Transmission electron microscopy of Co-Cr films for magnetic recording

J.-W. Lee, B. G. Demczyk, K. R. Mountfield, and D. E. Laughlin
Department of Metallurgical Engineering and Materials Science and Magnetics Technology Center, Carnegie-Mellon University, Pittsburgh, Pennsylvania 15213

The microstructure of Co-Cr thin films for perpendicular magnetic recording has been examined by transmission electron microscopy in both conventional (bright field/dark field and selected area diffraction) and convergent beam electron diffraction modes. Results of as-deposited samples indicate that the films consist of well-oriented *c* axis structures. A range of microstructures (columnar morphology, transition layers, twins, etc.) have been observed. These and other results will be presented.

INTRODUCTION

Previous investigations[1–6] revealed that Co-Cr thin films suitable for perpendicular recording media consist of conical columnar structures with randomly oriented transition layers in the region closest to the film/substrate interface, and well-oriented *c*-axis layers on top of the transition layers. Other investigators,[7,8] however, have reported straight columnar grains. This would mitigate against the presence of transition layers in these films, since the crystallographically preferred orientation of the grains seems to be established early in the growth process. Indeed Co-Cr films without transition layers have been reported.[9] Hence, the present authors believe that the presence or absence of a transition layer should be subjected to an experimental investigation, utilizing the convergent beam electron diffraction (CBED) technique, since the beam size in this technique can be as small as 2.0 nm.

In this investigation, we have examined the microstructural details of Co-Cr thin films and analyzed the crystallography of individual grains of the films.

EXPERIMENT

Two particular Co-Cr films on glass substrates were prepared from an alloy target of Co-22% Cr (at. %) by a dc magnetron sputtering system. The deposition conditions were kept approximately constant; the depostion rates were 13.8 nm/min for specimen A and 16.4 nm/min for specimen B. The methods of specimen preparation for transmission electron microscopy (TEM) have been described elsewhere.[6] TEM was performed using a Phillips EM420T analytical electron microscope operating at 120 kV. The crystallographic orientation of the films was determined using CBED patterns.

RESULTS AND DISCUSSION

CBED patterns from the top of plane view specimens showed that all films contain grains of hcp structure ($P6_3/mmc$) and exhibit predominantly the *c* axes perpendicular to the film plane. The misorientation about the *c* axis between adjacent grains is always less than 5°. No cubic phase ($Fm\bar{3}m$) was detected on the top of plane view specimens.

Figures 1 and 2 are typical bright-field TEM micrographs of specimens A and B together with their representative CBED patterns, respectively. In both instances, the grain size is shown to be larger at the top of the films than at the bottom of the films. At the top of the specimens, the crystals are oriented along the [0001] zone axis (z). From Fig. 1, the grain size of sample A (top view) is in the range of 50–60 nm, while in the bottom view it is in the range of 10–30 nm. Figure 2 shows that the grain size at the top of sample B is much larger (50–60 nm) than that at the bottom (10–15 nm). In both cases, this difference in size implies a conical

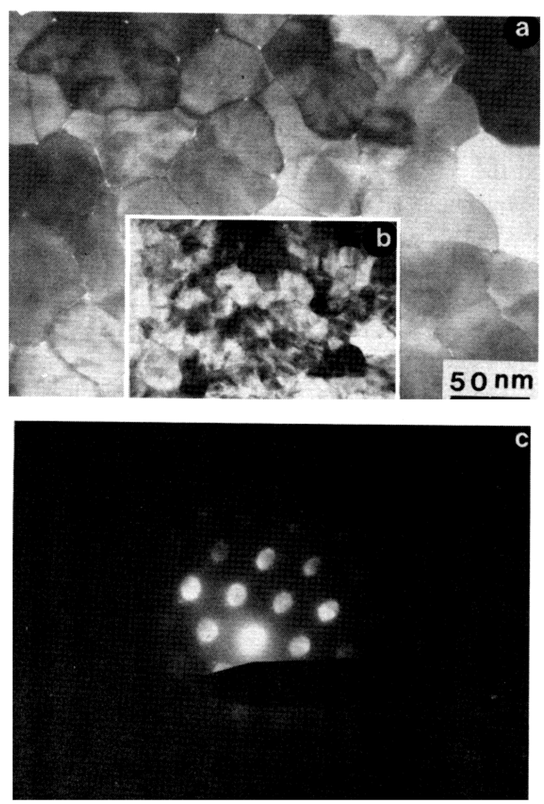

FIG. 1. TEM images of Co-Cr film (specimen A) at the top (a) and at the bottom (b) together with a representative CBED pattern (zone axis near [0001]) (c).

