

High coercivity in nanostructured Co-ferrite thin films

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Abstract. Three methods including sol-gel, rf sputtering and pulsed laser deposition (PLD) have been used for the fabrication of high coercivity Co-ferrite thin films with a nanocrystalline structure. The PLD method is demonstrated to be a possible tool to achieve Co-ferrite films with high coercivity and small grain size at low deposition temperature. High coercivity, over 10 kOe, has been successfully achieved in Co-ferrite films with a thickness of ~ 100 nm deposited using PLD with a substrate temperature at 550°C. The Co-ferrite films prepared by PLD at over 300°C on different substrates including amorphous glass, quartz and silicon exhibits an obvious (111) textured structure and possesses perpendicular anisotropy. Our study has also shown that the high coercivity is related with a large residual strain, which may induce an additional magnetic anisotropy (stress anisotropy) and at the same time serve as pinning centres, which can restrict the domain wall movement and therefore, increase the coercivity.

Keywords. Co-ferrite films; high coercivity; nanostructured.

1. Introduction

Co-ferrite thin films have attracted much attention in recent years as one of the candidates for high density magnetic recording and magneto-optical recording media because of their unique physical properties such as high Curie temperature, large magnetic anisotropy, moderate magnetization, excellent chemical stability and large Kerr and Faraday rotations (Fontijn *et al* 1999; Suzuki 2001). However, important problems that the researchers are concerned about are to make Co-ferrite films meet the requirement of high coercivity, perpendicular anisotropy and small grain size for high density magnetic recording media and to fabricate films using a simple technology with low temperature heat treatment and low vacuum. To settle these problems and achieve potential applications, Co-ferrite films have been fabricated using various sample preparation methods. Especially recently in 1998, Co-ferrite films were fabricated on thermally oxidized silicon wafer by a sol-gel method (Lee *et al* 1998). The films using this method showed no preferred growth direction, and the coercivity was strongly dependent on the annealing temperature but not on field-applied directions. The maximum coercivity value was 2.7 kOe after post-annealing temperature of over 950°C. Kitamoto *et al* (1999) prepared Co-ferrite films on the Corning 7059 glass substrate at 90°C using the spin-spray ferrite-plating method. The films by this method showed no textured structure but exhibited perpendicular anisotropy with the out-of-plane coercivity of over 2 kOe. The excellent perpendicular anisotropy is

required for high density perpendicular recording. In 2000, Co-ferrite films were prepared on silicon wafer using magnetron sputtering, and a high coercivity value of 4.9 kOe without magnetic anisotropy or preferred orientation structure was achieved after post-annealing at 700°C (Ding *et al* 2000), while the Co-ferrite films using similar rf sputtering method and *in situ* substrate heating by Lee's group in 2003 also showed no preferred direction but possessed a perpendicular anisotropy with a maximum value of $H_{c\perp}$, 3.9 kOe (Lee *et al* 2003). Moreover, Gu and Hua (2006) achieved nanocrystalline Co-ferrite films by rf sputtering on quartz substrate with different textured directions along (111), (220) and (311), but these textured films showed a maximum coercivity of only 2.8 kOe without anisotropy after annealing at 400°C. Meanwhile, pulsed laser deposition (PLD) has now proved to be a useful and rather simple technique to obtain thin films of ferromagnetic ferrites (Dorsey *et al* 1996; Oliver *et al* 2000). Co-ferrite films were successfully fabricated epitaxially on (100) MgO by PLD. These films deposited at 800°C showed perpendicular anisotropy with $H_{c\perp}$ as high as 6 kOe (Lisfi and Williams 2003). Based on the previous results by different groups, it is of great significance to develop Co-ferrite films with perpendicular anisotropy and high coercivity of over 6 kOe at low deposition temperature and on cheaper substrates than MgO such as amorphous glass or single crystal quartz and silicon substrates, compatible with the modern microelectronics technique.

In this work, we have prepared Co-ferrite thin films using three methods including sol-gel, rf sputtering and PLD. Magnetic properties of Co-ferrite thin films are strongly dependent on both their nanostructures and the deposition methods. The PLD method is demonstrated to be a suitable

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tool to achieve Co-ferrite films with high coercivity and small grain size at low deposition temperature. After optimization of substrate temperature, the Co-ferrite films derived by the PLD method with *in situ* substrate heating at 550°C possess a coercivity of over 10 kOe with a large perpendicular magnetic anisotropy. The large residual strain and textured structure as observed by both Raman spectrum and XRD may account for the high coercivity of these films.

