Improved Grain Isolation of Co₈₀Pt₂₀ Films via Grain Boundary Diffusion of Mn

Jie Zou, Bo Bian, David E. Laughlin, and David N. Lambeth

Abstract—Post-deposition rapid thermal annealing (RTA) was performed on the Cr-less Co20at%Pt alloy with a Mn diffusion layer on top. A Ku value of about 8×10^6 erg/cm³ was obtained for the as-deposited film and it decreased only up to about 10% after annealing. The high intergranular exchange coupling of the as-deposited CoPt film was suppressed by RTA and the coercivity increased dramatically from about 2200 to 6000 Oe. The energyfiltered TEM plane-view images showed depletion of Co and aggregation of Mn at the CoPt grain boundaries. This is a direct evidence supporting preferential diffusion of materials from adjacent layers into magnetic grain boundaries.

Index Terms—Annealing, anisotropy, CoPt, Mn, segregation.

I. INTRODUCTION

T O ACHIEVE low noise in hard disk media it is important that the magnetic film be composed of small and magnetically decoupled grains. Cr self-segregation to the grain boundaries has been the widely adopted approach to achieve magnetic grain isolation for a variety of CoCrX alloys. However, the addition of Cr to the Co alloys detrimentally decreases the crystalline anisotropy constant [1]. This makes this approach unfavorable with respect to thermal stability of future high density media.

Since grain boundary diffusion is known to have a lower activation energy than bulk lattice diffusion, the diffusion coefficient in grain boundaries can be orders of magnitude higher than in the bulk of the grain [2]. Thus under certain processing conditions, it may be possible to diffuse a significant amount of nonmagnetic materials from adjacent layers into the grain boundaries of the magnetic film to magnetically decouple the grains, while diffusing only a small amount into the bulk of the magnetic grains. Therefore, grain boundary diffusion may be a method of achieving grain isolation without a significant decrease in the anisotropy constant.

In earlier studies, it has been shown that, driven by postdeposition rapid thermal annealing (RTA), preferential diffusion from adjacent Cr, CrMn, or Mn layers was effective at improving the CoCrPt magnetic properties [3]–[6]. However, in those results, the role of Cr from the Co–Cr alloy versus Cr or Mn from an adjacent layer was unclear. In this paper, the role of grain boundary diffusion of Mn from an adjacent layer was investigated by using a Cr-less Co alloy.

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II. EXPERIMENTAL

The film structure on a glass substrate consisted of a 1000 Å NiAl underlayer, a 200 Å CrTi intermediate layer, a 150 Å CrPt magnetic layer, a 200 Å Mn diffusion layer, and a 200 Å CrTi protection capping layer. Samples were deposited by using RF diode sputtering without substrate preheating. RTA treatments were performed on the samples at atmospheric pressure under Ar flow. Magnetic and microstructural properties were examined for different annealing conditions.

The composition of the CoPt films was Co-20at.%Pt, determined by energy dispersive x-ray spectroscopy (EDX). The in-plane magnetic properties of the samples were measured using vibrating sample magnetometry (VSM), torque magnetometry and alternating gradient magnetometry (AGM). The plane-view elemental mapping was obtained by energy-filtered transmission electron microscopy (EFTEM).

III. RESULTS AND DISCUSSION

The same sequence of RTA treatments with increasing annealing temperatures for a fixed time of 1 minute were performed on a typical sample with the structure described in Section II (with Mn) and a reference sample with the similar structure but without the Mn diffusion layer. Fig. 1 shows their magnetic properties as functions of the annealing temperature. For the sample with Mn, Hc increased quickly up to about 6000 Oe and Ms decreased for the annealing temperature higher than 300° C. In the as-deposited state, the film had a very high S^{*} value of 0.96, which suggests the magnetic grains were highly exchange coupled [7]. This is expected since there is no Cr in the magnetic alloy. The S^* maintained the high value up to 350°C, and then dropped sharply to 0.78 after the 400°C annealing, reflecting the breakup of large magnetically coupled clusters of grains and reduced collective magnetic switching behavior. The sharp positive peak and maximum slope of the $\delta M(H)$ curve decreased significantly only after the RTA at 400°C, consistent with the S^* data, which suggests that the intergranular exchange coupling in the CoPt layer was suppressed by RTA [8].

For the reference films without the Mn layer, Hc and Ms exhibited similar behavior as a function of the annealing temperature but the change in the magnetic properties was much smaller when compared to the sample with the Mn layer. S^* was almost constant, which is consistent with the δM measurements. Therefore, it is apparent that the dramatic improvement in magnetic properties after RTA is due to the presence of Mn and most likely because of the reduction in intergranular exchange coupling through the diffusion of Mn into the CoPt grain boundaries. For the reference sample without the Mn layer, the

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Fig. 1. Hc, Ms, S^* , and $\delta M(H)$ curves as a function of the annealing temperature for the samples with and without the Mn layer.

increase in Hc after annealing may be due to the decrease in the crystallographic defects in the CoPt layer with annealing, even though magnetic grain isolation did not improve. This effect may be a factor for the increase in the coercivity of the sample with the Mn layer as well, but should be a smaller one than the improved magnetic grain decoupling by RTA.

