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TEM INVESTIGATION OF Co–Cr FILM MICROSTRUCTURE [a)]

C. HWANG [b)], D.E. LAUGHLIN [b)], P.V. MITCHELL [c)], A. LAYADI [d)] K. MOUNTFIELD [e)], J.E. SNYDER [f)] and J.O. ARTMAN [f)]

Magnetics Technology Center, Carnegie-Mellon University, Pittsburgh, PA 15213, USA

Transmission electron microscopy (TEM) was used to reconstruct the microstructue of Co–Cr thin films. It was found that the initial layer consists of randomly oriented small crystal grains, that further growth favors well-aligned grains and that microstructural details may be correlated with inferences from ferromagnetic resonance (FMR).

1. Introduction

Co–Cr thin films have been proposed as media suitable for perpendicular recording. This study correlates interpretations of ferromagnetic resonance (FMR) spectra [1] with the microstructural properties of Co–Cr thin films. Evidence from FMR and sputter-etching suggests that film growth proceeds by 1) the formation of a transition layer directly on top of the substrate, followed by 2) the deposition of a bulk layer which is characterized by the desired film properties [2]. Previous TEM investigations have revealed the presence of the well-known columnar stucture [3]. However, our motivation in this study is to establish the nature of the three-dimensional microstructure which can then be compared to inferences from our FMR results. Hence, a sequence of Co–Cr films of various thicknesses was examined in plane view and in cross-section by TEM; these procedures were supplemented by some electron diffraction observations.

2. Experimental procedure

A series of Co–Cr thin films, ranging in thickness from 10 to 1000 nm, was rf-sputtered from a composite target (22 at% Cr) onto glass substrates. The sputtering conditions were kept constant for all specimens and the sputtering time was varied in order to get films with different thicknesses. Plane view specimens were easily handled requiring only ion milling before TEM examination. Cross-sectioned specimens were more difficult to prepare. In this case identical specimens were first laminated together with the film surfaces touching; then the cross section was thinned by lapping before the ion milling began. All samples were examined with a JEOL 120CX STEM.

3. Results and discussion

Fig. 1 shows a composite TEM view of the top, side and bottom of a 1028 nm thick film, together with the corresponding selected area electron diffraction patterns. It is clear that the bottom (initial) layer of the film consists of numerous randomly oriented crystalline grains resulting from random deposition of Co and Cr atoms on the amorphous glass substrate. Further growth of the Co–Cr thin film took place by the selective growth of those grains with crystallographic *c*-axes normal to the film surface at the expense of the remaining randomly oriented grains. As a result, the grains grew not only perpendicular to the substrate surface, but laterally as well, forming conically expanding columns with well aligned crystalline *c*-axes. This is also illustrated by the corresponding electron diffraction patterns of fig. 1, where the elimination of the (0002) and $(01\bar{1}1)$ rings from the top layer pattern is an indication of (0002) *c*-axis texture. This microstructure is typical of the entire thickness series, the structures of the thinner films (those < 60 nm) correlating better with the microstructure of the bottom layer, which comprises only randomly oriented crystal grains. This latter structurally distinct region could be the so-called "transition layer" for which the crystalline anisotropy is diminished, resulting in a layer with dominant shape anisotropy, as indicated by FMR study [1].

Statistical measurements of grain size in each specimen have also been carried out to study the grain growth during deposition. This is shown in fig. 2. Each datum point was averaged from at least 200 randomly chosen grain size measurements. While the overall crystal grain size correlates fairly well with the similar curve reported by Lodder et al. [3], we found that there is a significant increase of grain size after about 50 nm of film thickness. This finding is also consistent with our FMR data which correspond to a transition layer rang-

a. Research funded in part by the Magnetics Technology Center, Carnegie-Mellon University, Pittsburgh, PA 15213, USA

b. Department of Metallurgical Engineering and Materials Science.
c. Department of Physics; supported by a Hughes Fellowship while on leave from Hughes Research Labs, Malibu, CA 90265, USA.
d. Department of Physics; supported by a Government of Algeria Fellowship.
e. Department of Physics.
f. Department of Electrical and Computer Engineering.

