Structural Characteristics of Bias Sputtered CoCrTa/Cr Films

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Abstract -- Crystallographic texture and lattice spacings of bias sputtered CoCrTa/Cr and Co/Cr thin films have been studied by x-ray diffraction. While only an insignificant change in the Co lattice spacing for the Co/Cr films was observed as the substrate bias voltage was increased, the increase in the CoCrTa lattice spacing for the CoCrTa/Cr films was substantial. The CoCrTa films showed nearly unchanged lattice spacings after a vacuum annealing for film stress release. The lattice increase appeared to be linearly proportional to the Ta content (ranging up to about 7%) in the CoCrTa film. This implies that the Ta atoms tend to substitute randomly onto the hexagonal sites within the CoCrTa crystal grains, and that very little, if any of the Ta segregates to the grain boundaries.

I INTRODUCTION

The microstructure of sputtered films largely determines their magnetic and recording properties [1-3]. Fundamental to realizing ultra-high density recording media is to achieve well isolated granular thin films [4]. Several investigations have shown that Cr segregates to the grain boundaries in sputtered CoCr-based alloy films [5-6]. The CoCrTa alloy has proven to be a particularly popular medium as recording studies indicate a very low recording noise. The question then naturally arises as to whether or not Cr or Ta segregates to the grain boundaries in this medium to provide grain isolation.

Recently, we have found that RF substrate bias can significantly change the composition of sputtered CoCrTa films, and that the magnetic properties of such films can be correlated with their composition [7]. In this paper, we report on our x-ray diffraction studies of the structural characteristics of the CoCrTa films as well as pure Co films prepared under the identical sputtering conditions. The focus here is on the crystallographic texture of the films and the change in lattice spacings as bias voltage is used. This provides indirect evidence as to whether phase segregation occurs at the grain boundaries in these films.

II EXPERIMENTAL PROCEDURE

Films were prepared by RF diode sputtering in a Leybold-Heraeus Z-400 sputtering system. The base pressure was about 7×10^{-7} Torr and the Ar sputtering pressure was fixed at 10 mTorr. The sputtering targets were 3" in diameter and included Cr, $Co_{86}Cr_{12}Ta_2$, $Co_{82.8}Cr_{14.6}Ta_{2.6}$, and pure Co. The cathode voltage ranged approximately from -1250 V to -1500 V. The sputtering power density on the targets was fixed at about 2.3 W/cm^2. The Cr underlayers were 1000 Å thick for all the films and were prepared on Corning 7059 glass substrates at a constant RF substrate bias (-200 V). Magnetic films (300 Å thick) were sputtered subsequently at various RF substrate bias voltages (0 to -300 V) either from the pure Co or from the Co alloys targets. The film growth rate decreased monotonically from 120 Å/min to 30 Å/min as the substrate bias voltage was increased from 0 V to -300 V [7]. The sputtering time was varied in order to obtain the same film thickness. The crystal structure of the films was studied by x-ray diffraction using Cu Kα radiation. The magnetic and compositional properties of the films prepared from the $Co_{82.8}Cr_{14.6}Ta_{2.6}$ and $Co_{86}Cr_{12}Ta_2$ targets have been reported in Ref. [7].

Vacuum annealing to release the film stress was performed by using a heater in a 1×10^{-6} Torr vacuum. The sample was heated from room temperature up to 300 °C in 30 minutes, and then held at 300 °C for an hour. The power was then switched off and the sample cooled back to room temperature within about 3 hours.