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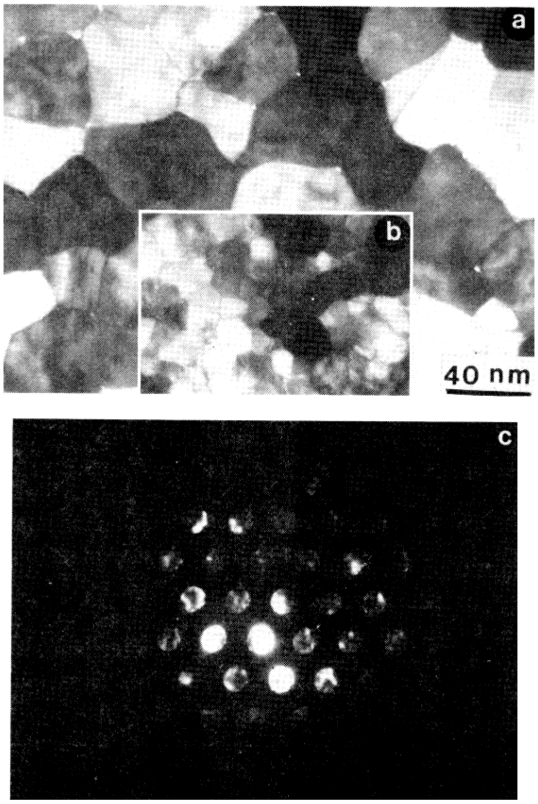

FIG. 2. TEM images of Co-Cr film (specimen B) at the top (a) and at the bottom (b) together with a representative CBED pattern (zone axis near [0001]) (c).

columnar microstructure in the films. It can be seen from the figures that the frequency of encountering large grains in the bottom region of the thin films is greater for film A. This means that the columnar microstructure in film A is more vertical in morphology.

Figures 3–5 are cross-sectional views of thin films A and B. In all instances, each column is continuous from structurally indistinguishable regions [see, e.g., region 2 in Fig. 3(b)] between glass substrate and column, and there appear to be no equiaxed grains at the regions (5–40 nm thick).

In Fig. 3 (specimen A), straight columns are well established [Figs. 3(a) and 3(b)] and a representative CBED pattern close to film/substrate interfaces shows $[2\bar{1}\bar{1}0]$ zone axis [Fig. 3(c)]. The presence of straight columns is not unexpected since the grain size in the top region of film A is found to be almost as large as that in regions close to the film/substrate interface (see Fig. 1). We observe the $[2\bar{1}\bar{1}0]$ zone axes frequently, but randomly oriented column grains with [0001], $[1\bar{2}1\bar{3}]$, or $[01\bar{1}2]$ zone axes have also been observed [see, e.g., Fig. 3(d)]. The infrequent observation of random orientations at the interfaces suggests that negligible amounts of transition layers exist in this film, and the *c* axis of each column was determined at the early stage of depostion. This suggestion has been confirmed by ferromagnetic resonance (FMR)[10] where no transition layers have been detected from specimen A.

