# THE EFFECTS OF SUBSTRATE PREHEATING AND POST-DEPOSITION ANNEALING ON CrMn/CoCrPt/CrMn/NiAl FILMS

JIE ZOU \*, DAVID E. LAUGHLIN \*\*, and DAVID N. LAMBETH\*

\*Department of Electrical and Computer Engineering, \*\*Department of Materials Science and Engineering, Data Storage Systems Center, Carnegie Mellon University, Pittsburgh, PA 15213, jzou@henry.ece.cmu.edu

## **ABSTRACT**

In this study, we examine the effects of introducing CrMn as an intermediate layer between the NiAl underlayer and the CoCrPt magnetic layer, and as an overlayer on top of the Co-alloy film. The effects of deposition temperature and post-deposition annealing on the texture and magnetic properties were studied. Post-deposition annealing effectively lowered the exchange coupling between grains, increased the coercivity by 38%, up to 4600 Oe, and moderately decreased the Ms by 15%. Interlayer diffusion of CrMn into the CoCrPt grain boundaries is believed to cause the change in magnetic properties. It was also found that granular exchange coupling seems to be correlated to the Co-alloy in-plane texture.

## INTRODUCTION

High Pt content CoCrPt alloys have relatively large Ku values [1], thus these are good candidates for high coercivity and thermally stable magnetic media. However, they exhibit larger noise than CoCrTa alloys because the granular exchange coupling is higher [2]. One approach to solving this problem is to increase the Cr concentration and the deposition temperature to isolate the grains by Cr segregation at the grain boundaries [3]. However, the Curie temperature and the Ms of the alloy decreases rapidly for higher Cr content. Another method proposed has been to surround the Co-alloy grains with non-magnetic materials to form granular media [4]. However, this kind of media suffers from relatively low Ms. Interdiffusion of Cr from the underlayer into the magnetic layer by post-deposition annealing was also investigated [5,6]. The coercivity increased, grain isolation improved but Ms decreased after annealing. Recently, L. L. Lee et al. found that CrMn underlayers induce higher coercivity in CoCrPt films than pure Cr underlayers when deposited at elevated temperatures [7]. Interdiffusion of Mn to the grain boundaries of the magnetic layer was proposed as being responsible for altering the magnetic properties.

In this study, we have investigated multilayer-structured media including a NiAl underlayer, a CrMn intermediate layer, a CoCrPt magnetic layer, and a CrMn overlayer. The effects of substrate preheating and post-deposition annealing on grain isolation and magnetic properties were studied.

## **EXPERIMENT**

CoCrPt films on CrMn, NiAl or combined CrMn/NiAl underlayers were studied. CrMn overlayers on top of CoCrPt films were also investigated. All films were deposited onto glass substrates by RF diode sputtering either with or without substrate preheating. The Ar sputtering pressure was 10 mTorr and the base pressure was about  $5 \times 10^{-7}$  Torr. Deposition was performed at a fixed AC power density of 2.3 W/cm². All CoCrPt and NiAl layers were 300 and 1000 Å thick, respectively. The CrMn layer thickness was varied. Post-deposition annealings were performed under Ar gas flow.

The compositions of the CoCrPt films determined by energy dispersive x-ray spectroscopy (EDX) were 78 at% Co, 6 at% Cr, and 16 at% Pt. The Mn concentration of the CrMn alloy films deposited in this study was 22 at%.

The in-plane magnetic properties of the samples were measured using a vibrating-sample magnetometer (VSM) and an alternating-gradient magnetometer (AGM) with a maximum applied field of 10 kOe. Film textures were examined by  $\theta$ -2 $\theta$  scan x-ray diffractometry with Cu-K $\alpha$  radiation.

## RESULTS AND DISCUSSION

The in-plane coercivities, Ms,  $\Delta$ M/Mr peak values, and S\* for CoCrPt/CrMn/NiAl films are plotted against the CrMn intermediate layer thickness in Fig. 1 (a)-(d), respectively. Data for samples deposited with substrate preheating to 260 °C and without preheating are shown. Selected x-ray  $\theta$ -2 $\theta$  scans of the above films are shown in Figs. 2 and 3 for comparison.

