MICROSTRUCTURAL DEVELOPMENT IN Co-Cr FILMS FOR PERPENDICULAR RECORDING MEDIA

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ABSTRACT

A series of Co-Cr films have been examined using transmission electron microscopy (TEM) in bright/dark field imaging, and selected area diffraction (SAD) as well as convergent beam electron diffraction (CBED).

Examination of the early stages of deposition by a DC magnetron provides evidence that amorphous Co-Cr films form prior to small randomly oriented equiaxed grains. Occasionally, a mixture of equiaxed and elongated grains were observed in the plane-view. Furthermore, microvoids are present running parallel to the columnar boundaries in cross-section view and at grain boundaries/edges in plane-view. In addition to conical and straight columnar grains, a mixture of small equiaxed and elongated grains were observed in cross-section view.

INTRODUCTION

The structural characteristics of Co-Cr films for perpendicular recording media have been extensively studied.[1-8] Recently, the present authors reported[8] that structurally indistinguishable regions are present within the first 5 to 40 nm of the deposited film. To our knowledge, this was the first time that such a region was reported in Co-Cr films. This observation is important to the understanding of film nucleation and growth. Hence, careful attention has been paid to a better understanding of the nucleation and growth of the Co-Cr films.

Previous TEM investigations of sputtered Co-Cr films[7] revealed the presence of microvoids, running parallel to the columnar boundaries. These microvoids would interrupt the movement of domain walls during the magnetization process. It remains uncertain how and why the microvoids form.

In the context of the above, the objectives of the present investigation were:

1. to investigate the formation of the structurally indistinguishable first formed regions;
2. to characterize microvoids found in the films.

EXPERIMENTAL

Co-Cr films were sputtered (DC magnetron) onto glass substrates from an alloy target of Co-22at.%Cr. The substrate temperature was varied while other parameters were kept approximately constant for all specimens, i.e.

deposition rate was about 14 nm/min, argon pressure ~ 2m Torr. The substrate temperature for specimens A and B were 25°C and 155°C, respectively. Also, a series of Co-Cr films ranging in thickness from 7.5 nm to 50 nm were sputtered onto carbon coated copper grids at room temperature for direct TEM examination. The methods of specimen preparation have been described elsewhere.[5] TEM investigations were performed on a Philips EM 420T analytical electron microscope operating at 120KV.

RESULTS AND DISCUSSION

Figs. 1-2 are electron micrographs and corresponding SAD/CBED patterns for Co-Cr films of four different thicknesses.

Fig. 1(a) (7.5 nm thick film) appears to be amorphous. Careful examination of the microstructure reveals nearly homogeneous honeycomb-like networks, suggesting incomplete coalescence of the networks during growth. This incomplete coalescence gives rise to voids between the networks. In addition, one can see that the formation of isolated crystalline Co-Cr grains (< 3.0 nm) occurs within the amorphous matrix. In Fig. 1(b), this is confirmed by a dark field image using the first ring of the SAD pattern (Fig. 1(c)). The first ring of the SAD pattern (Fig. 1(c)) exhibits a broad halo corresponding to a d-spacing of 0.21 nm. It should be noted, however that the SAD pattern by itself does not provide evidence for any crystalline Co-Cr. In contrast to this, the CBED pattern (Fig. 1(d)) reveals relatively sharp crystalline Co-Cr reflections from fine prominent Co-Cr grains. Thus, it appears as though the formation of amorphous Co-Cr regions precedes the development of crystalline Co-Cr grains, when the substrate is amorphous.

With increasing film thickness, one observes the development of crystalline Co-Cr regions from the amorphous regions. Diffraction patterns from these thicker films do not display any diffuse halos.

In Fig. 2(a_1) (10 nm thick film), the structure still exhibits honeycomb-like networks (as per Fig. 1(a)) but the number of crystalline Co-Cr grains increases drastically compared with the 7.5 nm thickness film. Similarly, stronger crystalline reflections of Co-Cr grains can be observed in the corresponding SAD pattern (Fig. 2(a_2)). The grains are randomly oriented since we observe all hcp reflections. Representative CBED patterns (Fig. 2(a_3)) reveal a zone axis near [0001] and a superposition of many (0110) reflections. The diffraction intensity of the amorphous Co-Cr and carbon decreases markedly.

