

Tuning Anomalous Hall Signal by Deposition Parameters in Hf/Gd-Fe-Co Bilayer Sheet Films

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ABSTRACT

Here we report the influence of deposition parameters on the magnetic properties of Hf/Gd-Fe-Co sheet films. Samples were grown in a magnetron sputtering where the target's power, pre-sputtering time and power, and capping layer were varied to find the stable perpendicular magnetic anisotropy (PMA) in the series. The magnetic properties of the sheet films were investigated using anomalous Hall effect measurements. It is observed that the Gd-Fe-Co with different Gd-compositions at a fixed FeCo-power exhibit better PMA at 135 W than the 200 W of FeCo. For samples with a MgO capping layer, the magnetic compensation point decreases from 135 W to 105 W (Gd-power), when FeCo-power is decreased from 200 W to 135 W. Moreover, the coercivity vs Gd-power plot diverges at the compensation point which shows an ideal behavior. Similar results are observed with the SiN capping layer with a compensation point at around 85 W. The pre-sputtering time of the targets is also a determining factor of AHE properties, and the longer the time is the better. Furthermore, target cleaning at larger pre-sputtering power reproduces the compensation point around 95 W. These results show ways to achieve the desired PMA and reproducible compensation in the Gd-Fe-Co films, which are potential candidates for the magnetic memory elements.

Keywords: Magnetron sputtering; thin film; Gd-Fe-Co alloy; magnetic compensation; anomalous Hall effect; perpendicular magnetic anisotropy.

1. INTRODUCTION

Rare-earth (RE) - transition-metal (TM) ferrimagnets have been attracted attention since their remarkable application in magneto-optical recording media (Bernacki, Wu, and Mansuripur 1993; Chaudhari, Cuomo, and Gambino 1973; Hajjar and Mansuripur 1992; Hansen *et al.* 1989; Hwang, Wu, and Shieh 1997; Terris *et al.* 1994; Wu *et al.* 1993). These alloys are amorphous and generally (heavy rare earth) possess strong perpendicular magnetic anisotropy (PMA)(Ramesh Chandra Bhatt *et al.* 2019; Hellman 1991; Ye *et al.* 2020). The RE and TM sublattice magnetization align antiferro-

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magnetically so that when changing the magnetization of RE or TM sublattices by changing temperature, concentration, or thickness the resultant magnetization can be changed (Ramesh Chandra Bhatt *et al.* 2019; Ma *et al.* 2018). At the magnetic compensation point, the resultant magnetization becomes zero. Recently, RE-TM has been intensively investigated for their possible use as a replacement of the existing ferromagnetic layer due to their antiferromagnet-like ultra-fast magnetization dynamics and easier magnetic state sensing due to the non-zero magnetization(Alebrand *et al.* 2014; Cai *et al.* 2020; Ivanov 2019). As near compensation, they possess low magnetization, therefore it is hard to sense these materials with a vibrating sample magnetometer (VSM). Thanks to the anomalous Hall effect (AHE) which can sense magnetic state near compensation due to non-zero spin polarization (Bhatt *et al.* 2021; R.C. Bhatt *et al.* 2019; Mimura, Imamura, and Kushiro 1976; Nagaosa *et al.* 2010). The sign of AHE resistance loops gives information about whether the film is RE or TM dominant. The magneto-transport properties of RE-TM alloys are highly sensitive to the change in composition. A slight change in the sputtering power of the cathode target can drastically change the magnetic characteristics, thus reproducing the desired composition in a magnetron sputtering is a challenge. Another problem is that the rare earth sputtering target (Gd, Tb, Dy, etc.), especially Gd, gets easily oxidized in the air which demands target cleaning before the deposition of the film. Therefore, the pre-sputtering condition is also crucial to get quality films. Moreover, the role of the capping layer is important in protecting the alloy from oxidation and affecting the PMA (Ching-Ming Lee *et al.* 2009). MgO can be used as a capping layer as well as the tunnel barrier in magnetic tunnel junctions (MTJ). However, to get a magneto-optic Kerr effect (MOKE) signal, SiNx (SiN) capping is preferred due to its better optical transparency. Therefore, studying the effect of these

