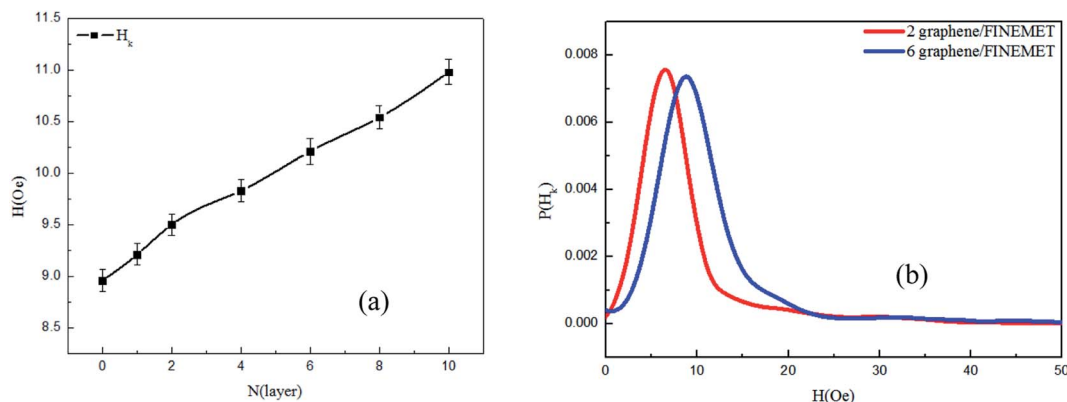


Fig. 6 (a) The field of H_k dependence of the FINEMET/graphene composite ribbons. (b) The H_k field distribution of FINEMET/graphene composite ribbons N (2, 6).

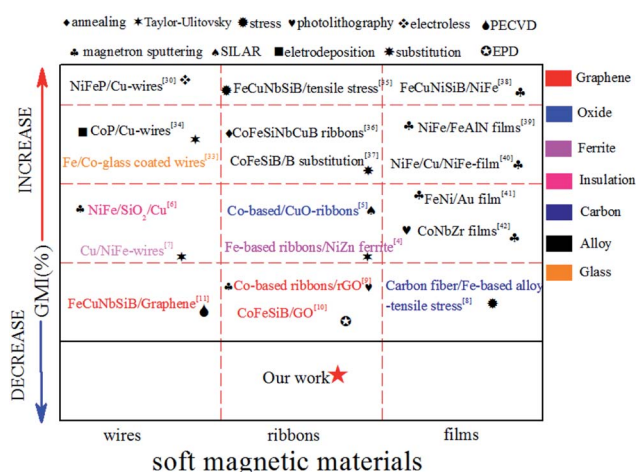


Fig. 7 Comparison our work with other team^{4,9,31,35–44} GMI work.

reaching a maximum at a characteristic frequency f_{\max} and then decreased for higher frequency. At very low frequency, GMI was relatively small due to the dominant contribution of the induced magneto inductive voltage. In the middle frequency range, the skin effect was dominant, hence a higher GMI was observed. With increasing the frequency, GMI decreased. This was related to the domain wall displacement, which was strongly damped owing to eddy current.^{29–31} From Fig. 4(b), as the number of graphene layers increased, the impedance ratio of the FINEMET/graphene composite ribbons decreased. One reason was that the current density of the graphene layer and FINEMET ribbon was different. With the increase of frequency, the current density of the graphene layer was greater than that of the FINEMET ribbon, and so the impedance ratio of the FINEMET ribbon would reduce. On the other, the stress exerted by graphene onto the FINEMET ribbon was increasing during the process of graphene transfer and hydrogen annealing, resulting in a worsening of the magnetic properties of the FINEMET ribbon and the change of effective permeability and leading to lower impedance ratio.

3.5 Hysteresis loop measurement

Fig. 5(a) showed the dependency relationship between the coercivity of FINEMET/graphene composite ribbons and the number of graphene layers. It could be seen from Fig. 5(a) that as the number of graphene layers increased, the coercivity of FINEMET/graphene composite ribbons increased gradually from 0.068 Oe to 0.391 Oe, which was related to stress change. As the number of graphene layers increasing, the stress generated on the FINEMET ribbon was getting larger, so the coercivity of FINEMET/graphene composite ribbons would get larger and larger. Fig. 5(b) showed a hysteresis loops diagram of FINEMET/graphene composite ribbons. In Fig. 5(b), as the number of graphene layers increased, the saturation magnetization intensity of the FINEMET/graphene composite ribbons gradually decreased from 183.56 emu g⁻¹ to 123.52 emu g⁻¹. The more number of graphene layers, the lower saturation magnetization. In addition, the hysteresis loops of FINEMET/graphene composite ribbons showed an S-shape. It demonstrated that the FINEMET/graphene composite ribbons actually hold a preferential transverse magnetic domain structure and the static magnetization process was dominated by the reversible magnetic moment rotation.