III RESULTS AND DISCUSSIONS

Figs. 1 and 2 show the x-ray diffraction spectra of the films prepared from the $Co_{82.8}Cr_{14.6}Ta_{2.6}$ and the Co targets, respectively. Due to the Ar bombardment, a single, and strong {110} texture forms in the bias sputtered Cr underlayer [8]. The subsequently deposited CoCrTa and Co films have two dominant textures, namely, {10$\bar{1}$1} and {10$\bar{1}$0}. The {10$\bar{1}$1} texture is the result of the epitaxial growth of the Co-based alloy film on the {110} textured Cr underlayer [3]. The cause of the {10$\bar{1}$0} texture formation, however, is not certain. The same crystallographic textures were also observed for the films prepared from the $Co_{86}Cr_{12}Ta_2$ target.
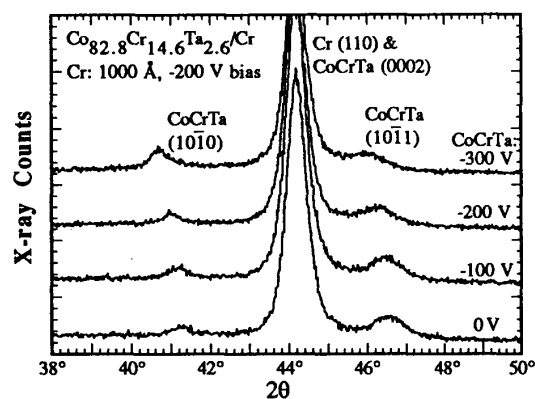
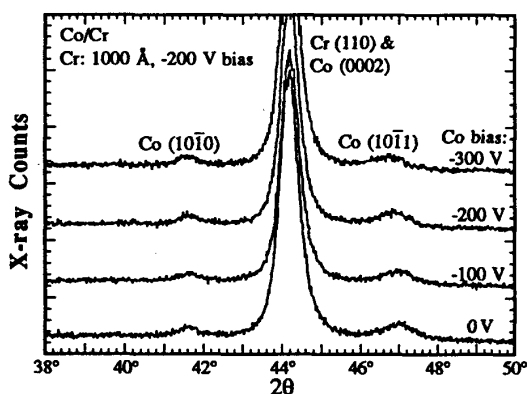Fig. 1 X-ray diffraction spectra for the bias sputtered CoCrTa/Cr films.

Fig. 2 X-ray diffraction spectra for the bias sputtered Co/Cr films.

Fig. 1 also shows clearly that as the substrate bias increases, the diffraction peaks for the CoCrTa ($10\bar{1}1$) and ($10\bar{1}0$) planes shift to the lower values of 2θ, indicating an increase in the d-spacing. This is shown explicitly in Fig. 3. The d-spacings of the ($10\bar{1}1$) and ($10\bar{1}0$) planes can be seen to increase with increasing bias voltage for the CoCrTa alloy films (similar results were also obtained for the films prepared from the $Co_{86}Cr_{12}Ta_2$ target). At the same time, however, the increase in the d-spacings for the pure Co films is very small.

It has been observed that the Co alloy diffraction peaks shifted as the thickness of the Cr underlayer changed [8]. The shift was large when the Cr underlayer was very thin. For all the film samples in this study, however, the Cr underlayer thickness was kept the same and relatively large; thus the variation in the CoCrTa lattice spacing caused by the Cr underlayer thickness should be the same, and yet small.
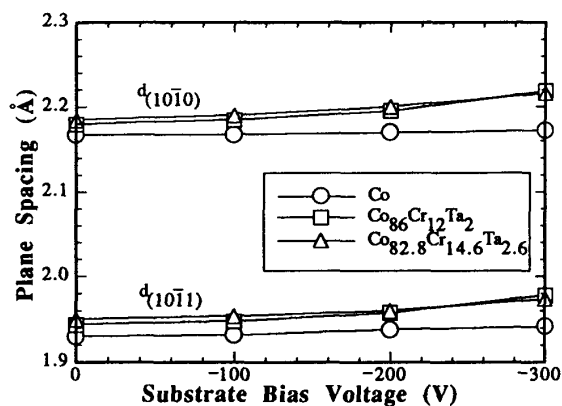

Fig. 3 CoCrTa and Co d-spacings vs. substrate bias voltage.

It has also been reported that substrate bias can slightly change the material lattice spacing by inducing stress and/or trapping Ar in the film [9]. Thus, the small change in the lattice spacings of the pure Co films can be regarded as the result of the film stress and Ar entrapment. Since the $Co_{86}Cr_{12}Ta_2$ and $Co_{82.8}Cr_{14.6}Ta_{2.6}$ films were prepared under the same sputtering conditions as the pure Co films, we would expect the same, small lattice spacing increase, due to the film stress and Ar entrapment, for both the pure Co and the CoCrTa films. Most of the increase in the CoCrTa lattice spacing, therefore, is more likely caused by a bias-induced compositional change, i.e., an increase in the Ta content in the films. Since Ta has a larger atomic size than Co or Cr, the lattice parameters and hence d-spacings would increase with increasing Ta content.

In order to confirm that the film residual stress causes only small changes in the CoCrTa spacings, vacuum annealing as described in the earlier section was performed. The annealing temperature and time used here were lower and shorter than those used in Ref. [10]; therefore, it could be expected that the annealing only released the film residual stress without causing a microstructural change as observed by Duan et al. [10]. We found that the lattice spacings for both the Co and the CoCrTa films remained nearly the same before and after the annealing, indicating that the film stress indeed caused only a small increase in the CoCrTa d-spacing.