two capping layers on film properties is also important. The sputtering parameters for TbFeCo have already been studied by researchers and the data can be found in several studies such as (Zhou *et al.* 2000). However, because of lack of data is available for GdFeCo, we think a detailed assessment of AHE for various deposition parameters is required. Therefore, here we investigate the effect of different deposition parameters (*i.e.*, pre-sputtering conditions, sputtering power change, capping layer) on magnetic properties of GdFeCo film using AHE resistance measurements. We show that the optimized deposition parameters provide a large PMA and reproducible magnetic compensation range in the Hf/Gd-Fe-Co bilayers.

2. SAMPLES AND EXPERIMENTS

The Hf/Gd_x(Fe_{0.8}Co_{0.2})_{1-x} bilayer films with different capping layers (MgO and SiN) on thermally oxidized Si-substrate were deposited using direct-current (DC)/radio-frequency (RF) magnetron sputtering. The sample was spun and orbited simultaneously inside the chamber. The base pressure reached $\sim 2.1 \times 10^{-7}$ Torr before deposition began. The DC power supply was used for sputtering the metal targets, whereas the RF power supply was used for sputtering the MgO and SiN targets. For Gd-Fe-Co deposition, the Gd and Fe_{0.8}Co_{0.2} (FeCo) targets were sputtered using different DC powers to achieve specific compositions. The DC power of the FeCo target was maintained at a fixed output, while the DC power of the Gd-target was varied in the range 60 W to 140 W to deposit different compositions. FeCo power was fixed at 200 W and 135 W for the two different sample series to optimize the film properties. The MgO capping layer was deposited at 200 W RF power and 15 mTorr working pressure. For SiN capping, 100 W RF power was used at 23 mTorr working pressure. Different pre-sputtering times for targets (6 minutes and 12 minutes) were used to observe the target surface cleaning effect on film properties. Similarly, different DC powers (50 W and 100 W) were used during the target pre-sputtering stage. The magnetic properties of the deposited sheet films were characterized using anomalous Hall effect (AHE) measurements. Measurements were performed in a probe station with a 3 kOe magnetic field strength electromagnet. The AHE resistance was measured at a 100 μ A constant DC sensing current.

3. RESULTS AND DISCUSSIONS

Figure 1(a) represents the schematic diagram of the Gd-Fe-Co film structure. The different compositions of the ferrimagnetic alloy are achieved by changing the DC power of the Gd target while fixing the FeCo target at either 200 W or 135 W as shown in Fig. 1(b) and (c), respectively. For FeCo 200 W, the measured AHE loops are shown in Fig. 1(b). We observed that, while increasing the Gd-content, the low Gd-content compositions (60 ~ 80 W) have no AHE hysteresis and the resistance keeps changing with the applied magnetic field due to the absence of perpendicular magnetic anisotropy (PMA) in the alloy. However, on further increasing the Gd-content, the AHE loops do appear exhibiting PMA in the alloy. When increasing the Gd-content up to a certain point (*i.e.*, Gd power from 130 W to 140 W), the sign of the AHE loop reverses from FeCo-dominant to Gd-dominant phase. This crossover suggests the magnetic

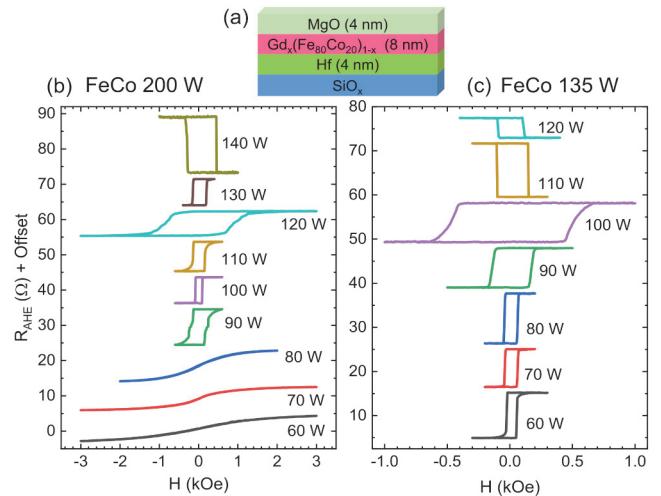


Fig. 1 (a) Schematic of the film stack structure, with corresponding layer thicknesses. Anomalous Hall resistance (R_{AHE}) hysteresis loops measured by perpendicular magnetic field scan for various compositions of Gd-Fe-Co where FeCo target power was fixed at (b) 200 W and (c) 135 W, respectively. The various powers in watt assigned to each curve, represents the corresponding Gd-target DC power.