2. Experimental

Co-ferrite thin films using sol-gel were synthesized by modified Pechini-type processing (Pourroy *et al* 1997). Stoichiometric quantities of $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ (AR) and $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (AR) were used as the starting materials. Citric acid (AR) was used in the preparation of the coating solution with uniformly dispersed metal ions. The raw materials were dissolved in de-ionized water in an ultrasonic bath for 30 min, and then placed in a furnace at 60°C for 24 h in order to form a viscous gel. A small part of the gel was spin coated onto the single crystal substrate (001)- SiO_2 at the spin speed of about 4000 rpm for 30 s. The coating was dried and preannealed at 120°C. The precursor films were annealed at various temperatures under air atmosphere, in order to obtain crystallized Co-ferrite films.

The targets with a stoichiometric composition of CoFe_2O_4 were prepared by sintering at 1300°C for both sputtering and PLD deposition methods. Film preparation by rf sputtering was described in a previous publication (Wang *et al* 2004). Film preparation by PLD was carried out using an ultrahigh vacuum PLD system. Prior to each deposition the vacuum chamber was pumped down to base pressure (2×10^{-7} torr). The beam of KrF excimer laser (248 nm, wavelength and 23 ns, pulse width) was focused with a lens on the rotated Co-ferrite target, where its energy density was estimated to be 3.5 J cm^{-2} . Moreover, the laser repetition rate and the distance between target and substrate were fixed during deposition at 10 Hz and 55 mm, respectively. PLD grown films were under two heat treatment processes. One is post-annealing in air atmosphere similar to that used in both sol-gel and sputtering methods, and the other is *in situ* heating, which means films were deposited under 2 mtorr oxygen gas pressures with the substrate kept at a certain temperature. The thickness of the films was adjusted by the deposition time. The annealed and *in situ* deposited films were investigated with different techniques such as X-ray diffraction (XRD), transmission electron microscopy (TEM), atomic force microscopy (AFM), and Raman spectroscopy for phase and microstructure analyses whereas magnetic properties measurements were performed using vibrating sample magnetometry (VSM) and alternating gradient force magnetometry (AGFM).

3. Results and discussion

Figure 1 shows the coercivity (in-plane coercivity, $H_{c\parallel}$ and out-of-plane coercivity, $H_{c\perp}$) of the 100 nm Co-ferrite films prepared by sol-gel, rf sputtering and PLD and then subsequently annealed at different temperatures. Figure 1(a) indicates that the coercivity of these sol-gel derived Co-ferrite films after annealing at various temperatures are all below 2.5 kOe and not dependent on the direction of applied field, which agrees well with the previous experimental results of Co-ferrite films using similar sol-gel method (Lee *et al* 1998). However, as shown in figure 1(b), the $H_{c\parallel}$ of the films prepared by sputtering first increases with increasing annealing temperature, reaches the optimized value of 7.5 kOe at 900°C, and then decreases with annealing temperature above 900°C, probably due to the growth of grain size as observed in the later XRD patterns. The $H_{c\perp}$ follows the same trend as $H_{c\parallel}$, possesses a value of 6.1 kOe at 900°C and is slightly lower than the $H_{c\parallel}$ at 900°C. A similar trend of coercivity in the function of annealing temperature was observed in the films prepared by PLD as indicated in figure 1(c). The major difference is that the optimized temperature for the highest coercivity is 800°C, and $H_{c\perp}$ is a little higher than $H_{c\parallel}$. The $H_{c\perp}$ and $H_{c\parallel}$ for the 100 nm PLD derived Co-ferrite film annealed at 800°C are 5.9 and 5.5 kOe, respectively. The relatively large $H_{c\perp}$ indicates that the PLD derived films may possess perpendicular magnetic anisotropy. These results reveal that different film deposition methods can result in different magnetic anisotropy and coercivity, probably due to their different nanostructures and growth mechanism.

Figure 2 shows the X-ray diffraction patterns of the 100 nm Co-ferrite thin films prepared by sol-gel, sputtering,

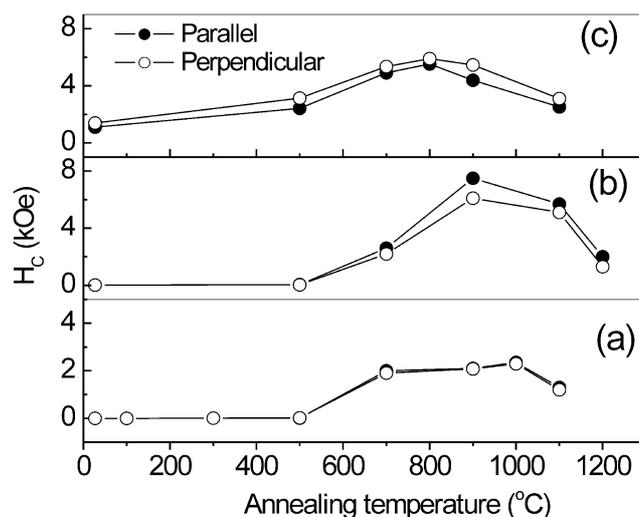


Figure 1. Coercivity of 100 nm Co-ferrite films prepared using sol-gel (a), sputtering (b) and PLD (c), and subsequently annealed at different temperatures ($H_{c\parallel}$ = in-plane coercivity; $H_{c\perp}$ = out-of-plane coercivity).