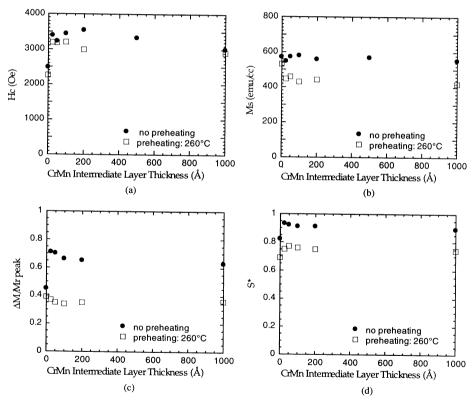
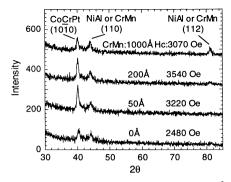


Fig. 1 In-plane magnetic properties (a) coercivity, (b) Ms, (c)  $\Delta M/Mr$  peak, and (d) S\* as functions of CrMn thickness for CoCrPt(300Å)/CrMn(x)/NiAl(1000Å) films deposited with or without substrate preheating.



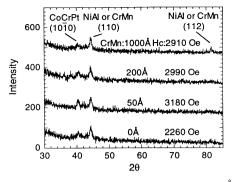


Fig. 2 X-ray diffraction spectra of 300 Å CoCrPt films on various thickness of CrMn intermediate layers on top of 1000 Å NiAl underlayers. No substrate preheating.

Fig. 3 X-ray diffraction spectra of 300 Å CoCrPt films on various thickness of CrMn intermediate layers on top of 1000 Å NiAl underlayers. Substrate preheating: 260 °C.

Without substrate heating, adding a CrMn intermediate layer as thin as 25 Å between the CoCrPt magnetic layer and the NiAl underlayer increased the coercivity by 900 Oe and improved the CoCrPt (1010) texture. No further improvement in the coercivity or the Co-alloy texture was obtained by increasing the CrMn thickness. This is similar to the effect reported for Cr and CoCrTa intermediate layers [8,9]. Contrary to the results reported for CrMn underlayers [7], we have found that for the combined CrMn/NiAl underlayers, applying substrate heating degraded the CoCrPt texture and the coercivity decreased.

The Ms values for samples prepared with no preheating are essentially independent of CrMn thickness and are around 560 emu/cc. When substrate preheating was applied, the Ms of the films with CrMn intermediate layers decreased about 20%, while for the sample with no CrMn layer, no significant decrease in Ms was observed. This is probably due to the diffusion of CrMn into the CoCrPt magnetic layer.

For the CoCrPt/CrMn/NiAl structure, elevation of the deposition temperature lowered the  $\Delta$ M/Mr peak by about half. Further increases in the thickness of the CrMn intermediate layer from 25 Å to 1000 Å resulted in no significant change in the  $\Delta$ M/Mr curves. This implies that only small amounts of CrMn need to diffuse into the magnetic layer to cause the decrease in the granular exchange coupling. This is consistent with the hypothesis that the grain boundary diffusion is the dominant transport mechanism at this relatively low temperature, since grain boundary diffusion is known to have a lower activation energy than bulk lattice diffusion and generally dominate in polycrystalline films at temperatures of less than one half of the melting temperature [10].

The  $S^*$  behavior is consistent with that of the  $\Delta M/Mr$  peak, and follows the concept that clusters of strongly exchange-coupled grains tend to switch together, causing a sharp M-H transition at the coercivity.

It should be noted that the CoCrPt/NiAl sample shows a significantly smaller  $\Delta M/Mr$  peak than the better-textured CoCrPt/CrMn/NiAl sample when deposited without substrate preheating. To possibly exploit the relationship between Co-alloy texture and the intergranular exchange coupling, the magnetic properties of samples with different in-plane CoCrPt texture were examined and the results are summarized in Table I. With no substrate heating, the  $(10\bar{1}0)$ 

textured CoCrPt film on the NiAl underlayer has a higher positive  $\Delta M/Mr$  peak than the ( $10\bar{1}1$ ) textured CoCrPt film on the CrMn underlayer. The CoCrPt/CrMn/NiAl sample that has a stronger CoCrPt ( $10\bar{1}0$ ) texture shows an even higher  $\Delta M/Mr$  peak. The stronger intergranular exchange coupling seems to be correlated with the better in-plane c-axis orientation.

