Correlation between Noise and Microstructure in CoCrTa Media

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A series of identical $\text{Co}_{82}\text{Cr}_{13}\text{Ta}_5/\text{Cr}$ thin-film longitudinal media were deposited at substrate temperatures (T_{sub}) of room temperature (RT), 150°C, and 250°C respectively. Tracks were written at linear densities of up to 250 kfc, and the recording performance was examined. An inverse relationship was observed between T_{sub} and noise levels. A variety of electron microscopy techniques were employed on the same films to investigate the origin of this relationship. Spin-echo nuclear magnetic resonance (NMR), TEM after wet chemical etching, and energy-dispersive X-ray (EDX) analysis reveal the existence of compositional separation in the media deposited at elevated temperatures (250°C). This causes a reduction in the intergranular exchange coupling as detected in ΔM measurements. The reduction can be directly observed in Lorentz images in the form of smaller magnetic clusters. Cluster size has a strong effect on the written tracks. Lorentz images for each medium show a strong correlation between the cluster size and the noise measured.

Key words: CoCrTa/Cr media, TEM, domains, compositional separation

Introduction

The reduction of media noise is of paramount importance for high density CoCr-X longitudinal recording media¹⁾⁻³⁾. By changing the substrate temperature in a series of otherwise identical $\text{Co}_{82}\text{Cr}_{13}\text{Ta}_5/\text{Cr}$ hard-disk media a substantial increase in SNR was observed. It is widely accepted that this increase in SNR as a function of T_{sub} is related to a lessening of the intergranular exchange coupling, which in turn allows for the formation of smaller magnetic clusters^{4),5)}. In this paper, we employ a range of experimental techniques (TEM, spin-echo NMR and bulk magnetic) on the same samples upon which the media recording performance had been

examined, in order to investigate the origin of the reduction in cluster size and to correlate domain structures observed with the recording performance.

Experimental

The thin film media were sputtered onto Al/NiP substrates at various T_{sub} (room temperature, 150 °C and 250 °C) with argon pressure of 5 mTorr. The CoCrTa layer thickness was 20 nm and the Cr underlayer thickness was 25 nm. Bulk magnetic characterisation was carried out using a VSM. A read-write tester was used to examine the recording performance up to linear densities of 250 kfc. A write head with a gap length of 0.5 μm and core width of 2.9 μm was used at a flying height of 50 nm. The main magnetic characteristics of the media studied are presented in Table 1. The method used to produce samples suitable for TEM analysis was as follows; the Al substrate was polished to a thickness of 100 μm , a 3x3 mm coupon was cut and dimpled from the substrate side until the NiP layer appeared. This region of the NiP was then removed in 40% conc. HNO_3 , producing a transparent region (approx. 200x200 μm) which was supported by the remaining Al substrate. Samples of written tracks and dc erased regions were prepared. The microscope used for Lorentz observations was a modified JEOL 2000FX operated at 200 kV and equipped with a non-immersion pole-piece. Energy dispersive X-ray (EDX) analysis was performed on a JEOL 2010 FE-TEM capable of producing a 1.5 nm probe at the sample surface. Preferential wet chemical etching of Co was achieved by dipping the films in aqua regia for a number of hours.

Fig. 1 Lorentz TEM images of tracks written in the three media.

Medium	T _{sub} (°C)	H _c (Oe)	B _s (G)	SNR (dB)
375-60 (A)	RT	880	5565	12
375-44 (B)	150	1140	5309	22
375-28 (C)	250	1900	5091	25

Table 1 Main media parameters

Results and Discussion

Figure 1 presents Lorentz TEM images of written tracks (running vertically) in all three media. The Fresnel (defocus) mode was chosen because it generates greater contrast from such thin films. For media A, magnetic contrast corresponding to large magnetic clusters is observed all across the film, thus making it difficult to delineate the written tracks. No difference in contrast is observed in the written region and the adjacent d.c. erased region. At elevated temperatures the contrast is somewhat different. In media B, still much cluster contrast is observed in the d.c. erased region, but its size is reduced and now the track itself is clearly discernible. In media C, virtually no d.c. erased contrast is observed and bit transitions within the track are clearly seen. We explain this observed behaviour, as others have also done, in terms of a reduction in magnetic cluster size, which is the basic magnetic unit attainable and thus the limiting factor as far as domain size is concerned⁶⁾. The decrease in magnetic cluster size is more easily obtained when Lorentz images of d.c. erased samples (no written tracks present) are observed. A decrease in average cluster size from 470 to 70 nm was measured in media A and C respectively. This corresponds well with magnetic force microscopy findings previously attained on similar media⁷⁾. From close examination of the track images, a reduction in cluster size has two effects; firstly the background d.c. erased region contributes considerably less contrast; and secondly the track itself is better defined. The edge is straighter and a smaller bit size is more readily attainable. It is obvious that the structure of the domains supported in media C would give rise to lower noise signals and allow a larger SNR to be realised. Thus from fig. 1 we can clearly assume that cluster size is directly related to the noise levels measured in each media. However thus far we have gained no information as to why the cluster size should reduce and it is this that we have studied in the remainder of this study.

From Table 1, it is observed that the in-plane coercivity is seen to increase monotonically with T_{sub}, suggesting an onset of less cooperative behaviour at elevated growth temperatures, whilst at the same time the SNR increases. Compositional separation (CS) due to increased atom mobility during growth can be one origin of such behaviour^{8),9)}. An indicator of the presence of CS in binary CoCr alloys is an increase in B_s.