As the film thickness increases even further (20 nm film), the honeycomb like networks disappear and the film is comprised only of crystalline Co-Cr grains (3 to 10 nm) (Fig. 2(b_1)). Although many grains are oriented along the [0001] zone axis, other zone axes ([2110], [1213] or [0112]) were occasionally observed. Fig. 2(b_3) shows that microcrystalline Co-Cr grains of the order of a few nanometers display halos composed of (0110) and (0002) reflections in their diffraction patterns.

In the 50 nm thickness film (Fig. 2(c_1)), the grain size is in the range of 8 to 15 nm, larger than that for thinner films (<3 nm at 7.5 nm thickness). This difference in size implies that the films grow in the form of conically expanding columns. Careful examination of the bright field image reveals microvoids (≈ 0.9 nm width) along grain boundaries (arrow X) and at grain edges (arrow Y). This observation suggests that microvoids form during deposition. Representative SAD patterns (Fig. 2(c_2)) show that the film for this thickness consists of less randomly oriented grains in comparison with thinner films. CBED results confirm this, as [0001] zone axes are generally observed (Fig. 2(c_3)), with [2110], [1213] or [0112] zone axes less frequently encountered as compared to thinner films. Although an examination of Fig. 1 of Futamoto *et al.*[4] suggests that

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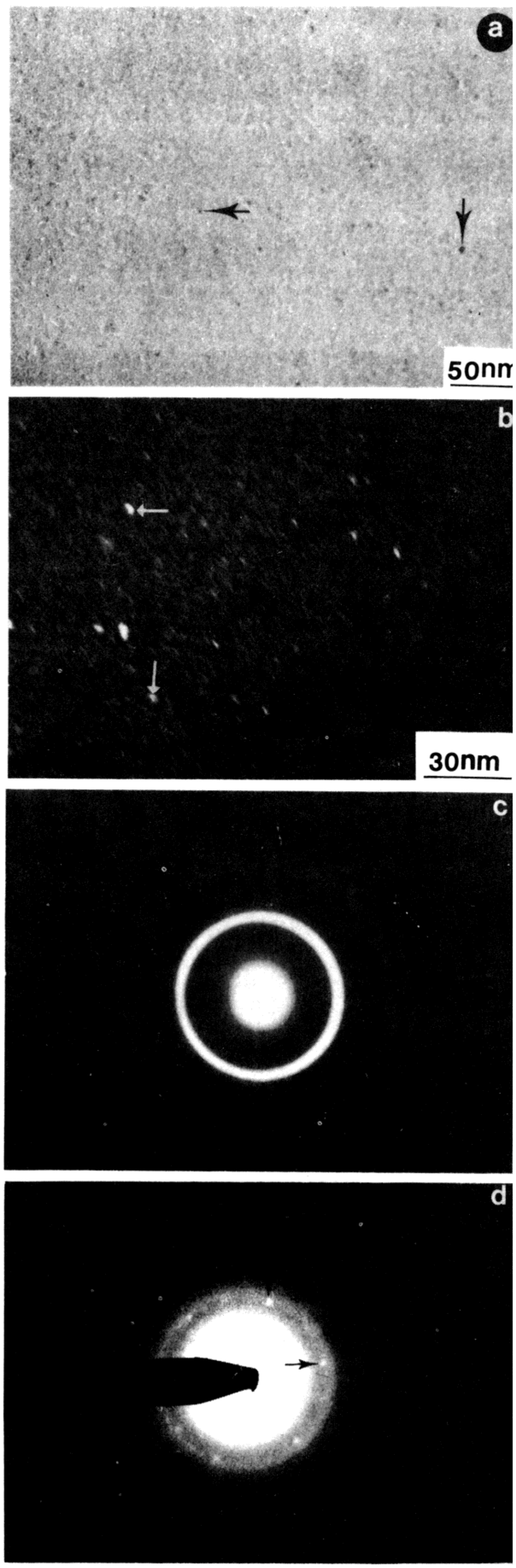

Figure 1 TEM image of 7.5nm thick Co-Cr film on carbon-coated Cu grid in bright field (a) and in dark field (b) together with SAD (c) and CBED (d) patterns.

