Magnetic, thermal, electrical properties and crystallization kinetics of $\text{Co}_{60}\text{Fe}_{20}\text{B}_{20}$ alloy films

Ke Wang^{*}, Zhan Xu, Ya Huang, Yuzhen Qiu and Shuo Dong

ABSTRACT Magnetic, thermal and electrical properties of sputtered $\text{Co}_{60}\text{Fe}_{20}\text{B}_{20}$ alloy films are investigated. An excellent linear relationship between the saturation magnetization per unit area and thickness is obtained in the as-deposited film with thickness ranging from 10 to 100 nm. For the as-deposited films a high effective saturation magnetization of $793.7 \pm 42.9 \text{ emu cm}^{-3}$ is derived from the fitting. An in-plane uniaxial magnetic anisotropy of $\sim 10^4 \text{ erg cm}^{-3}$ in magnitude is revealed by angular dependent magnetic measurements. A fourfold cubic magnetic anisotropy is found to develop with annealing, in line with the improvement of the crystalline structure in the film confirmed by X-ray diffraction measurements. Thermomagnetic measurements reveal a considerable variation of the saturation magnetization with temperature arising from the competition of the crystallization and the temperature dependent ferromagnetic behavior of the alloy film. Pronounced changes in electrical resistivity with temperature were observed in the alloy film, in accompany with the structural change. The activation barrier for crystallization was determined to be 1.43 eV from the electrical measurements with variable heating rates, showing a good thermal stability of the alloy film.

Keywords: $\text{Co}_{60}\text{Fe}_{20}\text{B}_{20}$, magnetic properties, thermal properties, electrical properties, crystallization

INTRODUCTION

Ferromagnetic CoFeB alloy has attracted special attention for its excellent magnetic and electrical properties including perfect soft ferromagnetism, high spin polarization and low damping properties [1–6]. The alloy can be used as ferromagnetic electrodes in combination with MgO or AlO_x barriers to fabricate magnetic tunnel junctions (MTJs) with a very high tunnel magnetoresistive (TMR) ratio [7–11]. It has been known that the crystalline CoFeB film in MTJs has enhanced spin tunneling efficiency and hence can exhibit a

higher TMR ratio than the corresponding amorphous one [12]. Therefore, an annealing process is required to induce crystallization of the as-deposited amorphous CoFeB films to increase TMR ratio [13–15]. Lots of works are focused on annealing effects on structural, magnetic and spin dynamic properties of CoFeB films [16–20]. However, thermal related properties and crystallization behavior of the alloy films have not been systematically studied yet [21]. It is of great importance to know more about the thermal related physical properties and structural change from the amorphous to crystalline states of the CoFeB films. In this work, we report the high temperature magnetic and electrical properties as well as crystallization kinetics of the $\text{Co}_{60}\text{Fe}_{20}\text{B}_{20}$ films. The activation barrier for the crystallization was determined by electrical measurements with variable heating rates. Our results show magnetic and electrical properties of $\text{Co}_{60}\text{Fe}_{20}\text{B}_{20}$ alloys are strongly related to the crystallization and structural changes at high temperatures.

EXPERIMENTAL DETAILS

Stacks of Ta (9 nm)/ $\text{Co}_{60}\text{Fe}_{20}\text{B}_{20}$ (10–100 nm)/MgO (1.8 nm) were sputtered onto Si(100) or glass substrates at room temperature with a base pressure lower than $6.0 \times 10^{-6} \text{ Pa}$. Prior to sputtering of the CoFeB film, a 9 nm Ta layer was deposited on cleaned substrates. The CoFeB alloy films with different thicknesses were direct current (DC) sputtered using a stoichiometric target. After growth of CoFeB layer, 1.8 nm MgO layer was sputtered on the top using radio frequency (RF) power. Finally, a 9 nm Ta capping layer was sputtered on the stacks to avoid oxidation. The growth rates of Ta, CoFeB and MgO are 0.078, 0.18 and 0.015 nm s^{-1} , respectively. Sample annealing was carried out in the vacuum chamber from room

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temperature (RT) to 600°C with a pressure of 10^{-4} Pa. The annealing time is half an hour for each temperature. The film structures were characterized by X-ray diffraction (XRD, RigakuSmartLab) with Cu K α radiation ($\lambda = 0.1541$ nm). The surface morphology of the films was examined using atomic force microscopy (AFM, Seiko SPA400) performed in the tapping mode. Magnetic and thermomagnetic properties were characterized using a vibrating sample magnetometer (VSM, Microsense, EZ9). The crystallization process and structural change were monitored by measuring the electrical resistivity in a rapid annealing process setup pumped down to a low vacuum pressure. Electrical resistivity was measured based on the standard four-point method using a combination of source meter and nanovoltmeter (Keithley 2400 and 2182A).