The effect of annealing time on magnetic properties was also investigated, as shown in Fig. 2. The annealing temperature was constant at 400°C. Hc increased and Mc decreased for longer annealing time. The ratio of Hc increase was much larger than the magnetization decrease, e.g., for the 135-second annealing, Hc increased more than 170% while Ms decreased only about 30%. The initial decrease in was fairly rapid and then the decrease rate slowed for longer annealing time. This is consistent with the grain boundary diffusion kinetics in polycrystalline films. The diffusant could move along the CoPt grain boundaries very fast causing the sharp decrease in the magnetic moment there. But after the magnetic moment in the CoPt grain boundaries drops to zero, the further buildup of nonmagnetic

Fig. 2. Hc, Ms, S^* , S, and $\delta M(H)$ curves as a function of the annealing time at 400°C for the sample with the Mn layer.

diffusant in the grain boundaries would result in no change in the average magnetization. The simultaneous direct bulk diffusion from the adjacent layers and the lateral diffusion from the grain boundaries to the bulk of the grains, which caused the continued decrease in the average magnetization, were much slower. S^* significantly decreased and the positive peaks in $\delta M(H)$ curves were flattened for longer annealing times, indicating that the intergranular exchange coupling was reduced. Remanence squareness S only slightly reduced as the annealing time increased.

The activation volume of magnetic reversal, V_a has been shown to be correlated with the intergranular exchange coupling in the media and the media noise [9], [10]. Smaller V_a that is close to the physical volume of the magnetic grains indicates that they are less exchange coupled, and the media normally will have smoother transitions and thus show lower noise. Fig. 3 shows V_a measured by using the wait time method [9] as a function of the reversal magnetic field for the as-deposited and annealed films. The physical volume was calculated from the average grain size (~ 15 nm) measured from TEM images and the thickness of the CoPt film (~ 15 nm). Activation volume



Fig. 3. Activation volume for as-deposited and annealed films.



Fig. 4. Anisotropy constant versus annealing time at the temperature of 400° C.

for the annealed film was significantly smaller than that for the as-deposited film, and a longer annealing time further reduced $V_{\rm a}$ approximately to the physical volume. This is consistent with the observation from the previous magnetic property measurements that the magnetic grains were decoupled as the result of the RTA.

Fig. 4 shows the experimental data of uniaxial crystalline magnetic anisotropy constant Ku, measured by using the out-of-plane torque method [11] in a field of 19 KOe, versus annealing time at 400°C. Anisotropy slightly decreased for a longer annealing time. Even for the longest annealing time of 135 seconds, Ku dropped only about 10%. This is consistent with the grain boundary diffusion model. Furthermore, this clearly shows the advantage of using RTA to achieve magnetic grain isolation over adding significant amount of Cr into Co-alloys to cause Cr self-segregation, which results in a significant decrease in Ku [1].

The anisotropy constant for the Co-at20%Pt films was almost twice of that of the bulk value for pure Co. This could be due to the formation of the ordered Co_3Pt phase [12], [13]. However, no (0001) superlattice peak was observed in the XRD spectrum of a 2600 Å thick Co-20at.%Pt film sputtered on a oxidized Si (100) substrate with no substrate preheating. Only mixed CoPt (10<u>1</u>0) and (0002) hcp textures were revealed. This indicates that the Co_3Pt ordered phase [13] was probably not present. The CoPt films on glass substrates with the original film structure showed hcp (10<u>1</u>0) texture only and annealing had no apparent impact on the texture.



Fig. 5. The plane-view mapping image of Co and Mn concentration in the annealed CoPt film.

To obtain the direct evidence of grain boundary diffusion of Mn in the CoPt alloy, energy-filtered TEM [14] was used to acquire the elemental mapping. A sample consisting of a 1000 Å thick NiAl underlayer, a 300 Å thick CoPt magnetic layer, a 200 Å thick Mn diffusion layer, and a 200 Å thick CrTi protection layer was deposited on a oxidized Si substrate with no preheating. No CrTi layer was deposited in direct contact with the CoPt layer to prevent the diffusion of Cr into CoPt layer. This is necessary, because Cr cannot be distinguished from Mn due to the overlap of the energy loss peak of Mn with that of Cr. When preparing the TEM sample, the CrTi and Mn top layers were etched from the annealed sample by ion milling. Fig. 5 shows the plane-view mapping image of Co and Mn concentration in the CoPt film obtained by energy-filtered TEM. The depletion of Co at the CoPt grain boundaries is clearly visible. The width of the depletion region is typically about 2-3 nm. Although the contrast of the image is not very good, Mn segregation at the CoPt grain boundaries is visible. Because Mn atoms can only diffuse from the top layer into the CoPt layer, this direct evidence clearly supports the grain boundary diffusion model that has been strongly suggested by the magnetic data. The preferential diffusion of Mn into the CoPt grain boundaries is thus proved to be the major factor in the reduction of intergranular exchange coupling and the increase in Hc.

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