3.6 The H_k field dependence of graphene-FINEMET ribbon

Fig. 6(a) showed the changing trend of magnetic anisotropy field of FINEMET/graphene composite ribbons. Fig. 6(b) was the distribution of magnetic anisotropy field of FINEMET/graphene composite ribbons. The anisotropy field H_k have been obtained from the maximum of the second derivative of the hysteresis loops.^{32,33} It can be seen from Fig. 6(a) that as the number of graphene layers increased, the magnetic anisotropy field of FINEMET/graphene composite ribbons gradually increased. This is because that the FINEMET ribbon will undergo a slight stress effect after the graphene is coated. In our experiment, we mainly consider the influence of stress energy. The increase of magnetic anisotropy field was due to the increase of stress in FINEMET ribbon after coating graphene. The stress energy (F_σ) is given as,³⁴



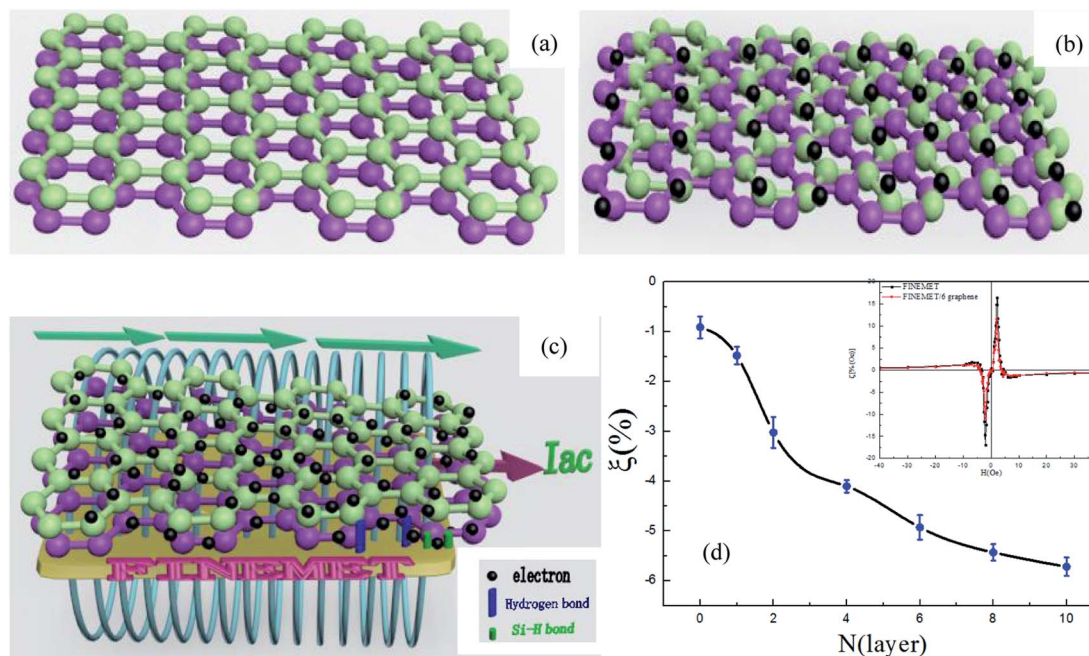


Fig. 8 Mechanism and decomposition diagram of FINEMET/graphene composite ribbons. (a) Layer by layer graphene film by CVD; (b) electronic motion state of graphene film; (c) the entire mechanism diagram of FINEMET/graphene composite ribbons; (d) sensitivity of FINEMET/graphene composite ribbons at characteristic frequencies N (0, 1, 2, 4, 6, 8, 10). Inset chart is the correspondence between sensitivity and magnetic field of FINEMET ribbon and 6-graphene layer-FINEMET ribbon.

$$F_{\sigma} = -\frac{3}{2}\lambda_s\sigma \cos^2 \theta \quad (6)$$

where λ_s is the magnetostriction coefficient and σ_i is the internal stress value, θ is the angle between stress direction and magnetization vector.

3.7 Mechanism and sensitivity of FINEMET/graphene composite ribbons

Fig. 7 was a comparison of the GMI performance of the preparation of soft magnetic materials. There are mainly three aspects of comparison in Fig. 7. Firstly: preparation method (magnetron sputtering, electroless plating, evaporation, electrodeposition, Taylor-Ulitovsky, EPD, PECVD). Secondly: coating type (ferrite, metal, insulating layer, oxide, carbon material, glass). Thirdly: material structure (wire, ribbon, film). The results of the comparison were that our experimental preparation method, performance changes and coating types were different from other research groups. More importantly, our experimental results are mutually verified and complementary to the results of other research groups. Fig. 8(a–c) showed the decomposition and mechanism diagram of FINEMET/graphene composite ribbons. It can be seen from Fig. 8(c) that as the frequency increased, the current mainly passed through the graphene layer, and the current density through the FINEMET ribbon decreased, resulting in a lower dynamic permeability and thus a lower GMI effect. Meanwhile, as the number of graphene layers increased, the FINEMET/graphene composite ribbons were more difficult to reach saturation, and then the sensitivity rate of FINEMET/graphene composite ribbon got greater. Fig. 8(d) was the trend of sensitivity of FINEMET/graphene composite ribbons. It was shown in Fig. 8(d) that as

the number of graphene layers increased, the sensitivity of FINEMET/graphene composite ribbons varied from -0.912 to -5.719 . The supporting data were shown in Fig. S2.†

4. Conclusion

We fabricated the graphene film by CVD method and then transferred it onto the FINEMET ribbon. It was found that the GMI performance of the FINEMET/graphene composite ribbons decreased with the increase of the number of graphene layers. It was due to the stress of FINEMET/graphene composite ribbons gradually enhanced as the number of graphene layers increase. Besides, we also investigated the change law of the transition state of the domain wall displacement to the rotation of magnetic moment. In addition, as the frequency increases, the current density through the Finemet/graphene composite ribbon decreases, which is also the reason of the GMI ratio reduction. In summary, graphene had regulated the magnetic properties of the FINEMET ribbon, which was an important guiding significance in industrial applications.

Conflicts of interest

There are no conflicts to declare.

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