and PLD and then subsequently annealed at different temperatures for 120 min. After crystallization, all the films using the three methods show a single spinel phase without any preferred orientation, and all peaks are consistent with those of typical Co-ferrite powders prepared by the conventional method (Ding *et al* 1994). The crystallization of the spinel phase for both sol-gel and sputtering grown films starts at above 700°C, which is higher than that for the PLD derived films with the crystallization temperature of 500°C. Furthermore, after annealing at the same temperature for the same annealing time, the peaks from films by PLD are sharper than that from the other two films with similar thickness, possibly due to the difference in kinetic energy of the atoms in the as-deposited stage prepared by the different preparation methods (Hubler and Chrisley 1994). The XRD patterns also reveal that an increase in annealing temperature yields sharpness of these major peaks, indicating an increase in grain size. Figure 3 shows the grain size calculated using Scherrer's equation for 100 nm Co-ferrite films prepared using the three methods, and subsequently annealed at different temperatures. It can be seen that the sol-gel grown films have a grain size of 50 nm, even at an annealing temperature of 1100°C, which is much smaller than those films prepared by sputtering and

PLD. The PLD derived films can form a film structure with a large grain size, which reaches over 200 nm after annealing at 1100°C, while the grain size is around 130 nm for the film prepared by sputtering after annealing at the same 1100°C. Figure 3 also shows that the grain size for these films annealed at 900°C are 24, 75.1 and 80.4 nm for sol-gel, sputtering and PLD grown films, respectively. These results demonstrate that the PLD method may be a possible tool to develop Co-ferrite films with small grain size at a low preparation temperature.

Surface morphologies of these ferrite films were also characterized using AFM. Figure 4 shows AFM images of these Co-ferrite films (100 nm) prepared by different methods and subsequently annealed at 900°C. As indicated by figure 4, the films consist of nanoscaled particles with sizes of 22 nm, 70 nm and 78 nm for the films prepared by sol-gel, sputtering and PLD, respectively. The root-mean-squared (rms) surface roughness of the sol-gel derived films is around 1.5 nm, which is smoother than that of the films derived by sputtering (2 nm) and PLD (3.6 nm). It can also be seen that PLD derived Co-ferrite film has a relatively large broad distribution of particle size in the range 20–140 nm. However, the grain size around 80 nm and the rms value of 3.6 nm with broad particle size distribution are too large to be used in the high density recording media because the large grain size reduces the recording density, and large surface roughness decreases the signal-to-noise ratio (SNR). According to Murdock's estimation that <20 nm grains will be required to be magnetically decoupled from neighboring grains for over 100 Gbit/in² recording media (Weller and Doerner 2000), low temperature film deposition processes should be developed to decrease the grain size and surface roughness. In this work, 100 nm Co-ferrite films have also been prepared by

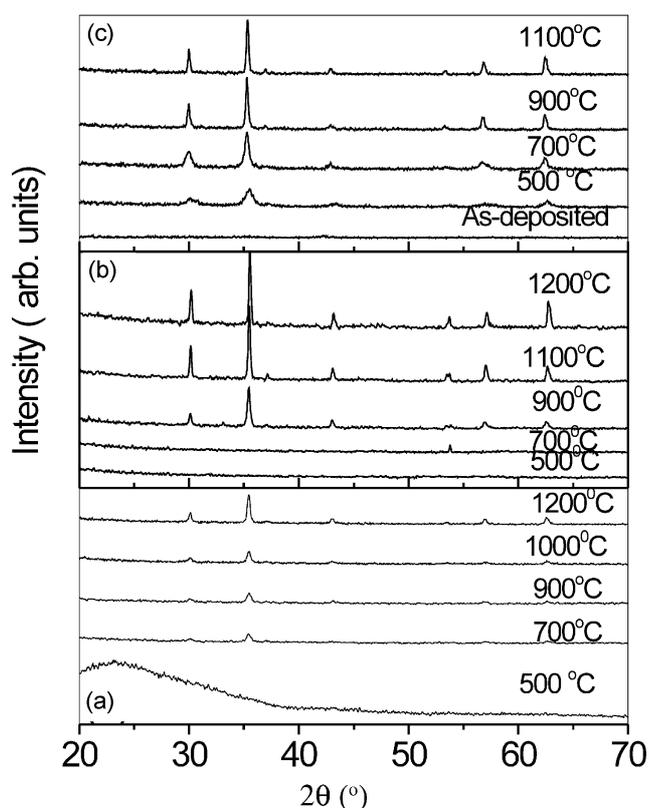


Figure 2. XRD patterns of 100 nm Co-ferrite films prepared on SiO₂-(100) substrates using sol-gel (a), sputtering (b) and PLD (c) methods and subsequently annealed at different temperatures for 120 min.