Table I In-plane magnetic properties of samples with different CoCrPt texture.

Sample	Heating	Texture	Hc (Oe)	Ms (emu/cc)	S*	ΔM/Mr peak
CoCrPt(300Å)/CrMn(1000Å)	No	(1011)	1810	541	0.73	0.21
	260 °C	(1120)	3510	433	0.71	0.16
CoCrPt(300Å)/NiAl(1000Å)	No	(1010)	2480	571	0.82	0.45
	260 °C	(1010)	2260	530	0.69	0.39
CoCrPt(300Å)/CrMn(1000Å) /NiAl(1000Å)	No	(1010)	3000	550	0.89	0.63
	260 °C	(1010)	2910	419	0.74	0.36

As shown in Table II, adding a CrMn overlayer on top of the CoCrPt magnetic films increases the coercivity and further decreases the  $\Delta M/Mr$  peak when substrate preheating is applied. This is further evidence that CrMn interlayer diffusion into the CoCrPt grain boundaries might be responsible for the change in the magnetic properties. As anticipated the CoCrPt texture does not change with the addition of an overlayer.

Table II In-plane magnetic properties of  $CoCrPt(300\text{\AA})/CrMn(1000\text{\AA})/NiAl(1000\text{\AA})$  films with and without a 200Å thick CrMn overlayer.

Heating	r	10	260 °C		
CrMn Overlayer	no	yes	no	ves	
Hc (Oe)	3000	3290	2910	3300	
Ms (emu/cc)	550	524	419	428	
S*	0.89	0.87	0.74	0.72	
ΔM/Mr peak	0.63	0.60	0.36	0.22	

Deposition at elevated temperatures can lower the intergranular exchange coupling but it also appears to cause the Co-alloy texture to degrade. Post-deposition annealing may be a better solution. Annealing was performed on the samples with a CrMn overlayer prepared without substrate heating. The overlayer has the function of preventing the CoCrPt film from being oxidized during the annealing and it should be more efficient for CrMn to diffuse from both sides into the magnetic layer. Starting from 250 °C, the sample was annealed at the series of temperatures, 250 + 50N °C (where N = 0, 1, 2, 3, 4) for 1 minute. The in-plane magnetic properties are plotted vs. the annealing temperature in Fig. 4. The  $\Delta M/Mr$  peaks and Ms decrease, and the coercivity increases for the higher annealing temperatures. At the annealing temperature of 450 °C, the  $\Delta M/Mr$  peak drops to about 0.1, the coercivity increases by 40% to 4700 Oe, while the Ms decreases about 30%. The decrease in Ms, as the processing temperature is increased, is most likely a Co bulk alloy dilution effect due to the incorporation of Cr or Mn. However, it is not known how, if at all, the anisotropy energy density, Ku, changes with these dilutions. Since Hc is usually viewed as being proportional to Hk, and since Hk = 2Ku/Ms, only a portion of the Hc increase is due to the 30% Ms decrease. To account for the full 40% increase in Hc, either the Ku must have increased or more likely, the increase in Hc is reflecting a decrease in magnetic reversal via domain wall motion across grain boundaries due to a decrease in the granular exchange coupling [11].

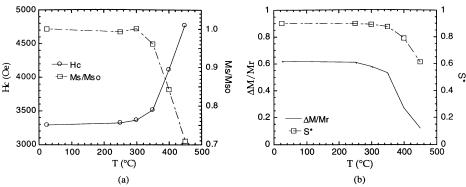


Fig. 4 (a) coercivity and normalized Ms to Mso, the value before annealing (b)  $\Delta M/Mr$  peak and S\* as functions of the annealing temperature for the CrMn(200Å)/CoCrPt(300Å)/CrMn(1000Å)/NiAl(1000Å) films deposited with no heating.