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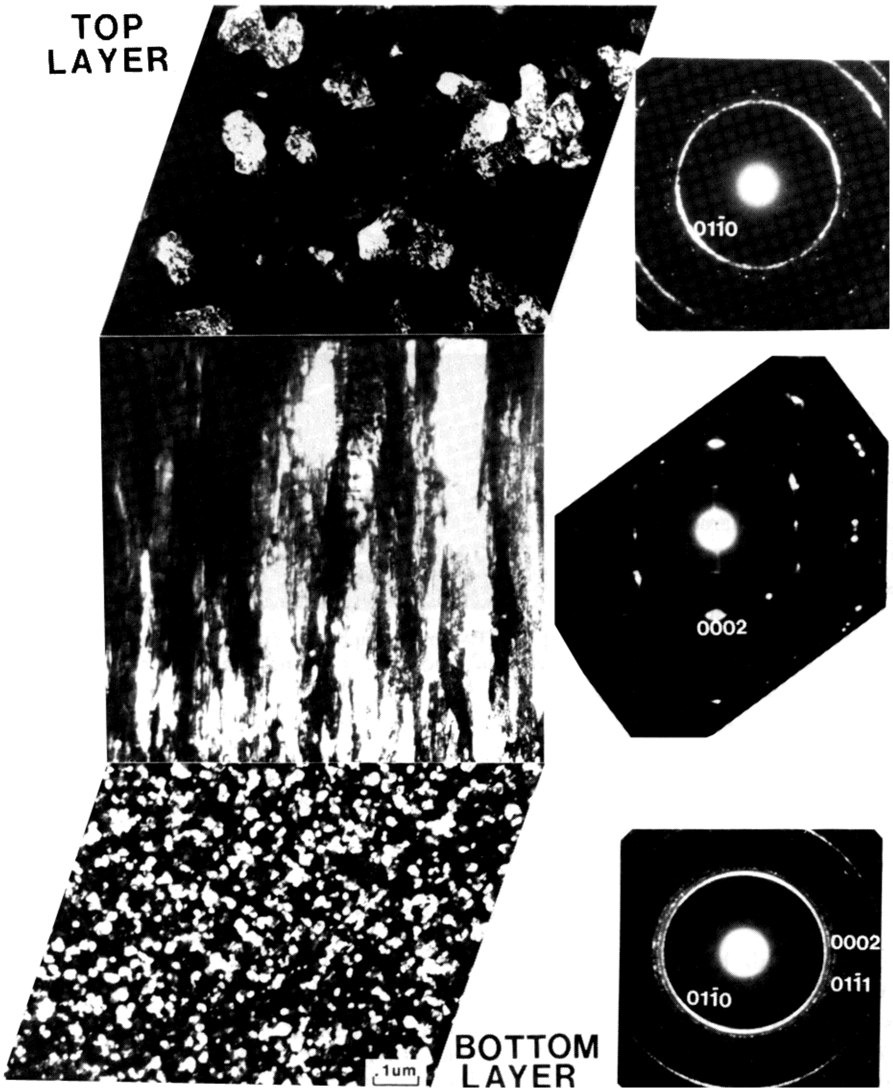

Fig. 1. TEM composite view (dark field) of the top, side and bottom of a 1028 nm thick Co–Cr film, together with corresponding electron diffraction patterns.

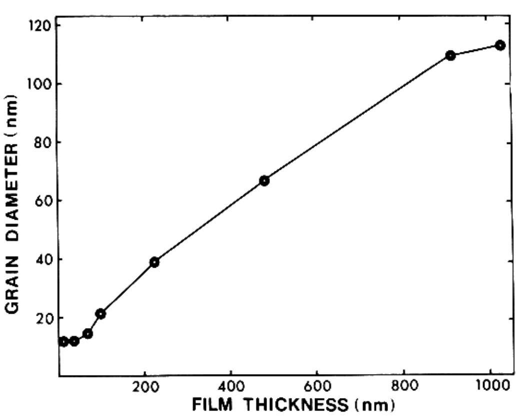

Fig. 2. Surface grain diameter as a function of film thickness.