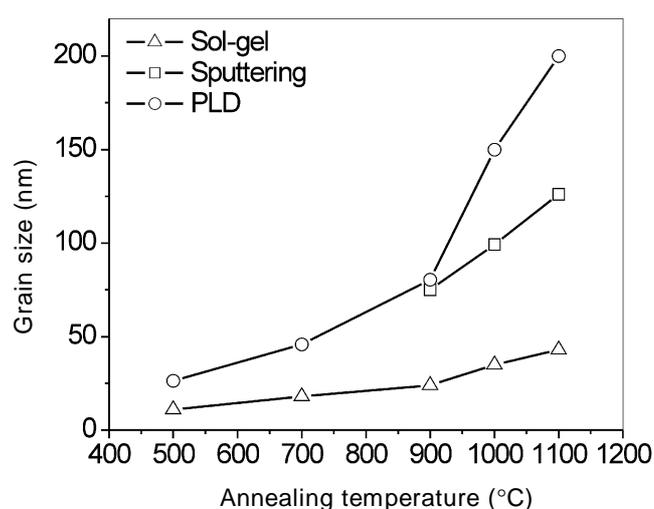


Figure 3. Grain size calculated from Scherrer's equation for 100 nm Co-ferrite films prepared using sol-gel, sputtering and PLD methods, and subsequently annealed at different temperatures.

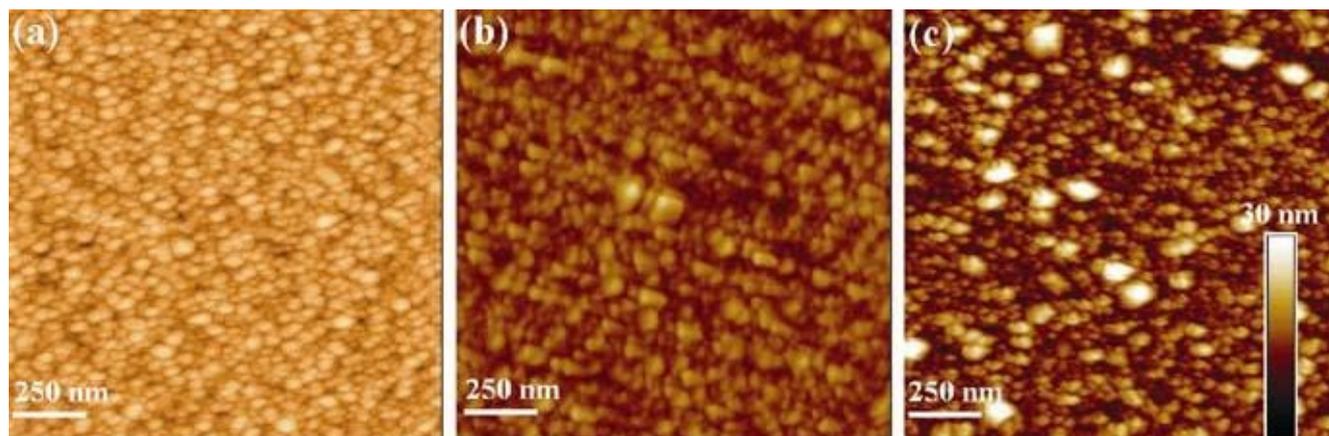


Figure 4. AFM images of 100 nm Co-ferrite films prepared using sol-gel (a), sputtering (b) and PLD (c) and subsequently annealed at 900°C for 120 min.

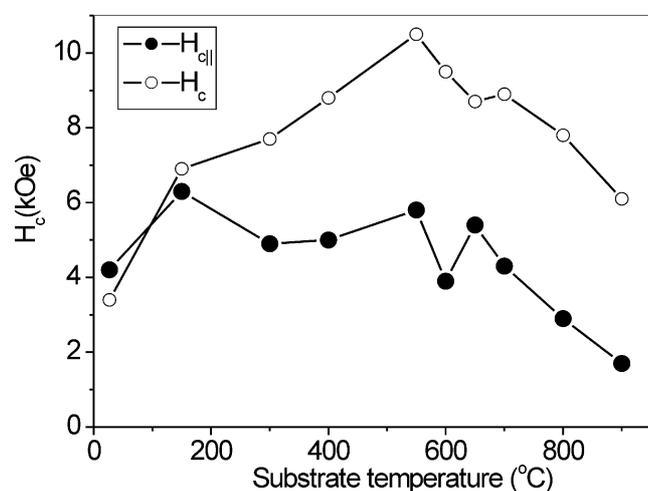


Figure 5. The coercivity of Co-ferrite films deposited on quartz substrate at different substrate temperatures using PLD ($H_{c||}$ = in-plane coercivity; $H_{c\perp}$ = out-of-plane coercivity).