Figure 4 is a higher magnification image from a different region of specimen A and clearly shows the straight co-

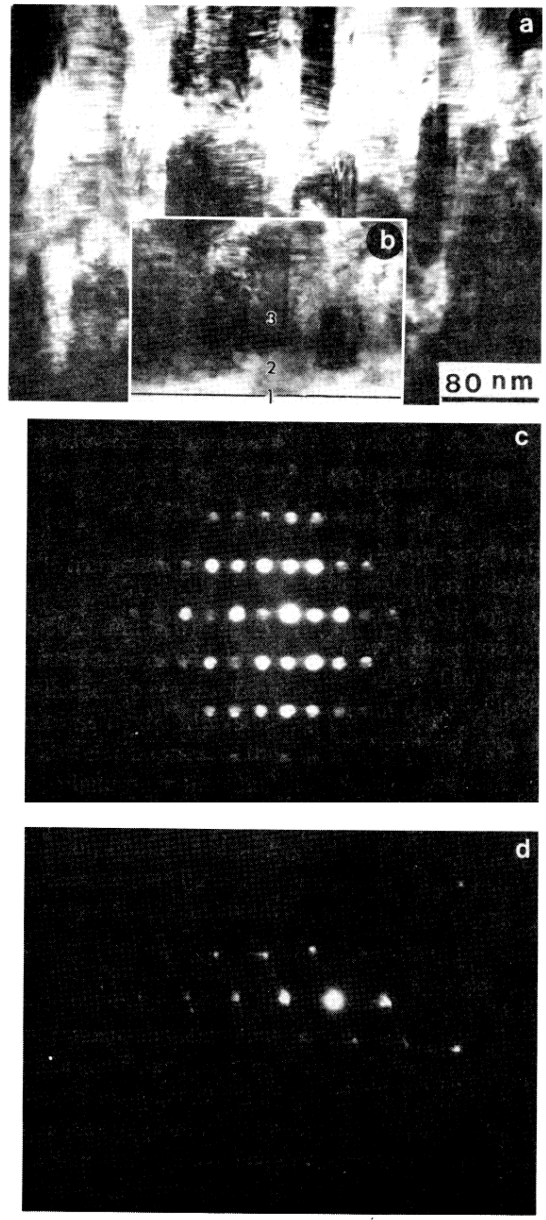

FIG. 3. TEM images of cross-sectioned specimen A over entire film (a) and at a region close to the glass substrate (b), together with their representative CBED patterns [(c) and (d)]. (c) Zone axis near $[2\bar{1}\bar{1}0]$ and (d) zone axis near [0001].

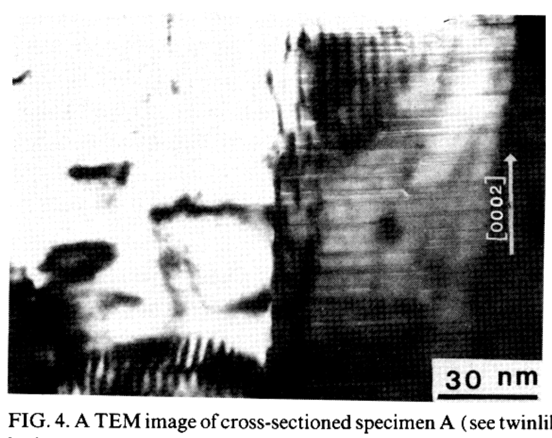

FIG. 4. A TEM image of cross-sectioned specimen A (see twinlike morphologies).

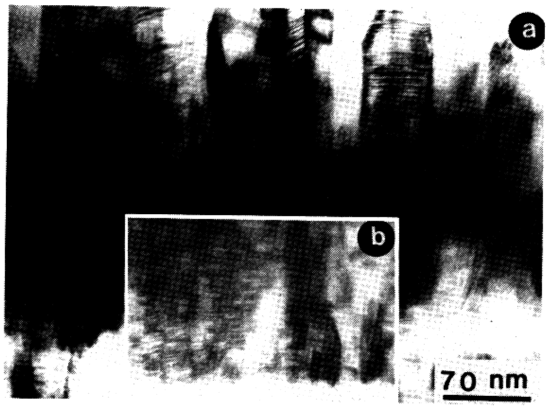

FIG. 5. TEM images of cross-sectioned specimen B over entire film (a) and at a region close to the glass substrate (b).

lumns. The columnar grains exhibit the crystallographic c axis parallel to the column.