Fig. 4 shows how the d-spacings for the ($10\bar{1}1$) and ($10\bar{1}0$) planes change with the Ta content (at. %) in the CoCrTa films. Only the Ta content was chosen as a variable since Co and Cr have nearly the same atomic size but Ta is much larger. The Ta content was determined using x-ray fluorescence spectroscopy with a 0.3 % accuracy [7]. The d-spacing was calculated by applying the Bragg's equation to the x-ray diffraction data. The contribution from the film stress and Ar entrapment has been removed by subtracting the corresponding increase in the d-spacing of the pure Co films for a given bias voltage. Fig. 4 shows that both the ($10\bar{1}1$)
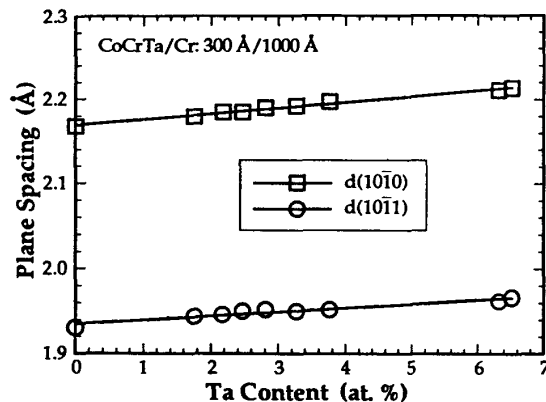
Fig. 4 D-spacings of the CoCrTa $(10\bar{1}0)$ and $(10\bar{1}1)$ planes vs. Ta content in the film.

and $(10\bar{1}0)$ d-spacings increase linearly with increasing Ta content over the range of 0 to about 7 at. %.

The dependence of the hcp CoCrTa lattice constants "a" and "c" on the Ta content calculated from the data in Fig. 4 is shown in Fig. 5. We can see that both "a" and "c" also increase linearly with increasing Ta content. Their increasing rates, i.e., the slopes, from fitting the data are 7.9×10^{-3} and 10.2×10^{-3} Å per Ta atomic percent, respectively. These are only slightly greater than the values predicted by Vegard's law, 4.1×10^{-3} and 7.0×10^{-3} Å per Ta atomic percent, respectively [11]. This suggests that the Ta atoms tend to substitute randomly onto the hcp crystal sites [11]. It is, therefore, doubtful that the Ta atoms (0 to about 7 at. %) segregate to the grain boundaries in these bias sputtered CoCrTa films. However, this does not rule out the possibility of Cr segregation to the grain boundaries [5-6], nor does it rule out the possibility that the increase in the Ta content may influence the Cr segregation.
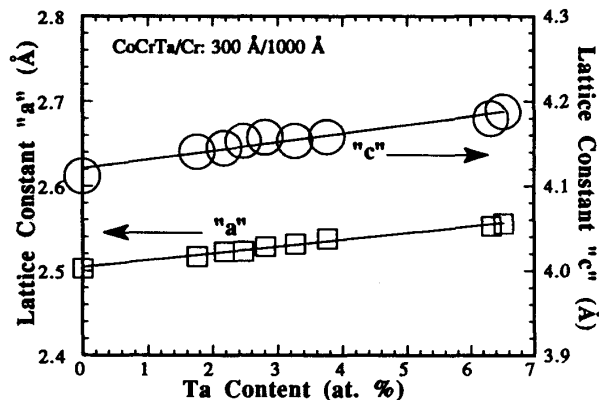

Fig. 5 CoCrTa lattice constants "a" and "c" vs. Ta content. The vertical size of each data point represents the measurement error of the lattice constants.

IV CONCLUSIONS

Bias sputtered Cr underlayers on glass show a strong {110} texture. The subsequently bias-sputtered Co alloy films have two dominant textures, $\{10\bar{1}1\}$ and $\{10\bar{1}0\}$. Due to the substrate bias, the lattice spacing of the Co alloy increases. The increase in the d-spacing due to the change in the film composition is more significant than that caused by the film stress and Ar entrapment. Our data show unambiguously that the CoCrTa lattice spacing increases linearly with increasing Ta content (0 to 7 at. %) in the film. This suggests that the Ta atoms randomly substitute on the atomic sites within the CoCrTa grains and hence, very little, if any, of the Ta segregates to the grain boundaries.

ACKNOWLEDGMENTS

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