PLD with substrate *in situ* heating at different temperatures under 2 mtorr oxygen gas pressures.

Figure 5 shows coercivity (in-plane coercivity, $H_{c||}$ and out-of-plane coercivity, $H_{c\perp}$) of the Co-ferrite films with a thickness of ~ 100 nm deposited on quartz single crystal substrates at different substrate temperatures. It is found that all these films possess coercivity of relatively high values, which are much higher than that of the bulk Co-ferrite materials (~ 1 kOe). The films deposited at 150°C and below show an in-plane magnetic anisotropy, while out-of-plane anisotropy is found in the films deposited at 300°C and higher. $H_{c\perp}$ increases with the substrate temperature, and reaches a maximum of 10.5 kOe for the film deposited at 550°C. After 550°C, $H_{c\perp}$ decreases with increasing substrate temperature. The $H_{c||}$ of these films shows a similar trend in the range between 1 and 6 kOe. The films using PLD at higher substrate temperatures

appear to be perpendicular anisotropic. Hence, the substrate temperature may play a significant role in adjusting magnetic anisotropy in these PLD derived Co-ferrite films.

Figure 6 shows the hysteresis loops of the 100 nm Co-ferrite film deposited on quartz at 550°C using PLD. The film is obviously high perpendicular anisotropic with $H_{c\perp}$ over 10 kOe as well as lower $H_{c||}$ of 5.8 kOe. High remanence ratio of 0.83 (M_r/M_s) is achieved in the perpendicular direction without the correction of demagnetization field. In the parallel direction, remanence also exceeds 50% ($M_r/M_s = 0.61$). The magnetic anisotropy energy, K_u , of this film can be estimated using the equation

$$K_u = M_s H_k / 2,$$

where H_k is the anisotropy field which can be estimated by extrapolating the magnetization curve of in-plane curve (hard axis) to that of out-of plane (easy axis) (Luo *et al* 2004). The calculated value for K_u is 4.2×10^6 erg/cm³, which is larger than the reported magneto-crystalline anisotropy constant, $K_1 = 2 \times 10^6$ erg/cm³ of bulk Co-ferrite materials (McCurrie 1994). This result indicates the possible presence of an additional magnetic anisotropy. Furthermore, inset in figure 6 shows a negative ΔM curve, which suggests dominance of dipolar interactions in this film (Henkel 1964). As reported previously, dipolar magnetostatic interactions might result in an increasing coercivity of films (Zhu and Bertran 1988). Hence, the dipolar interaction appeared in the film may be one of the possible reasons for high coercivity of the film, and the decoupling dipolar interaction is favourable to decrease SNR of recording media.

Figure 7 shows the XRD patterns of the 100 nm films deposited on quartz at different substrate temperatures using PLD. With substrate temperature at 150°C, the film shows (2 2 0) textured orientation as well as the reflection peak (3 1 1) with a small intensity, while at 550 and 800°C, the films are both highly (1 1 1) textured. The (1 1 1) textured structure may be a possible reason for the per-

pendicular anisotropy of the films in contrast to the trend of coercivity as shown in figure 5. A lattice constant of $a = 0.8482$ and 0.8435 nm were deduced by fitting the spectra for the films at 550°C and 800°C . These results are larger than the target lattice parameter, $a = 0.8382$ nm. The textured structures of Co-ferrite films were also observed previously (Gu *et al* 1996, 2006). The grain size for the film deposited at 550°C is calculated to be below 20 nm, which is much smaller than that of these films prepared by PLD with post-annealing heat treatment.

The XRD fitting analysis also indicates presence of a relatively large residual strain in these films with high

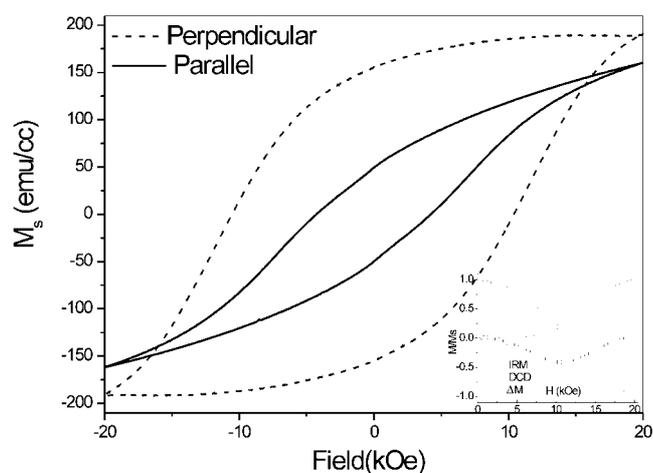


Figure 6. The magnetization hysteresis loops of the 100 nm Co-ferrite films deposited at 550°C on the quartz substrate using PLD. The inset is normalized magnetization DCD (H), IRM (H) and ΔM curves for this film.