compensation point lies between 130 and 140 W. Figure 1(c) shows the AHE hysteresis loops for the various Gd-Fe-Co compositions deposited at fixed FeCo power of 135 W, while changing the Gd power from 60 W to 120 W. It is evident from the Fig. 1(c), that the entire series has rectangular AHE loops, and therefore the films consist significant PMA. When increasing the Gd-content, the Hall sign remains the same up to 100 W, which corresponds to the FeCo-dominant phase, however, the sign reversal takes place thereafter, indicating the Gd-dominance. The crossover from FeCo-dominant to Gd-dominant in the 100 W to 110 W range represents the magnetic compensation point. For further analysis of the AHE loops, the R_{AHE} and H_C have been plotted as a function of Gd-power (*i.e.*, Gd-content).

Figure 2 shows the H_C and $R_{\text{AHE}} (= \Delta R/2)$ trend as a function of Gd-content. Ideally, the H_C should diverge at the magnetic compensation point (Ye *et al.* 2020). However, in Fig. 2(a), when the FeCo target is fixed at 200 W, the H_C trend reached a peak value at Gd 120 W, which is inconsistent with the ideal behavior. The peak H_C value of 852 Oe at 120 W is much larger than the other coercivity values suggesting that this composition is close to the compensation. R_{AHE} is generally proportional to the perpendicular component of magnetization, and therefore it is expected to follow the trend of saturation magnetization (M_S) variation with Gd-content (R.C. Bhatt *et al.* 2019; Zhao *et al.* 2015). By definition, M_S drops to zero at the magnetic compensation point. As we see in Fig. 2(a), the R_{AHE} is monotonically decreasing before compensation and suddenly jumps thereafter. This large value of $R_{\text{AHE}} \sim 8 \Omega$ is obtained when the AHE behavior of composition is Gd-dominant, which is about twice the values corresponding to the FeCo-dominant region. There is a notable absence of PMA in the 60, 70, and 80 W samples as shown in Fig. 1(b). The R_{AHE} anomaly seems to be originating from the inhomogeneity in the film, possibly due to the high sputtering power of the FeCo. The higher power provides greater kinetic energy to the sputtering atoms, causing the interdiffusion of FeCo

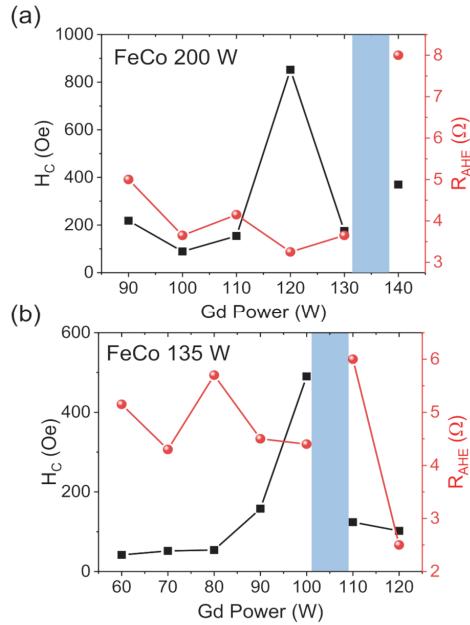


Fig. 2 The variation of coercivity (H_c) and anomalous Hall resistance (R_{AHE}) as a function of Gd-power when the FeCo-power was fixed to (a) 200 W and (b) 135 W, respectively. The shaded region indicates the magnetic compensation range.

