Microstructure of Co-Cr film prepared by Facing Targets Sputtering System

Masao HIRASAKA¹, Sadao KADOKURA², and Shigenobu SOBAJIMA¹

1) Material Analysis Research Center, TEIJIN Ltd., 4-3-2 Asahigaoka, Hino, Tokyo 191, JAPAN
2) Advanced Thin Film Research Labs. TEIJIN Ltd., 4-3-2 Asahigaoka, Hino, Tokyo 191, JAPAN

A Co-Cr film was prepared on polynethylene naphthalate (PEN) film using the Facing Targets Sputtering System. The microstructure of the Co-Cr film was observed using transmission electron microscopy (TEM). Although grain sizes of Co-Cr crystals were 10 nm in an initial stage of deposition, the grain sizes of the Co-Cr crystals grew to 30 nm in both middle layer and surface layer of Co-Cr films. A high resolution TEM image of the Co-Cr film showed that neither void nor amorphous region exists at the grain boundaries of the Co-Cr film. The segregated microstructure was also observed using the acid etching method. A TEM image of segregated structure showed that the magnetic and non-magnetic regions were clearly separated from each other in the crystal.

I. Introduction

Co-Cr thin films have been widely studied for advanced magnetic recording media. The magnetic properties of the Co-Cr films were considered to depend on the hcp crystal structure, the c axis orientation of the crystal and a phase separation in the crystal. The phase separation could be confirmed from transmission electron microscopy (TEM) image of segregated microstructure in chemically etched Co-Cr film and was caused by the existence of both ferromagnetic and non-magnetic regions in the crystal[1]. A pattern of segregated structure is known as chrysanthemum-like pattern structure (CP structure)[2]. Those microstructures are strongly influenced by the plasma environment of the deposition process as well as the substrate temperature in the preparation of Co-Cr film.

A Facing Targets Sputtering System (FTSS) is one of the preparation methods and is known as a superior plasma confinement technique[3]. FTSS was considered to provide plasma free conditions and high mobility of sputtered atoms on the deposition plane[4,5]. Recently, a new FTSS was developed for use as a large scale web coater. The cathodes of this FTSS provided in-plane and perpendicular magnetic field in combination with electron reflection plates[6]. It was reported that polyethylene-2,6-naphthalate (PEN) film satisfies all the characteristics of the substrate for ultra-high density magnetic tapes when Co-Cr thin film media are fabricated by FTSS[7]. Because PEN film has higher flexural rigidity, lower level of oligomer extraction and higher heat resistance than polyethylene-terephthalate (PET) film. In this study, microstructure of the Co-Cr films prepared on PEN films by the new FTSS were revealed by using TEM.

II. Experimental

Co-Cr films were deposited on PEN films maintained at 150°C by FTSS. The target was a Co-17 at.% Cr alloy and the rate of deposition was about 0.3 μm/min. The thickness of the Co-Cr film was 150 nm. The perpendicular coercivity (Hcp) of the Co-Cr film was 1150 oe and the perpendicular anisotropy magnetic field (Hk) was 3.1 Koe.

The specimens were put in double grids (VECO DH50/100) and the PEN substrates were dissolved by mixed organic solvents (hexafluoroisopropanol:CHCl₃ = 1:1). The Co-Cr films in the grids were milled using an ion milling machine (GATAN DIM600) under the following conditions; accelerating voltage of 3.0 kV, Ar-ion current of 1.0 mA, and incident angle of 15°. Patterns of segregated microstructures were observed after chemical etching of the thin specimens[2]. A TEM instrument (JEOL JEM-2010) was used at an accelerating voltage of 200 kV. CERIUS software (Molecular Simulation Inc.), multi-slice method calculation, was used to simulate TEM images.

III. Result and Discussion

The Co-Cr film was divided into three layers for characterization of crystal growth in the deposition process. The polymer substrates were removed from the specimens and the Co-Cr films were uniformly etched on surface side, substrate side or both sides by ion milling. The TEM image of each layer was observed from 001 direction as shown in Fig.1. The sizes of the crystal grains in the initial layer of the substrate side were about 10 nm. The crystal grains in the middle layer were bigger than those in the initial layer and the sizes were measured to be about 30 nm. Further, the crystal grains in the layer of the surface side were the same as those in the middle layer. This result showed that large numbers of small grains formed a closely packed structure in the initial stage of deposition. Then, the size of grain increased with the growth of Co-Cr films until the grain size reached to about 30 nm.