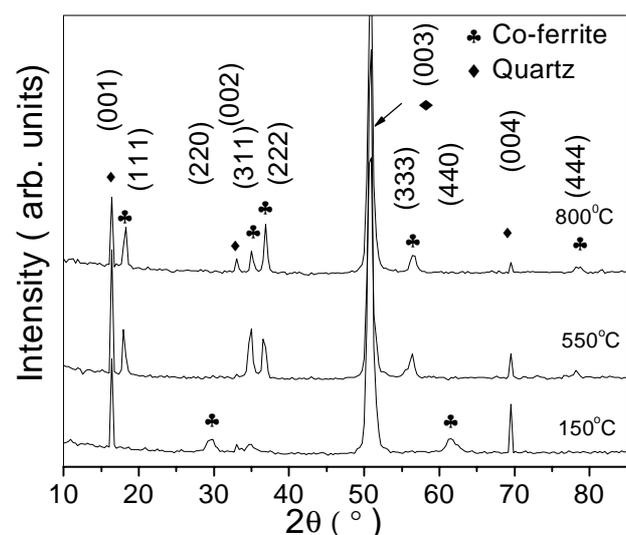


Figure 7. XRD patterns for the 100 nm Co-ferrite films deposited on quartz substrate at different substrate temperatures using PLD.

coercivity. According to previous reports (Wakiya *et al* 2004), a large residual strain in Co-ferrite films may result in strong stress anisotropy and then high coercivity. Similar experimental results were also observed in the Co-ferrite films by sputtering in this work. We have prepared Co-ferrite films with a thickness of 50 nm using sputtering and subsequently annealed at 900°C for 2 h, the coercivity increased to 8.4 kOe. Furthermore, an increase in coercivity to a higher value of 9.3 kOe was achieved by a reduction of annealing time to 15 min for the 50 nm film. This annealing-time-dependent coercivity probably is related to the effect of the residual microstrain, which may induce large stress anisotropy. As annealing time decreased from 120 min to 15 min, the lattice strain may not be relaxed in such a short time and then resulted in a higher coercivity.

Raman spectroscopy is a powerful tool for the examination of residual strain in thin films (Yu *et al* 2002a). A peak shift in the Raman spectra of the 50 nm film by sputtering can give strong evidence that a large residual strain may be a possible reason for high coercivity of these Co-ferrite films using sputtering (Wang *et al* 2004). For the PLD derived films, figure 8 shows the Raman spectra

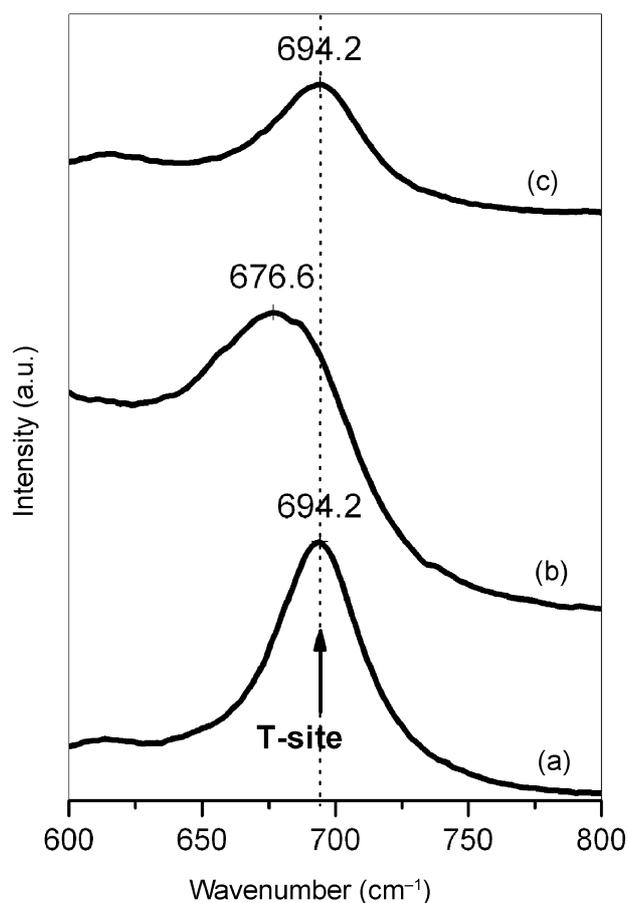


Figure 8. Raman spectra for the 100 nm Co-ferrite films prepared using PLD with 800°C post-annealing (a) and *in situ* heating (b) as well as the Co-ferrite target (c).