In contrast to Figs. 3 and 4, Figs. 5(a) and 5(b) (specimen B) reveal conical columnar microstructures which are consistent with Fig. 2 where grain size in the top region of the film is larger than that in the region close to the film/substrate interface. The CBED patterns showed that most of the columns are perpendicular to (0001) plane near the film/substrate interfaces. Nevertheless, occasionally we have observed other zone axes such as [0001] and $[1\bar{2}1\bar{3}]$ near the interfaces. This latter observation is consistent with FMR[10] where small amounts of transition layers were detected from specimen B.

The formation mechanism of conical columnar microstructures has been well documented.[4,6,8,11] Survival of the fastest growing orientation for texture formation is reasonably applicable for the formation of the conical columnar microstructure since the columns are usually observed to be composed of (0001) layers of hcp structure. Therefore, nuclei form randomly on the top of the glass substrate and subsequently, the nuclei with (0001) plane grow selectively at the expense of other column grains with random orientation. They laterally expand along the growth direction of the columns.

Similarly, the sequence of the formation event for straight columnar microstructures is essentially the same as that outlined for the conical columnar microstructures. At present it is uncertain why straight columns have been formed at film A.

In addition, horizontal striations have been found to lie perpendicular to the column direction. They change their direction as they impinge upon column boundaries [see Figs. 3(a) and 3(b), and Fig. 5], implying that the features occur on specific crystal planes. In Fig. 3(d), the CBED pattern ($Z = [0001]$) reveals streaks in the $\langle 01\bar{1}0\rangle^*$ directions. This implies that the features are either twins or stacking faults. Indeed, the features are quite similar to twins in morphology (Fig. 4). These features have been interpreted to be stacking faults.[5,12] However, stacking faults in an hcp structure should give rise to streaks along the $\langle 0001\rangle^*$ directions. Hence, these features are better described as twins with the twinning plane being most probably of the type $(10\bar{1}2)$. We are currently determining whether these twins existed in the as-deposited state or are a result of specimen preparation for TEM.

CONCLUSIONS

(1) The Co-Cr films consist of randomly oriented transition layers on the top of glass substrate, followed by well-oriented c axis layers.

(2) Microtwins are observed perpendicular to the growth direction of the column grains.

(3) The straight column grains show negligible amounts of transition layers while the conical columnar grains reveal very thin layers of randomly oriented grains.

ACKNOWLEDGMENTS

This research was funded in part by the Magnetics Technology Center, Carnegie-Mellon University, Pittsburgh, PA 15213. We thank Professor J. O. Artman and P. V. Mitchell for helpful discussions.

[1]S. Yasui, S. Honda, M. Ohkoshi, and T. Kusuda, J. Appl. Phys. **52**, 2298 (1981).
[2]K. Ouchi and S. Iwasaki, IEEE Trans. Magn. **MAG-18**, 1110 (1982).
[3]T. Wielinga, J. C. Lodder, and J. Worst, IEEE Trans. Magn. **MAG-18**, 1107 (1982).
[4]J. C. Lodder, T. Wielinga, and J. Worst, Thin Solid Films **101**, 61 (1983).
[5]M. Futamoto, Y. Honda, H. Kakibayashi, T. Shimotsu, and Y. Uesaka, Jpn. J. Appl. Phys. **24**, L460 (1985).
[6]C. Hwang, D. E. Laughlin, P. V. Mitchell, A. Layadi, K. Mountfield, J. E. Snyder, and J. O. Artman, J. Magn. Magn. Mater. **54–57**, 1676 (1986).
[7]M. Ohkoshi, H. Toba, S. Honda, and T. Kusuda, J. Magn. Magn. Mater. **35**, 266 (1983).
[8]T. Chen, G. B. Charlan, and T. Yamashita, J. Appl. Phys. **54**, 5103 (1983).
[9]C. Byun, J. M. Sivertsen, and J. H. Judy, J. Appl. Phys. **57**, 3997 (1985).
[10]P. V. Mitchell (unpublished).
[11]E. Bauer, *Single Crystal Films*, edited by M. H. Francombe and H. Sato (Pergamon, New York, 1964), p. 43.
[12]Y. Niimura and M. Naoe, IEEE Trans. Magn. **MAG-21**, 1447 (1985).