atoms to the bottom layer. Moreover, as the deposition power on the FeCo target increases, the deposition rate also increases. This, in turn, affects the uniformity of the film. Therefore, the magnetic properties of the series are inconsistent with the ideal behavior. Conversely, when FeCo power is 135 W as shown in Fig. 2(b), the H_c diverges at the compensation point somewhere between 100 and 110 W. This is consistent with expected H_c vs rare earth (RE)-content behavior. The R_{AHE} remains at around 5 Ω in the FeCo-dominant region but drops to 2.5 Ω for Gd-dominant composition (Gd 120 W). The drop in R_{AHE} at Gd-rich composition can be justified by the lack of FeCo sublattice moments which are shown to be responsible for AHE contribution (Bhatt et al. 2021; Okuno et al. 2016). Our results show that FeCo sputtering power when fixed is crucial for the magnetic properties of the Gd-Fe-Co and high FeCo sputtering power leads to irregular deposition. To investigate further if the irregular deposition problem at high sputtering power might be arising from the insufficient capping layer of MgO, we substituted the MgO capping layer with SiN.

Figure 3 shows the AHE results for the Gd-Fe-Co with the SiN capping layer. When the FeCo power is 200 W, the low Gd power compositions exhibit no PMA in the film; however, on reaching 110 W of Gd power, the film shows significant PMA and FeCo-dominant AHE behavior. For 135 W of FeCo sputtering power, the film shows good PMA, even in the low Gd-content compositions. The AHE sign-reversal takes place from FeCo-dominant to Gd-dominant for 80 to 90 W, respectively. However, the Gd-rich composition at 110 W of Gd-power shows no PMA in the film. The H_c in the series is larger at FeCo-rich and Gd-rich regions, rather than close to the compensation point. This could be because the window for the composition change near the compensation may be very small, meaning that the composition changes quickly around the compensation

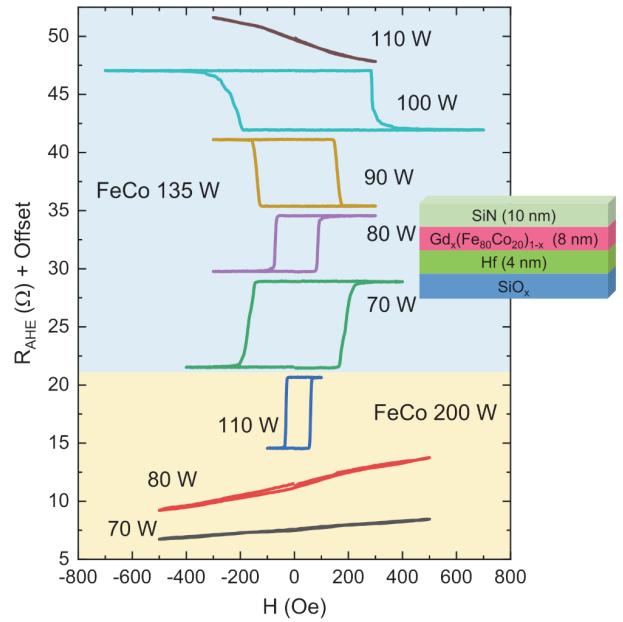


Fig. 3 The anomalous Hall resistance (R_{AHE}) hysteresis loops for different Gd-powers when the FeCo-power was fixed at 200 W and 135 W, respectively, for the light yellow and light blue shaded regions. The embedded schematic shows the film stack structure.

where H_c diverges. The overall behavior of the Gd-Fe-Co films with the MgO and SiN capping layer suggests that FeCo at 135 W provides the most consistent result.