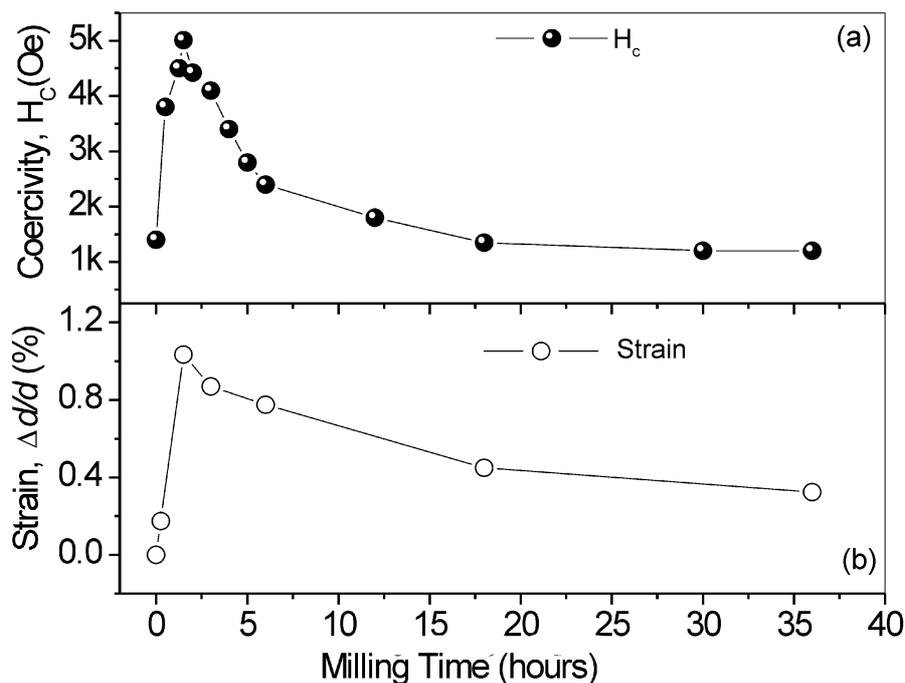


Figure 9. (a) Milling-time dependent coercivity (H_c) and (b) strain of the Co-ferrite powders.

of tetrahedral site for the low coercivity films after post-annealing at 800°C (a), the high coercivity film with *in situ* heating at 800°C (b), and the Co-ferrite target (c). Normally, there are two sites (octahedral and tetrahedral, as denoted by O- and T-sites) for 3d-ions in the spinel structure (Yu *et al* 2002b). Because of a strong overlapping of the O-site peak with the peaks of quartz substrate, only the peak of the T-site is shown in figure 8. The low coercivity Co-ferrite film has the same peak position (694.2 cm^{-1}) for T-site Raman peak as the Co-ferrite target, while the Co-ferrite film with *in situ* heating with the position of 676.6 cm^{-1} has an obvious Raman shift, which possibly suggests that the film with high coercivity possesses a large residual strain inside.

In order to further study the relationship between the coercivity mechanism and residual strain, mechanical milling was employed as an effective way to build up stress-induced high coercivity in Co-ferrite powders. The starting Co-ferrite powders were synthesized by co-precipitation processes and then annealed at 1000°C (Shi *et al* 2000). The powders after annealing had a grain size of around 240 nm, and the original coercivity of 1.4 kOe. The mechanical milling of the annealed powders was conducted using a Spex-8000 miller for different periods of time (up to 36 h). XRD and TEM were used for the microstructure study. Based on the XRD analysis, the grain size and the residual strain were deduced using the Williamson–Hall (W–H) plots (Cullity and Stock 2002).

Figure 9(a) shows the room-temperature coercivity, H_c , of the Co-ferrite powders after mechanical milling for different periods of time. The coercivity, H_c , increases fast initially and reaches maximum values after a short milling time (5.1 kOe after milling for 1.5 h). Further milling leads to the decrease in H_c , which finally reaches a value of around 1 kOe after a prolonged milling of the sample. The residual strain derived from the Williamson–Hall plots is shown in figure 9(b). As seen from this figure, it is clear that the trend of residual strain is consistent with that of coercivity as shown in figure 9(a). It also suggests that the milling-induced high coercivity is closely related with the large residual strain introduced by mechanical milling.

The detailed microstructural evolution of Co-ferrite powders during mechanical milling has also been investigated by TEM analysis. Before milling, Co-ferrite powders are well crystallized with few defects and a low level of strain, as revealed by the high-resolution TEM (figure 10(a)). After mechanical milling for 1.5 h, a large quantity of shear bands with large lattice distortion is observed, as indicated by the arrows in the bright-field TEM image in figure 10(b). A high density of defects inside these shear bands are observed, as evidenced by the dislocation-typed defects and the correlated strong strain contrast (figure 10(b)). Such defective structures are certainly associated with the relatively large residual strain as revealed by XRD. The defects can increase magnetic anisotropy (stress