RESULTS AND DISCUSSIONS

Fig. 1a shows the hysteresis loops measured on the as-deposited CoFeB films with the field applied along the [110] direction of the Si(100) substrates. The saturation magnetization increases with the thickness of the films. Inset shows the plot of the saturation magnetic moment per unit area against CoFeB thickness. This thickness dependence of the magnetic moment per unit area was used to determine the effective saturation magnetization and the magnetic dead layer. By linear fitting the effective saturation magnetization M_s and the magnetic dead layer thickness are determined to be 793.7 ± 42.9 emu cm $^{-3}$ and 5.5 ± 3.1 nm, respectively, from the slope and the horizontal axis intercept. The M_s is in good agreement with the reported value of ~ 800 emu cm $^{-3}$ for the amorphous Co $_{60}$ Fe $_{20}$ B $_{20}$ alloy [22]. The

Figure 1 Magnetization hysteresis loops measured along the easy-axis [110] direction of the as-deposited CoFeB films in the thickness range between 10 and 100 nm. Inset shows the plot of saturation magnetization per unit area against film thickness.

presence of magnetic dead layer is often found in amorphous CoFe-based alloy films with Ta layers, which is thought to come from the intermixing at the interfaces [23].

Angular dependent measurements on coercivity H_c and remanent ratio M_r/M_s for the as-deposited CoFeB films are plotted in Fig. 2. The measurements were performed by rotating the film about the (100) direction in the presence of in-plane field. Both angle dependent H_c and M_r/M_s in the as-deposited films show well-defined anisotropy with twofold symmetry. The [110] and $[1\bar{1}0]$ directions are the easy and hard axis of the films, respectively.

The magnetic anisotropy of the films can be determined from the angular dependent magnetic anisotropy energy density ε , which is the integration of the magnetic hysteresis loops [24]. For the films with in-plane uniaxial magnetic anisotropy (UMA), the magnetic anisotropy energy density ε can be written as

$$\varepsilon(\theta) = K_0 + K_u \sin^2(\theta), \quad (1)$$

Figure 2 Angular dependent coercivity H_c and remanent ratio M_r/M_s measured from the as-deposited CoFeB films with various thicknesses from 10 to 100 nm.

where θ is the angle between the field and the easy axis direction, K_0 is the isotropic contribution and K_u is the uniaxial anisotropy constant. From the best fitting using Equation (1), K_u is determined.

Fig. 3 presents the fitted uniaxial anisotropy constant K_u for the as-deposited films in the thickness range between 10 to 100 nm. The K_u initially increases significantly with the film thickness reaching a maximum anisotropy of 2.46×10^4 erg cm $^{-3}$ and beyond this critical thickness it falls. In our experiments, no external magnetic field or oblique deposition geometry was employed during the film sputtering. The demagnetization correction can be ignored as the measuring field is applied in the plane of the nearly square shaped film samples. The strong UMA observed in the as-deposited films can be attributed to the stress during the growth. Similar trend was observed in CoFeB materials deposited on Si substrates, which was attributed to stress relief in the film at a specific thickness [25,26]. Again, it suggests the strong UMA observed in the as-deposited films mainly be induced by stress during the growth. The lowest anisotropy K_u demonstrated in the 10-nm-thick film is consistent with its smallest difference in angular dependent coercivity. The fitted anisotropy K_u is in the same order of 10^4 erg cm $^{-3}$ as that reported in MgO/Co $_{40}$ Fe $_{40}$ B $_{20}$ /Ta structure with similar thickness [26]. The magnitude of the stress may be estimated if the uniaxial magnetic anisotropy in amorphous CoFeB is assumed to be fully induced by the stress. The stress σ can be given by the equation $\sigma = 2K_u/3\lambda_s$, where K_u is the magnetic anisotropy and λ_s is the magnetostriction coefficient. Here, K_u is $\sim 1.8 \times 10^4$ erg cm $^{-3}$ and the value of λ_s is assumed to be 35 ppm for the amorphous CoFeB alloy [27]. The stress is estimated to be ~ 3.4 MPa in magnitude.

Figure 3 The fitted uniaxial anisotropy constant K_u for the as-deposited films with various thicknesses.