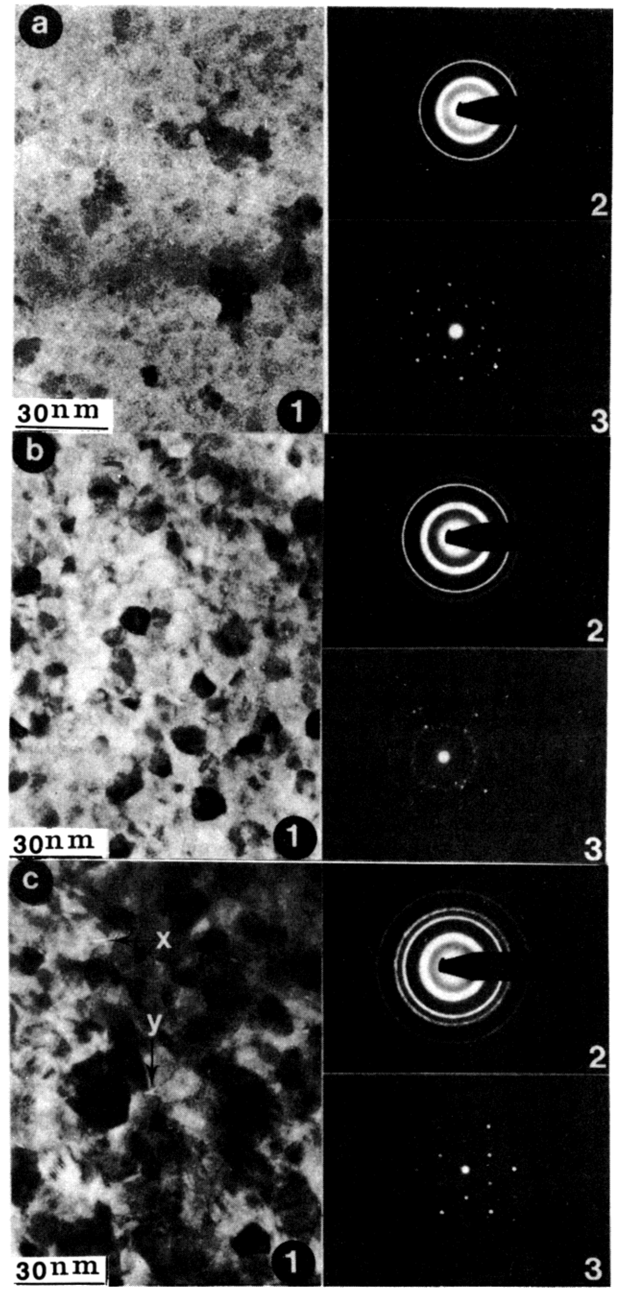

Figure 2 Bright field TEM images (1) of Co-Cr films on carbon-coated Cu grid together with their representative SAD (2) and CBED (3) patterns; a) 10nm thick, b) 20nm thick and c) 50nm thick.

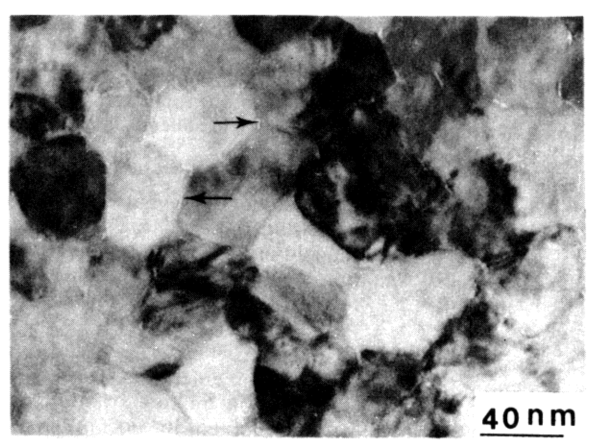

Figure 3 A TEM image of specimen A.