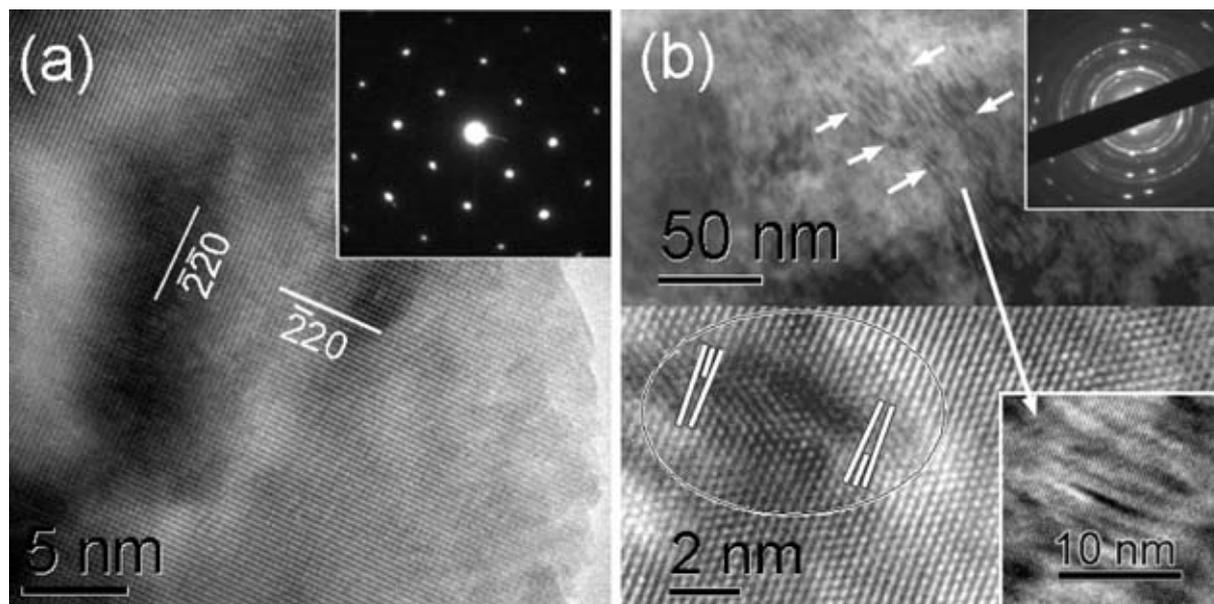


Figure 10. Bright-field and high-resolution TEM images with electron diffraction patterns of Co-ferrite powders: (a) before milling and (b) after milling for 1.5 h.

Table 1. Magnetic properties of 100 nm Co-ferrite films deposited at 550°C on different substrates (coercivity, $H_{c\parallel}$ and the remanence ratio $(M_r/M_s)_{\parallel}$ measured in the film plane-in-plane; coercivity, $H_{c\perp}$ and the remanence ratio $(M_r/M_s)_{\perp}$ measured perpendicular to the plane-out-of-plane).

Substrate	$H_{c\parallel}$ (kOe)	M_r/M_s In-plane	$H_{c\perp}$ (kOe)	M_r/M_s Out-of-plane
(001)-SiO ₂	5.8	61.5	10.5	82.8
Amorphous glass	4.0	58.6	8.4	73.4
(111)-Silicon	5.2	55.2	7.2	62.0

anisotropy) and probably play as pinning centres at the same time, leading to a great increase in coercivity (from 1.4–5.1 kOe). The coercivity mechanism related with residual strain of the high coercivity Co-ferrite films and powders needs to be studied in the future.

In addition, the substrate effect on the Co-ferrite films prepared using PLD has also been conducted in this work. We have deposited 100 nm Co-ferrite films at 550°C on other substrates including glass (amorphous) and (111)-silicon except for quartz. The magnetic properties (coercivity and remanence measured in the two directions: in-plane and out-of-plane) of these films are summarized in table 1. All these films show perpendicular magnetic anisotropy even on amorphous glass or silicon substrates. The $H_{c\perp}$ is over 8 kOe for the film on glass. Moreover, the remanence for these films at both directions is higher than 0.5. XRD results also show a (1 1 1) textured structure for these films on different substrates, and AFM images indicate the rms roughness to be below 1 nm. The high coercivity, perpendicular anisotropy and smooth surface of

these films on amorphous glass or silicon make them very attractive for technological applications.

4. Conclusions

In this work, we have used three methods including sol-gel, rf sputtering and pulsed laser deposition for the fabrication of Co-ferrite thin films with a nanocrystalline structure. We have demonstrated that it is possible for the PLD method to be a suitable tool to achieve Co-ferrite films with high coercivity and small grain size even at low deposition temperature. After optimization of the substrate temperature, high coercivity of over 10 kOe has been successfully achieved in PLD derived 100 nm Co-ferrite films at 550°C. The film exhibits an obvious (111) textured structure and possesses a high perpendicular anisotropy. Our study has also shown that high coercivity mechanism is associated with a relatively large residual strain, which possibly results in additional stress anisotropy.

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