This is not observed in our ternary alloy system in which B_s actually decreases as a function of T_{sub} and the probable cause of this is the formation of non-magnetic (or weakly ferromagnetic) phases during the separation process. We endeavored to provide evidence of the existence of CS through a number of experimental methods.

⁵⁹Co spin-echo nuclear magnetic resonance (NMR) is an established technique for probing the atomic environment of Co in ferromagnetic alloys and multilayers^{10),11)}. Spin echo signals from these media proved difficult to attain, although a significant increase in signal at around 190 MHz was observed for media B and C and not for the room temperature grown media A. The difficulties encountered in this measurement could be due to the larger than usual Ta addition (thus reducing M_s), the thinness of the magnetic layer (20 nm) or the measurement temperature of (77 K). It is likely that all these parameters can adversely effect the intensity of echo signal. However a main peak at around this frequency was previously measured for CoCrTa, although a thicker film was involved¹²⁾. Thus we believe that this peak is not an artifact but rather results from the existence of ferromagnetically enriched microregions which are separated from each other by areas that are either non-magnetic or weakly ferromagnetic. More conclusive evidence of the presence of CS at elevated growth temperature was found from TEM analysis. Figure 2 is a TEM image of the microstructure of media C following preferential chemical etching of Co in an aquaregia solution (0.6N HCL + 0.06N HNO₃). The bright areas correspond to ferromagnetically enriched regions. They form a fine and dense structure across the whole film. This chemically etched pattern is somewhat dissimilar to the better known chrysanthemum pattern (CP) observed in (thick) perpendicular CoCr films¹³⁾. We believe however that we are observing the same phenomenon of which the CP pattern is just one manifestation.

Fig. 2 TEM image of media C following etching.

Indeed a dense honeycomb pattern has been previously observed in CoCrTa/Cr and CoCr/Cr films^{12),14)}.

To further confirm the existence of ferromagnetically enriched regions, we carried out EDX analysis in a field emission TEM operated at 200 kV. In order to achieve quantitative results from this analysis, it was necessary to remove the Cr underlayer. This was achieved by repeated low angle (10°) Ar ion-milling followed by checking the mean composition until it agreed with the nominal composition given for the media. The global composition was checked by acquiring spectra from a large area consisting of many tens of grains. Subsequently, a 1.5 nm electron probe was used for spot analysis across a number of grains in this area. Taking into account beam broadening, we can predict a 2 nm accuracy in this measurement. A series of spectra were taken for each grain, analysed and considerable differences in the relative concentrations of Co and Cr were observed.

Table 2 presents averaged EDX data obtained. It is clear that there is substantial Cr segregation to the grain boundary, resulting in a ferromagnetically enriched grain interior. When the spectra were analysed individually, it was also clear that there were some small Cr enriched regions within the grain which could reduce intragranular exchange coupling and is in keeping with some previous results and also with the TEM image presented in figure 2. Figure 3 plots the results from the spot analysis averaged over the seven grains investigated. The Cr concentration at the grain boundary is almost double that found in the interior, whilst the Co concentration is seen to increase by up to 25% in the centre of the grain. No inference is made on the possible separation of Ta atoms from this study, though others have shown that Ta preferentially separates to the Cr enriched region¹⁵⁾ thus further reducing those areas' magnetisation. This argument agrees well with our observation of reduced B_s at elevated growth temperatures.

From the results presented in the previous section it is clear that CS exists in CoCrTa/Cr films deposited at elevated temperatures. To study the effect that this CS has upon the granular interactions we performed ΔM measurements. This type of measurement has shown to be invaluable in terms of clarifying the sort and strength of granular interactions present in thin magnetic films¹⁶⁾ which have a direct bearing on the size of magnetic cluster that the film can support. The results are presented in Figure 4. Media A and B exhibit strong positive peaks, indicative of intergranular exchange coupling.

Spectra type	at. % Co	at. % Cr	at. % Ta
Area	81.6 ± 0.4	12.8 ± 0.4	5.6 ± 0.3
Boundary	78.6 ± 1.3	14.7 ± 1.1	6.6 ± 0.5
Interior	88.6 ± 0.7	8.8 ± 0.5	6.5 ± 0.4

Table 2 Relative concentrations of Co and Cr from EDX analysis.

Both media also exhibit an undershoot to negative values which may be explained by the presence of large clusters of grains which are coupled by considerably weaker exchange interactions and may switch at lower fields. In contrast however, media C exhibits only negative values, suggesting that magnetostatic intergranular coupling is the dominant mechanism for this media. It should be noted that none of the plots start at $\Delta M=0$ and this is due to incomplete a.c. erasure which is difficult to achieve for strongly coupled films. However of most importance is the relative differences between each plot and that is clearly illustrated in fig. 4.

Conclusions

We have shown that by undertaking a full range of systematic microstructural, bulk magnetic and recording experiments on a single series of identical CoCrTa/Cr media deposited at increasing substrate temperature it is possible to show that T_{sub} alone is responsible for the differences in recording performance measured.

Fig. 3 Plots of Co and Cr concentrations across grains in media C.

Fig. 4 Delta M plots for the three media studied

In particular, an elevated growth temperature induces compositional separation between Co and Cr, which we confirmed by the use of spin-echo NMR, wet chemical etching and EDX. The presence of CS directly effects the coupling mechanism of the media and from ΔM measurements it is clear that in these media, the dominant mechanism shifts from exchange to (weaker) magnetostatic as T_{sub} increases. It is these magnetostatic interactions that allow the existence of smaller magnetic clusters and results directly in better defined written information, as observed by Lorentz microscopy and thus lower noise levels.

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