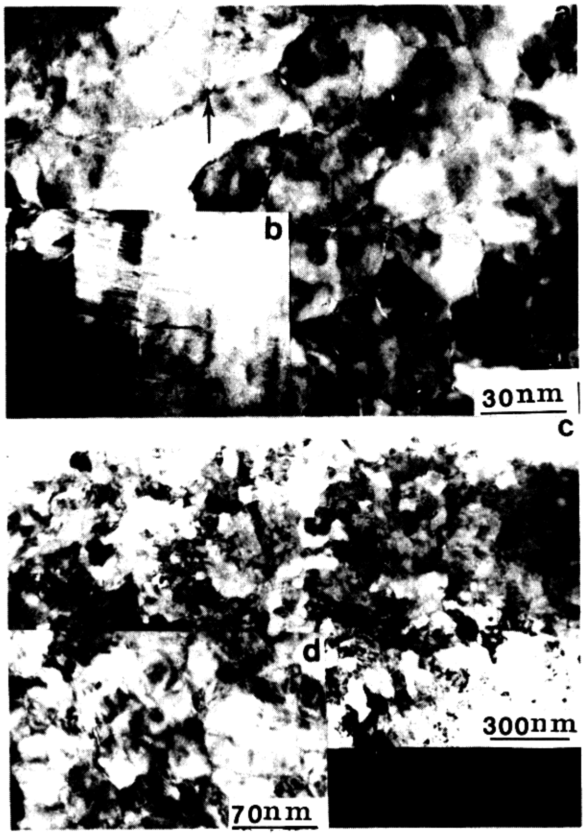

Figure 4 A TEM image of specimen B at the top (a) together with columnar grains (b) and a mixture of equiaxed and elongated grains (c) and (d) (magnified image) in cross-section view.

amorphous regions were present in their very thin films (5.0 nm thick), their results were inconclusive. The present work represents the first unambiguous reporting of the development of an initial amorphous Co-Cr film on amorphous substrates during deposition.

Fig. 3 is a typical plane view bright field micrograph of specimen A. In this instance, the film shows equiaxed grains and a large number of microvoids at grain boundaries and edges as observed in Fig. 2(c_1). Similarly, specimen B exhibits microvoids at grain boundaries and grain edges (Fig. 4(a)) as well as at columnar boundaries when viewed in cross-section (arrow in Fig. 4(b)). This corroborates that microvoids form during deposition.

The formation of microvoids may be related to the growth of films. When first formed, the randomly oriented grains grow laterally until impingement with adjacent grains. Since the growth rate varies with the orientation,[9] it can be postulated that microvoids are produced in the regions where the different columns meet.

Plane-views of Specimen B reveal an occasional mixture of equiaxed and elongated grains (Fig. 4(a)). Such elongated grains are thought to be due to sectioning of columnar grains inclined with respect to the substrate normal (see ref. 10).

We reported previously[8] that Co-Cr films are comprised of both straight and conical columnar grains in cross-section view. In addition, a mixture of elongated and equiaxed grains have been observed infrequently in cross-section view (Figs. 4(c) and 4(d)). At present, it is uncertain why a mixture of equiaxed and elongated grains has formed only in specimen B.

SUMMARY

1. An amorphous Co-Cr region was found to lie below the randomly oriented equiaxed grains. From this we conclude that an amorphous region forms initially.

2. A mixture of equiaxed and elongated grains were present in plane-view.

3. Microvoids were observed at grain boundaries and grain edges as well as columnar boundaries.

4. In addition to conical and straight columnar grains, a mixture of small equiaxed and elongated grains were present in cross-section view.

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