Angular dependence of remanent ratio M_r/M_s for the films before and after annealing is shown in Fig. 4a–c. The twofold symmetry in the angle dependence of remanence M_r/M_s maintains in the film annealed up to 300°C. After annealing at higher temperatures a higher order of anisotropy is present along the $[1\bar{1}0]$ direction, which is the hard axis of UMA. Further annealing at the temperatures higher than 500°C leads to an almost isotropic pattern, as seen in angular dependence of remanence. XRD spectra for a 75-nm-thick CoFeB film annealed at different temperatures are presented in Fig. 4d. Except from the Si substrate, no peaks from the film are detected in the as-deposited film, showing the amorphous nature. A hump at around 45° appears in the film after annealing at 300°C. Further annealing at higher temperatures gives rise to the sharp (110) peak of bcc CoFeB phase. The average size of the crystallites d is estimated from the (110) peak using Paul Scherrer formula $d = 0.89\lambda/(\beta\cos\theta)$. Here, λ is the X-ray wavelength (Cu K α , $\lambda = 0.1541$ nm), β is the half width of the (110) peak in radians, and θ is the Bragg's diffraction angle. The average size of the crystallites with the annealing temperature is presented in Fig. 4e. The grain size in the annealed film is estimated in the range of 9.5–29.1 nm, suggesting that nanocrystalline grains are embedded in the amorphous matrix. A monotonic increase in the grain size of nanocrystallites with annealing temperature is exhibited, reflecting that the quality of the structural long-range order improves.

Apart from the uniaxial anisotropy, the additional contribution from the fourfold anisotropy should be included for the annealed films. Consequently, the magnetic anisotropy energy density is given by [24]:

$$\varepsilon(\theta) = K_0 + K_u \sin^2(\theta) - \frac{1}{4} K_c \sin^2(2\theta), \quad (2)$$

where K_c is the crystalline anisotropy constant. The anisotropy constants K_u and K_c derived from the best fitting using Equation (2) are plotted in Fig. 5. For all the annealed films, K_c increases with annealing temperature reaching the maximum and then falls. In contrast, K_u exhibits only a small variation with temperature in our measurement range, which is $\sim 10^4$ erg cm $^{-3}$ in magnitude. Interestingly, the magnetocrystalline anisotropy K_c shows strong film thickness dependence at high annealing temperatures. This can be explained by relating the grain size d to the magnetic exchange length L_{ex} using the random anisotropy model (RAM) [28]. Large grain can be developed in the bulk of the thick film during the annealing. When the grain size increases up to the magnetic exchange

Figure 4 (a–c) Angular dependent remanent ratio M_r/M_s measured from the films with thickness of 25, 50 and 75 nm, respectively, before and after annealing. (d) XRD patterns for a 75-nm-thick CoFeB film annealed at different temperatures and (e) the corresponding grain size obtained from the Scherrer formula.

length ($L_{ex} \sim 20$ nm for the CoFeB), the averaging of the magnetocrystalline anisotropy K_c starts to fall [29]. Hence, a rapid decay of K_c is witnessed in thick films at high annealing temperatures. Consequently, strong film thickness dependent K_c is observed at high temperatures after crys-

tallization.

Fig. 6 presents the thermomagnetic curves of a 50 nm CoFeB film measured at a magnetic field of 1000 Oe with a heating rate of $25^\circ\text{C min}^{-1}$. In the temperature range from RT to $\sim 200^\circ\text{C}$ the saturation magnetization M_s monotoni-

Figure 5 The extracted uniaxial anisotropy constant K_u and cubic anisotropy constant K_c for the annealed CoFeB films with thickness of 25, 50 and 75 nm, respectively.

Figure 6 M_s vs. temperature measured from RT to 700 °C with a magnetic field of 1000 Oe under the protective Ar gas atmosphere. Red curve shows a thermal cycle obtained by heating the film up to 350 °C and then cooling back to RT.

cally decreases as the temperature approaches to the Curie temperature of amorphous phase. In the moderate temperature range from 200 to 450 °C, M_s increases due to the formation of more nanocrystalline magnetic phase, as we observed in the XRD measurements. At high temperatures above 500 °C magnetization decreases again, as we expected, for the temperature approaching to the Curie temperature of crystalline magnetic phase. Red curve shows a thermal cycle obtained by firstly heating the film up to 350 °C and then cooling it back down to RT. A pronounced increase in M_s during the cooling reflects typical ferromagnetic magnetization behaviour. The saturation magnetization of 806 emu cm⁻³ is achieved in the film after cooling, in good agreement with the reported values between 800 and 880 emu cm⁻³ in crystalline Co₆₀Fe₂₀B₂₀ alloy films [22,30].

