ELECTRON MICROSCOPY STUDY ON GROWTH PROCESS OF VACUUM DEPOSITED Co-Cr THIN FILMS

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Abstract. – Morphology and crystallographic structure of polycrystalline Co-Cr films are investigated as a function of thickness by scanning and transmission electron microscopy. A method of controlling the Co-Cr columnar diameter while keeping the c-axis growth orientation is proposed to prepare Co-Cr films suitable for high density perpendicular magnetic recording.

The read/write characteristics of Co-Cr perpendicular magnetic recording medium [1] can be improved by employing highly c-axis oriented Co-Cr films with large perpendicular magnetic anisotropies. We have previously shown that it is effective to deposit Co-Cr films on amorphous Ge layers to promote c-axis oriented columnar growth [2]. An optimization of microscopic film structure becomes very important when the recording density exceeds 100 kFCI where the magnetic bit length approaches to that of the columnar diameter. The recorded magnetization structure will be sensitively affected by the presence of magnetic inhomogeneties such as columnar boundaries or Cr segregation.

In the present research, the growth process of vacuum deposited Co-Cr film is investigated to understand the growth mechanism. Considering the growth mechanism, a double-seed-layer method is proposed and tried to control the diameter as well as the crystallographic orientation of grains which form a polycrystalline Co-Cr film.

Thin films were prepared by an electron-beam-heated evaporation method [2, 3]. Cleaved NaCl crystals and 50 μm thick polyimide films were used as the substrates. The substrates were heated at 180 °C during film deposition. The Co-Cr film thickness was varied from 5 to 350 nm. The in-plane microstructure was observed by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Some Co-Cr film specimens were cut and thinned to observe the cross-sectional microstructures [4].

Figures 1a-1d show the electron micrographs of 20 and 50 nm thick CoCr films grown on NaCl substrates. The SEM picture of 20 nm thick Co-Cr film shows that the film surface is rough consisting of many humps which form the surface undulations. These humps are formed by many small particles less than 5 nm in diameter.

The existence of these micro-particles show that evaporated Co and Cr atoms are clustering when they arrived at the film surface during deposition. Bulk diffusion beneath the surface must be taking place to form Co-Cr crystal grains. The TEM micrograph and the selected area diffraction shown in figure 1b clearly indicate that the 20 nm thick film consists of randomly oriented Co-Cr grains with an hcp crystal structure. The grain diameter estimated from the TEM pattern
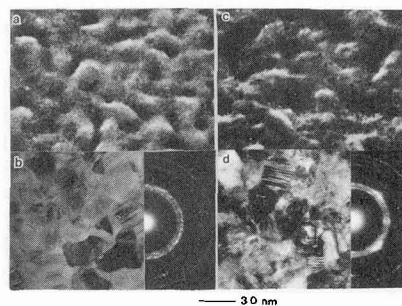

Fig. 1. – Electron micrographs of Co-Cr films deposited on NaCl. Film thickness: 20 nm [SEM: (a), TEM: (b)], 50 nm [SEM: (c), TEM: (d)].

of 20 nm thick film is 15-30 nm, which is nearly equal to the size of surface undulations observed in the SEM pattern. With increasing the film thickness, the grain diameter increased and the prefered growth orientation of c-axis became noticable as shown in figure 1d. Small particles were seen also for the 50 nm thick Co-Cr film. On NaCl substrates, the Co-Cr grains with the c-axis growth orientation dominated at a greater film thickness than 100 nm. The growth process and the microstructure are basically in agreement with those of sputter deposited Co-Cr films formed on Si and glass substrates [5, 6].

The surface undulations of Co-Cr films deposited on Ge layers are smaller than those of Co-Cr films deposited on NaCl as shown in figure 2. The selected area diffraction patterns from the Co-Cr films (Figs. 2b, 2d) are not continuous circular rings. Such diffractions are observable when a small number of grains are included in the selected area for taking the diffraction patterns. Although the grain boundaries are not clear in the TEM micrograph, the average diameter of Co-Cr grains are considered to be larger than that of the CoCr grains in the film deposited on NaCl. Therefore in this case, the surface undulations in the SEM pattern do not correspond to the grain diameter.