Another series of annealings were performed, in which the temperature was held constant at 400 °C and the sequence of annealing times were 1, 2, 4, and 8 minutes. The M-H loops and  $\Delta M$  curves are shown in Fig. 5 and Fig. 6, respectively. The  $\Delta M/Mr$  peak and Ms decrease, and the coercivity increases for the longer annealing times. At the annealing time of 8 minutes, the  $\Delta M$  curve is significantly flatter and the coercivity increases by 38%, to about 4600 Oe. The decrease in the Ms is about 15%. The difference in the percentage of the increase in the coercivity from the decrease in the Ms is larger than the above case. The improvement in grain isolation apparently contributes even more to the coercivity increase. These annealing conditions appears to be sufficient to drive the CrMn diffusion along the CoCrPt grain boundaries but not quite enough to overcome the energy barrier to enter the bulk of the CoCrPt grains. The post-deposition annealing of samples prepared without substrate preheating yields better properties than depositing films at elevated temperatures. This approach significantly lowers the granular exchange coupling, dramatically increases the coercivity, but causes a smaller decrease in Ms.

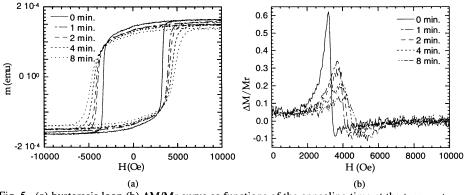


Fig. 5 (a) hysteresis loop (b)  $\Delta$ M/Mr curve as functions of the annealing time at the temperature of 400 °C for the CrMn(200Å)/CoCrPt(300Å)/CrMn(1000Å)/NiAl(1000Å) films deposited without substrate preheating.

#### CONCLUSIONS

Inserting a CrMn intermediate layer between the CoCrPt magnetic layer and the NiAl underlayer improves the CoCrPt ( $10\overline{1}0$ ) texture and increases the coercivity. Applying substrate preheating to 260 °C lowers the granular exchange coupling but degrades the Co-alloy texture. Post-deposition annealing was found to be an even better method for lowering the intergranular exchange coupling. Under the optimal annealing conditions found in this study, the  $\Delta M/Mr$  peak dropped to almost zero and the coercivity increased by 38%, to 4600 Oe, while the Ms moderately decreased by 15%. CrMn interdiffusion into CoCrPt grain boundaries is believed to increase the grain to grain isolation. The exchange coupling between grains seems to be correlated with the Co-alloy in-plane texture. The better the texture, the stronger the exchange coupling.

#### **ACKNOWLEDGMENTS**

This material is based upon work supported in part by Seagate Technology, Inc. and by the Data Storage Systems Center of Carnegie Mellon University under a grant from the National Science Foundation # ECD-8907068. The government has certain rights to this material.

#### **REFERENCES**

- M. Futamoto, N. Inaba, and A. Nakamura, Inst. Elect. Infor. Comm. Eng. (Japan) MR96-43, pp 47 (1996).
- 2. J. Nakai, E. Kusumoto, M. Kuwabara, T. Miyamoto, M. R. Visokay, K. Yoshikawa, and K. Itayama, IEEE Trans. Magn. 30, pp 3969 (1994).
- 3. M. F. Doerner, T. Yogi, D. S. Parker, S. Lambert, B. Hermsmeier, O. C. Allegranza, T. Nguyen, IEEE Trans. Magn. 29, pp 3667 (1993).
- 4. A. Murayama, S. Kondoh, and M. Miyamura, J. Appl. Phys. 75, pp 6147 (1994).
- 5. T. Kawanabe, et al, IEEE Trans. Magn. 26, pp 42 (1990).
- 6. Y. C. Feng, D. E. Laughlin, and D. N. Lambeth, IEEE Trans. Magn. 30, pp 3948 (1994).
- 7. L.-L. Lee, D. E. Laughlin, and D. N. Lambeth, to be published by IEEE Trans. Magn.
- L.-L. Lee, D. E. Laughlin, L. Fang, and D. N. Lambeth, IEEE Trans. Magn. 31, pp 2728 (1995).
- 9. J. Zou, D. E. Laughlin, and D. N. Lambeth, to be published by IEEE Trans. Magn.
- 10. D. Gupta in <u>Diffusion Phenomena in Thin Films and Microelectronic Materials</u>, edited by D. Gupta and P. S. Ho, Noyes Publications, Park Ridge, New Jersey, 1988, pp 33-51.
- 11. J.-G. Zhu and H. N. Bertram, J. Appl. Phys. 69, pp 6084 (1991).