Fig. 7a presents RT magnetic hysteresis loops of the 50

nm film after VSM heating. The loops were obtained with the field applied along [110] and $[1\bar{1}0]$ axes in the sample plane. A significant increase in M_s from the original value of 654 up to 806 emu cm⁻³ is observed in the film after heated up 350 °C, showing more crystalline ferromagnetic phase is formed. Further heating up to 700 °C, a large coercivity of 139.3 Oe is observed. This pronounced increase in H_c can be ascribed to the enhanced pinning effects due to the surface roughness, which will be confirmed by AFM measurements later. Fig. 7b shows angular dependent magnetic properties of the films before and after VSM heating. Angle dependence of magnetic measurements shows a higher order contribution along the $[1\bar{1}0]$ direction is formed in the film after VSM heating up to 350 °C. The magnetic anisotropy K_u and K_c are estimated to be 2.65×10^4 and 1.2×10^4 erg cm⁻³, respectively, using the above hysteresis area method. When the film is further heated up to 700 °C, a fourfold symmetric pattern in angular dependent coercivity is formed. The magnetic anisotropy K_u and K_c are estimated to be 0.92×10^4 and 1.84×10^4 erg cm⁻³, respectively. Additionally, for the as-deposited film the anisotropy K_u can also be estimated from the easy- and hard-axis loops using the equation $K_u = 1/2(H_k - H_c)M_s$. Here, the saturation field H_k is ~100 Oe and easy axis coercivity H_c is 37 Oe. The saturation magnetization M_s is 654 emu cm⁻³. The anisotropy K_u is calculated to be $\sim 2.06 \times 10^4$ erg cm⁻³, which agrees well with the fitted value of 2.02×10^4 erg cm⁻³. The magnetic parameters obtained in the films before and after VSM heating are summarized in Table 1.

Grazing incident X-ray diffraction (GIXRD) measurements were performed on the film after VSM heating up to 700 °C, Fig. 8 shows the GIXRD pattern of the annealed film and the pattern of the as-deposited film is also included for the comparison. No peaks from the film are detected in the as-deposited film, confirming the amorphous nature. After being heated up to 700 °C apart from the (110) peak of bcc CoFeB phase a strong (104) peak of γ -Fe₂O₃ appears, clearly showing oxidation of iron occurs. The oxidation happens more easily for Fe atoms than Co atoms due to the energetically favorable Fe–O bond formation in comparison with the Co–O bond formation. It reveals that the considerable reduction in M_s at high temperatures in Fig. 7 comes from the oxidation of ferromagnetic elements in the alloy.

Surface morphology for the films rapidly annealed from RT to 700 °C was investigated using AFM. The AFM images with the area of $1.5 \mu\text{m} \times 1.5 \mu\text{m}$ are presented in Fig. 9a–e. The root mean square (RMS) roughness of the film as a function of annealing temperature is shown in Fig. 9f. For

Figure 7 (a) RT magnetic hysteresis loops and (b) angular dependence of magnetic measurements on the 50 nm CoFeB film after VSM heating. The magnetic measurements on the as-deposited film are included for the comparison.

Table 1 Magnetic parameters obtained for the 50 nm CoFeB film before and after VSM heating

Process temperature	H_c (Oe)	M_s (emu cm $^{-3}$)	K_u (10 4 erg cm $^{-3}$)	K_c (10 4 erg cm $^{-3}$)
As-deposited	37	654	2.02	0
350°C	37.2	806	2.65	1.2
700°C	139.3	546	0.92	1.84

Figure 8 GIXRD patterns for the 50-nm-thick CoFeB film before and after VSM heating up to 700°C.

the as-deposited film a small RMS roughness of 0.79 nm is determined. A rapid increase in roughness occurs with the annealing temperature over 400°C after crystallization. The RMS roughness increases to 14.16 nm for the film annealed at 700°C. Large size of grains is formed at high temperatures after crystallization, which enhances the pinning effect and consequently leads to the rapid increase in H_c observed in Fig. 7.

Temperature dependent sheet resistance measurements were performed to investigate the kinetics of the structural change. As the film was annealed, the microstructure transformation could result into a change in electrical resistivity [31]. Fig. 10a presents the temperature dependent sheet resistance of the stack with a 50 nm alloy film deposited on the glass substrates measured at various heating rates. A small decline in sheet resistance with temperature is

Figure 9 (a) AFM images of the 50 nm films before and after rapid annealing: (a) the as-deposited, (b) 200°C, (c) 400°C, (d) 550°C, (e) 700°C. (f) Roughness of the film against the annealing temperature.