The diffraction pattern also shows that most of the grains are well c-axis oriented in the early stage of film growth. The unclear grain boundaries in the TEM micrograph are probably due to the fact that most of the crystal grains have the same vertical orientation, even though their in-plane orientations may be different. In this case, the TEM diffraction contrast be-

comes minimum, thus leading to unclear boundaries between grains in the TEM picture.

A c-axis oriented Co-Cr crystal grain has many humps on the top surface. These humps are observed in the SEM pattern of figure 2a. Bulk diffusion beneath the surface must be occuring to form a crystallographically continuous large grain during film formation process. These microscopic undulations on the growth front of each grain gradually disappeared when the Co-Cr film thickness was increased. The top shape of each grain became rounded for films thicker than 100 nm, as shown for example in figure 3. It can be concluded that Co-Cr grains on a Ge layer are highly c-axis oriented from an early stage of film growth, though the morphology of the growth front of grains is irregular microscopically.
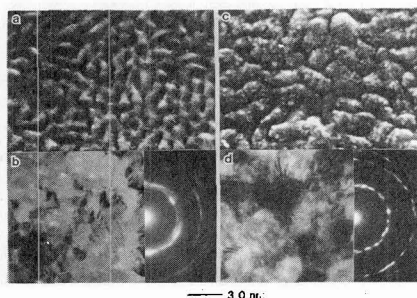

Fig. 2. – Electron micrographs of Co-Cr films deposited on Ge/polyimide. Film thickness: 20 nm [SEM: (a), TEM: (b)], 50 nm [SEM: (c), TEM: (d)].

The c-axis oriented grain growth on a Ge layer was observed for Ti film which has the same crystal structure (hcp) with the Co-Cr. The surface of Ti film was smoother than that of Co-Cr film with same thickness. Furthermore, the distribution of Ti grain diameter was also small. As Co-Cr films grow epitaxially on Ti films [2], this highly c-axis oriented Ti film formed on a Ge layer can serve as a seed-layer to control the columnar diameter of Co-Cr film when Co-Cr is deposited sequentially on the Ti film.

Cross-sectional microstructure of Co-Cr films formed on Ge/polyimide and Ti/Ge/polyimide substrates are compared in figure 3. These samples were
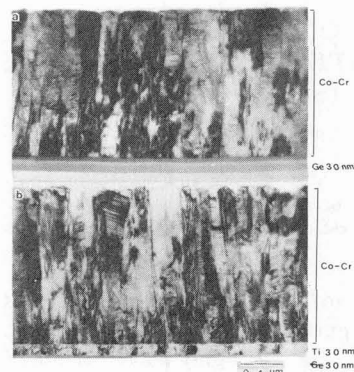

Fig. 3. – Cross-sectional TEM micrographs of 350 nm thick Co-Cr films. (a): on Ge/polyimide. (b): on Ti/Ge/polyimide.

prepared at the same time in a vacuum chamber using the two shutter method [3]. Well oriented pillar-like columns with the c-axis perpendicular to the substrate are grown for both films. The columnar diameter is, however, more uniform for the Co-Cr film formed on the Ti/Ge/polyimide ($d = 20 - 40$ nm) than that for the Co-Cr film formed on the Ge/polyimide ($d = 20 - 80$ nm).Another TEM analyses showed that the Co-Cr columnar diameter was nearly equal to that of Ti grain formed on the Ge layer.

The magnetic properties are compared in table I. The perpendicular magnetic anisotropy and the coercivity are increased for the Co-Cr film grown on the Ti/Ge/polyimide. The average columnar diameter of Co-Cr grains could be changed in a range approximately between 10 and 60 nm by adjusting the grain diameter of Ti film, which depended on the Ti deposition condition. It is possible to change the distribution profile of Cr atoms in the Co-Cr film while maintaining the columnar diameter at the same value which is determined by the underlying Ti grains, since the substrate temperature for Co-Cr deposition can now be varied independently. Segregation of Cr in a Co-Cr film will be promoted at higher substrate temperatures. We regard that this double-seed-layer method is very effective to optimize the microstructure of Co-Cr film in achieving high-density perpendicular recording.

Table I. – *Magnetic properties of Co-Cr films.*

	M_s	$H_{c\perp}$	$H_{c\parallel}$	$S_\perp$	$S_\parallel$	H_k
CoCr/Ti/Ge/polyimide	284 emu/cc	975 Oe	305 Oe	0.36	0.08	5.8 kOe
CoCr/Ge/polyimide	280	760	215	0.29	0.08	4.6

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