The top surface of targets, especially Gd, is supposed to be rapidly oxidizing or contaminating when exposed to moisture, air, or contaminations from other targets. To add additional control to the deposition parameters, we deposited Gd-Fe-Co films, as shown in Fig 4(a), using two different pre-sputtering times. In the first case, we pre-sputtered Gd and FeCo targets for 6 minutes and in the latter case for 12 minutes. Figure 4(b) shows the AHE results for the 6 minutes pre-sputtering case. All the compositions exhibit FeCo-dominant behavior, except for Gd 80 W film, which shows no PMA. Figure 4(d) shows that the coercivities of the films increase with an increase in the Gd-content from a minimum value of 40 Oe at 70 W, to 953 Oe at 110 W. The large H_c at 110 W indicates the near-compensation behavior of the film. Figure 4(c) shows the AHE results for the 12 minutes pre-sputtering of the targets. All of the compositions exhibit PMA, and the compensation point lies between the 80 and 90 W, where the sign-reversal of AHE loops takes place. The H_c of the compositions follows the expected trend of a ferrimagnetic alloy, as shown in Fig. 4(e). One key concern for the deposition of Gd-Fe-Co, is the oxidation of Gd, as explained in the previous section. This can be avoided by proper target surface cleaning, which in practical terms can be considered increasing the pre-sputtering time. Another way to perform the surface cleaning would be to monitor the kinetic energy of the bombarding Ar-ion, by increasing the pre-sputtering power. Therefore, we pre-sputtered targets at two different powers of 50 W and 100 W to examine the differences in outcomes.

Figure 5(a) shows the AHE response of the compositions corresponding to Gd-power of 80 W, 90 W, and 100 W, where the targets were pre-sputtered at 50 W. The bottom and top three loops

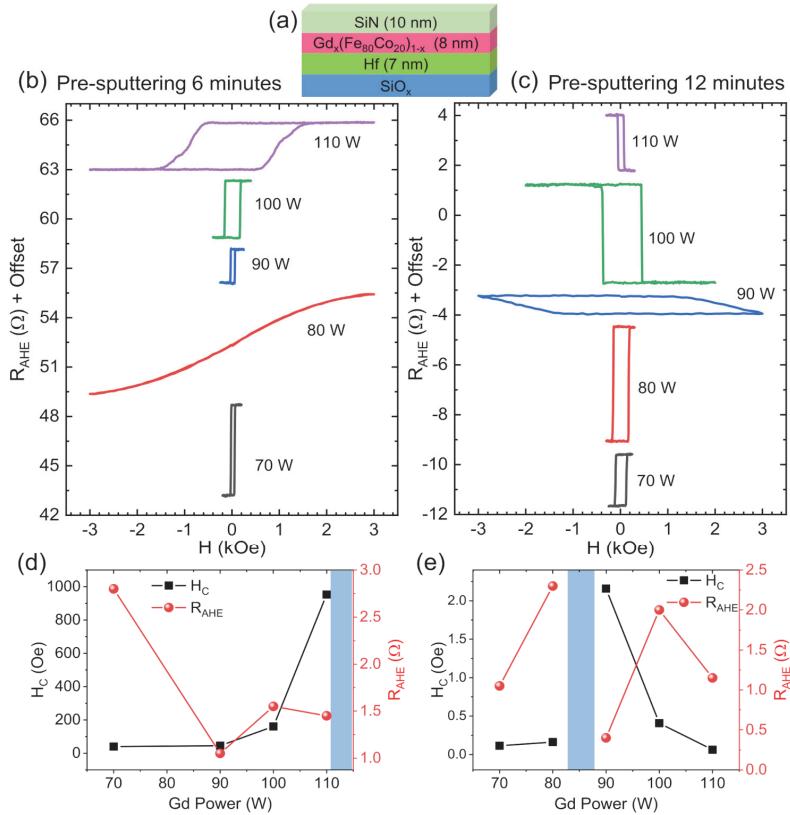


Fig. 4 (a) Schematic of the film stack structure, where SiN is used as a protecting layer. (b) and (c) show the AHE hysteresis loops for different Gd-powers where the targets were pre-sputtered for 6 minutes and 12 minutes, respectively. The magnetic compensation point lies between 80 and 90 W for case (c), which is absent until at least 110 W for case (b). (d) and (e) represent H_c and R_{AHE} ($= \Delta R/2$) as a function of Gd power, for the 6 minutes and 12 minutes pre-sputtering cases, respectively.