ing from 20 to 40 nm in thickness. A comparison of grain size near the bottom layer of different thickness films indicates significant grain growth during deposition: 12 nm grain size for a 14 nm thick film and 16.5 nm grain size for 1028 nm thick film. This may indicate that, for thicker films, there is an annealing effect on the bottom layer stucture. Thicker films necessarily require longer deposition times; possibly annealing is promoted by the inevitable plasma heating during sputtering. This is not unexpected, since, in another paper [4] at this conference, we show that the transition layer is extremely sensitive to the preferential grain growth that occurs during annealing.

In general, as the films became thicker $\Delta\theta_{50}$ decreases; see table 1. Undoubtedly, as the well aligned

Table 1
Alignment parameters for the Co–Cr thickness series

Thickness (nm)	Number of FMR Lines	$\Delta\theta_{50}$ ** (deg)
14	1 *	17.2
39	1 *	10.8
67	1 *	10.7
96	4^{++}	7.4
223	2^{+}	5.8
481	4^{++}	9.4
913	2^{+}	4.8
1028	2^{+}	4.2

* Transition layer resonance; $^{++}$ multiple resonances including transition and bulk layer resonances; $^{+}$ transition and bulk layer resonances; ** about the [0002] X-ray peak.

bulk layer becomes thicker, it will dominate the X-ray rocking curve. The 481 nm thick specimen characteristics depart from this trend. This particular specimen is also, judging by FMR, a magnetically poor film with four resonances instead of the usual two (bulk and transition) which characterize films of this thickness. Electron diffraction patterns of this particular film also show unusually poor alignment of *c*-axis orientation even at the top of this film; see fig. 3 and compare with fig. 1. The extra diffraction spots may indicate the presence of phases other than hcp structure; these as yet unidentified structures may correlate with the extra FMR lines observed in this particular film.

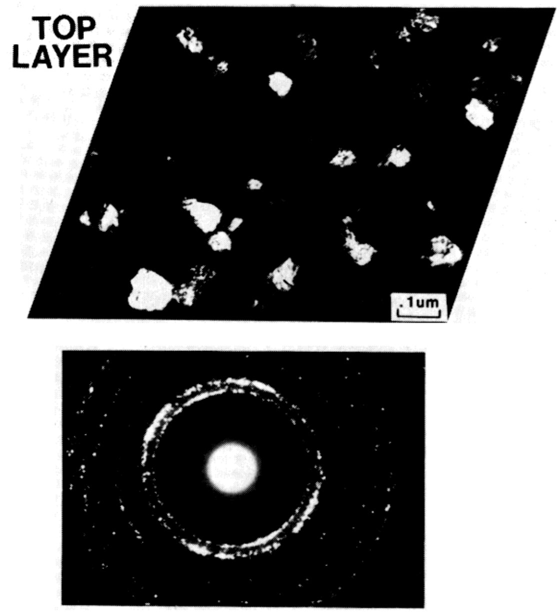

Fig. 3. TEM micrograph and diffraction pattern of the top layer grain structure for a 481 nm thick Co–Cr film.

4. Conclusion

The transition layer can be identified with the initial seed layer and contains small, randomly oriented grains. It is 40 to 60 nm thick; nevertheless, it can not be thought of as a sharp layer with a distinctive boundary – as in an oxide layer. Continued growth of film past the transition layer thickness is characterized by expanding conical grains and some grain growth within the transition layer.

[1] P.V. Mitchell, A. Layadi, N.S. VanderVen and J.O. Artman, J. Appl. Phys. 57 (1985) 3976.
[2] P.V. Mitchell, K. Mountfield, A. Layadi, J.E. Snyder and J.O. Artman, Digest of the ICMFS-11 (Asilomar).
[3] J.C. Lodder, T. Wielinga and J. Worst, Thin Solid Films 101 (1983) 61.
[4] P.V. Mitchell, A. Layadi, K. Mountfield, C. Hwang, J.E. Snyder and J.O. Artman, J. Magn. Magn. Mat. 54–57 (1986) these proceedings.