Lattice fringes were clearly observed in the TEM image of the crystal grains of each layer. For example, the lattice image of the middle layer is shown in Fig.2. The lattice fringes are not in disorder; they run parallel within the grain. At the grain boundaries, neither amorphous area nor lattice disorder can be observed. Further, small crystal grains exist...
in the boundary space as shown in Fig. 2 (arrow). Small crystal grains appear to fill the voids which are formed in the same corners of grain boundaries. From these results, it is concluded that the Co-Cr films deposited by FTSS have the closely packed microstructures and the non-disordered interface at the grain boundaries.

A high resolution TEM image of one crystal grain was also observed. Ordered bright contrast spots were clearly shown in the spaces which are surrounded by Co atoms.

Fig. 1 TEM images of Co-Cr film prepared by FTSS. (a) surface side (b) middle layer (c) substrate side
Fig. 2 TEM images of Co-Cr film prepared by FTSS. (a) an arrow shows a small crystal in the grain boundaries. (b) an interface at a grain boundary.

Fig. 3 A high resolution TEM image of Co-Cr film prepared by FTSS and simulated image of Co crystal. Ordered bright spots show the spaces which are surrounded by Co atoms.
Fig.4 Crystal model of Co and simulated TEM image. (a) a super lattice (3x3) of 001 plane of Co, (b) a simulated TEM image: thickness of 20nm, (c) a simulated TEM image: thickness of 40nm

Fig.5 A TEM image of a segregated structure in a crystal grain observed in the lattice image as shown in Fig.3. In order to understand these bright-contrast spots, lattice images were simulated using the multi-slice method. In this simulation, the crystal structure was set to the hcp structure of Co, the space group of the lattice is P63/mmc, the lattice size was determined by electron diffraction (a = 0.264 nm, c = 0.420 nm). The simulated lattice images were changed in both focus length and thickness. This simulated result shows that dark contrast spots around bright spot are not clearly separated from each other. The bright contrast spot is vacant which is surrounded by Co atoms as shown in Fig.4. The simulated image which was similar to the TEM image of Fig.3 was gotten under following condition: thickness of 20 nm and under focus of 45 nm. This simulated image is put
on the TEM image of Fig.3. From this result, there is neither dislocation nor defect in the grain. Therefore, the Co-Cr film prepared by FTSS was also considered to have defectless lattice structure.

The other hand, a segregated microstructure was observed in one grain after chemical etching. The specimen of the middle layer was etched using acids and the TEM image was observed as shown in Fig.5. Although lattice fringes were not observed in the chemically etched area, the lattice fringes were clearly observed in the other area. The chemically etched area and the other area corresponded to the Co-rich and the Cr-rich regions, so those were considered to be the ferromagnetic region and the nonmagnetic region respectively[1,2]. From the TEM image of Fig.5, the magnetic and non-magnetic regions were clearly separated from each other in the crystal grain and the areas of both regions were from 5 to 10 nm. Such a clearly phase separation was considered to be a good property for ultra-high density magnetic recording.

IV. CONCLUSION

The Co-Cr film was prepared on PEN film by FTSS and the microstructure of the Co-Cr film was observed using TEM. Although grain sizes of Co-Cr crystals were 10 nm in the initial stage of deposition, the grain sizes of the Co-Cr crystals grew to 30 nm in both the middle layer and the surface layer of the Co-Cr films. The high resolution TEM image of the Co-Cr film showed that neither void nor amorphous region exists at the grain boundaries. Further, neither dislocation nor defect was also observed in the grain. Therefore, the Co-Cr film prepared by FTSS was considered to have defectless crystal structure. The segregated microstructure was also observed by using acid etching method. The TEM image of the segregated structure showed that the Co-rich and Cr-rich regions were clearly separated from each other in the crystal grain.

REFERENCES