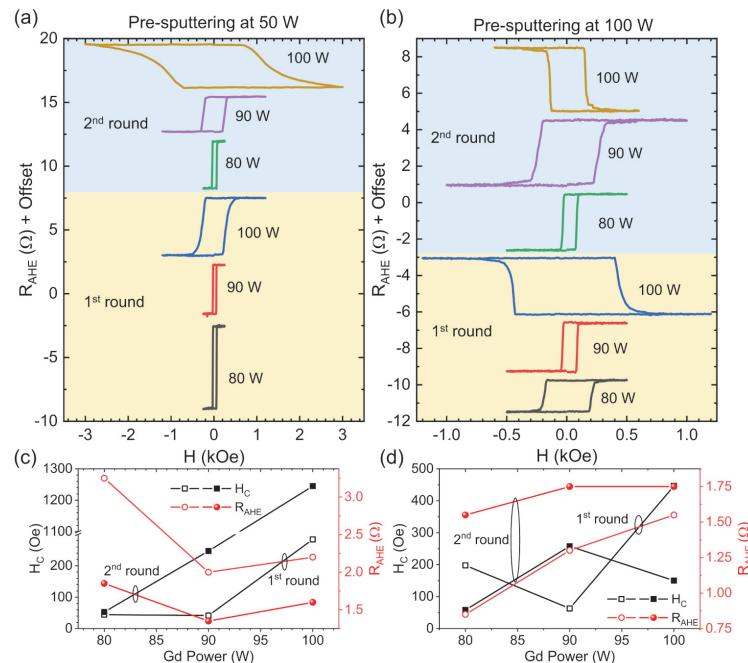


Fig. 5 AHE hysteresis loops when the targets were pre-sputtered at (a) 50 W and (b) 100 W, respectively. Each figure shows two rounds of deposition (i.e., 1st and 2nd round) in the two shaded regions. The magnetic compensation point is repeatable for case (b). (c) and (d) show the H_c and R_{AHE} variation as a function of Gd-power for 50 W and 100 W pre-sputtering power, respectively.

represent the 1st and 2nd rounds of deposition. In the 1st round, all the three AHE loops exhibit the same Hall sign, indicating FeCo-dominance. However, in the 2nd round, the AHE loops show a Hall sign reversal at 100 W. This is the crossover from FeCo-dominant to the Gd-dominant phase. On the other hand, for 100 W pre-sputtering, as shown in Fig. 5(b), the AHE loops show a Hall sign reversal in both the 1st and 2nd rounds, and the compensation composition falls between 90 W and 100 W.

Figures 5(c) and (d) show the H_c and R_{AHE} trend, as a function of Gd-power for the 50 and 100 W pre-sputtering cases, respectively. In fig. 5(c), the H_c is a minimum of 41.5 Oe at 90 W, and a maximum of 284 Oe at 100 W. In the 2nd round, the H_c changes gradually as Gd-power is increased, i.e., 52.9 Oe, 247 Oe, and 1245 Oe for 80 W, 90 W, and 100 W, respectively. The R_{AHE} values for the 1st round compositions are greater than the 2nd round compositions. In Fig. 5(d), the H_c for the 1st round drops to 63 Oe at 90 W composition, whereas for the 2nd round, the H_c follows the general trend i.e., increases toward the compensation point. R_{AHE} values for the 1st round are smaller than for the 2nd round. Overall, pre-sputtering at 100 W has the advantage of producing a specific compensation composition for the same Gd-power range, with a significant PMA.

4. CONCLUSIONS

In summary, we studied and documented the influence of different deposition parameters such as the sputtering target power, pre-sputtering time and power, and capping layer effects on the magnetic properties of Hf/Gd-Fe-Co sheet films. The samples were deposited on diffused Si -substrates in a magnetron sputtering by co-sputtering of Gd and FeCo targets. Several compositions at varying DC power of the Gd-target show a better anomalous Hall effect (AHE) signal at 135 W FeCo-power. Samples with MgO capping showed ideal behavior of coercivity versus Gd-composition plot and demonstrated that the magnetic compensation point decreased as FeCo-power decreased from 200 W to 135 W. For samples with SiN capping, the compensation point was observed at relatively low power levels. Furthermore, targets that were pre-sputtered for longer, exhibited better AHE properties. Target cleaning at a higher power level was shown to reproduce the compensation point. The present findings from this study are relevant to the deposition of Gd-Fe-Co films that require a stable PMA and compensation point for deployment in magnetic memory systems.

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