Figure 10 (a) Temperature dependent sheet resistance measurements on the 50 nm CoFeB alloy film deposited on glass substrates performed with variable heating rates ranging from 2 to 12°C min⁻¹. (b) Kissinger plots for determination of the activation barrier from a set of electrical measurements with different heating rates.

noticeable in the low temperature region, reflecting the change in electrical transport properties due to the structural relaxation in the amorphous film [32]. Further when the temperature rises to $\sim 300^\circ\text{C}$ a pronounced drop of sheet resistance occurs. This obvious reduction in resistivity corresponds to the crystallization of the alloy, which

agrees with the reported temperature range of 280–340°C for the onset of crystallization of CoFeB alloy films [33,34]. The rapid increase in resistance at the temperature higher than 500°C can be attributed to oxidation, which often happens in the film stacks in this temperature range [35].

Transformation kinetics was examined by applying the

Kissinger analysis. The Kissinger equation is given by $\ln(\beta/T_c^2) = -E_a/RT_c + \text{constant}$, where β denotes the heating rate and T_c is the transition temperature. E_a is the activation barrier and R is the gas constant [36]. The transition temperature T_c is determined by the minimum in the first derivative of the sheet resistance with respect to temperature. The plot of $\ln(T_c^2/\beta)$ as a function of $1/T_c$ should be a linear function with a slope of E_a/R . Consequently, the activation barrier could be obtained by fitting the experimental data in Fig. 8a measured with variable heating rates. Using the data from the electrical measurements an activation barrier of $E_a = 1.43 \pm 0.19$ eV for crystallization was determined from the slope of the straight line in Fig. 8b. This value is slightly larger than the value of 1.32 eV for crystallization of $\text{Co}_{40}\text{Fe}_{40}\text{B}_{20}$ alloy films [34]. It confirms that thermal stability of $\text{Co}_{60}\text{Fe}_{20}\text{B}_{20}$ is better than that of $\text{Co}_{40}\text{Fe}_{40}\text{B}_{20}$ [37,38]. Additionally, the onset of temperature with rapid increasing resistivity in the high temperature region can also be fitted. The barrier deduced at higher temperatures from the fitting is 1.74 ± 0.13 eV, which can be related to the oxidation process of the film stacks.

CONCLUSIONS

In short, we investigated magnetic, thermal and electrical properties of $\text{Co}_{60}\text{Fe}_{20}\text{B}_{20}$ films sandwiched by Ta and MgO layers. The as-deposited amorphous films possess a high effective saturation magnetization of $793.7 \text{ emu cm}^{-3}$. Well-defined in-plane UMA is revealed in the as-deposited alloy films. The magnetic reversal process of the annealed $\text{Co}_{60}\text{Fe}_{20}\text{B}_{20}$ films is found to be governed by the superposition of the uniaxial and cubic magnetic anisotropy, in accord with the film structures. Crystallization is shown to have a significant effect on the magnetic and electrical properties in the films. The activation barrier for the crystallization is electrically determined to be 1.43 eV, showing good thermal stability in the alloy films.

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Author contributions Wang K designed the experiments and wrote the manuscript; Xu Z conducted the magnetic measurements and the fitting; Huang Y fabricated the alloy films; Qiu Y performed the temperature dependent electrical measurements; Dong S carried out XRD and AFM measurements. All authors contributed to the general discussion.

Conflict of interest The authors declare that they have no conflict of interest.

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$\text{Co}_{60}\text{Fe}_{20}\text{B}_{20}$ 合金薄膜磁、热、电特性及晶化动力学研究

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摘要 本文研究了溅射制备的三明治结构Ta/ $\text{Co}_{60}\text{Fe}_{20}\text{B}_{20}$ /MgO合金薄膜的磁、热、电及晶化动力学特性. 10–100 nm厚度的沉积态合金薄膜单位面积磁化强度和厚度呈现了极好的线性相关性, 由此拟合出沉积态合金薄膜有效饱和磁化强度高达793.7 emu cm^{-3} . 转角磁测量揭示了沉积态的合金薄膜具有面内磁单轴各向异性, 其大小为 10^4 erg cm^{-3} 数量级. 发现4重对称的立方结构磁各向异性随着退火温度升高而增大, 这与XRD衍射测量的样品结构的变化一致. 热磁曲线揭示了合金薄膜随温度有相当大变动的饱和磁化强度, 这来源于合金晶化与温度相关的铁磁行为的竞争作用. 伴随结构变化, 观察到明显的合金薄膜电阻随温度的变化. 通过变速率加热电阻测量得到晶化激活能为1.43 eV, 表明该合金材料具有很好的热稳定性.