Electron microdiffraction of faulted regions in Co-Cr and Co-Ni-Cr thin films

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The planar defects which are commonly observed in deposited Co-Cr and Co-Ni-Cr magnetic thin films have been characterized by electron microdiffraction and trace analysis. It was unambiguously shown that these planar defects are (0001) stacking faults, which are thought to be formed to reduce the growth stress of the film during deposition.

The magnetic properties of microstructure of various thin metallic films have been extensively investigated in search of a high-density magnetic storage medium.[1,2] Among those, Co-Cr thin films have received the most attention as promising candidates for perpendicular recording.[3,4] On the other hand, Co-Ni-Cr thin films deposited on a Cr underlayer have been shown to display good in-plane coercivity, and hence have been regarded as good candidates for longitudinal recording.[5,6] Since the magnetic properties of these media are intimately related to their microstructure, many investigations have been performed to clarify the microstructural details of these films.[7-10] Both of these alloys exhibit the hcp structure ($P6_3/mmc$).

A noticeable feature of the microstructure of these as-deposited films is the existence of an appreciable number of planar defects,[7-10] which are observed as striations in bright field transmission electron microscopic (TEM) images. These planar defects may be either twins or stacking faults, both of which are commonly observed in as-deposited thin films.[11,12] Although the microstructural details of Co-Cr and Co-Ni-Cr thin films were studied extensively, a detailed discussion of the planar defects in these films has not been given. In their paper, Futamoto *et al.*[13] reported that horizontal lines seen in columnar grains are stacking faults existing parallel to the hcp (0001) basal planes. However, it should be noted that what they observed "parallel to (0001)" was the trace of the planar defects in the cross-sectional view. If the planar defects were $(10\bar{1}2)$ twins, their trace in a $(10\bar{1}0)$ section would also be parallel to the basal plane. Furthermore, in recent studies,[8,9] it was proposed that these planar faults in Co-Cr films were $\{10\bar{1}2\}$ twins. Hence, in order to determine the nature of the planar defects, additional TEM studies are necessary.

The aims of the present study are thus twofold; one is to resolve whether the planar defects are twins or stacking faults in Co-Cr, and the other is to establish the type of planar defects in Co-Ni-Cr. In this study, the individual grains in Co-Cr and Co-Ni-Cr thin films were investigated by electron microdiffraction and trace analysis. It is unambiguously shown that the planar faults observed in both types of films are (0001) stacking faults. No diffraction evidence to support the presence of $\{10\bar{1}2\}$ twins was found.

The Co-Cr thin films were deposited on a glass substrate using a Varian dc magnetron sputtering system. The Co-Ni-Cr thin films were prepared by a rf sputtering system, L-H Z400. The compositions of the targets were Co-22 at. % Cr and Co-30 at. % Ni-7.5 at. % Cr, respectively. Some of the Co-Ni-Cr films were deposited onto a Cr underlayer to obtain a $\{10\bar{1}1\}$ texture[5]; the others were deposited on a glass substrate. The thickness of the films was in the range of 50–100 nm. Those films were thinned from the substrate side by an argon ion mill. The microdiffraction patterns were taken on a Philips EM420 operating at 120 kV. The nominal probe size was 20 nm.

Typical in-plane microstructures of a 100 nm Co-Ni-Cr film deposited on a 350 nm Cr underlayer and a ~1-μm-thick Co-Ni-Cr film deposited on a glass substrate are shown in Figs. 1(a) and 1(b), respectively, together with the corresponding selected area diffraction (SAD) patterns. The 100 nm film deposited on the Cr underlayer has a significantly smaller grain size (~40 nm) than the other (~80 nm). A noticeable difference between these two films is, however, the number of the grains exhibiting striations. In Fig. 1(a), most of the grains show striations, while only a small number of the grains show striations in Fig. 1(b). The SAD pattern shows that the Co-Ni-Cr film shown in Fig. 1(a) is not strongly *c*-axis textured, since all the diffraction rings are present. On the other hand, several rings are not present in the SAD pattern shown in Fig. 1(b); only the rings of the form {hki0} are present. This means that the film shown in Fig. 1(a) is strongly textured along [0001] zone axis. It

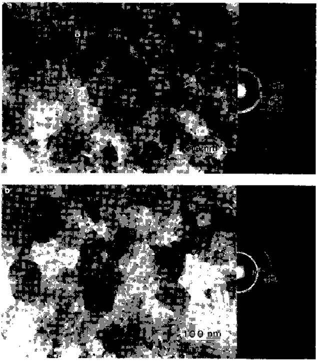

FIG. 1. Bright field images of (a) 100 nm Co-Ni-Cr film deposited on a 350 nm Cr underlayer, and (b) ~1 μm Co-Ni-Cr film deposited on a glass substrate. The corresponding selected area diffraction patterns are also shown.

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should also be noted that the $\{200\}_{fcc}$ ring is clearly observed in the SAD pattern shown in Fig. 1(b), which indicates that the fcc phase is also present in this film. This ring was always observed in Co-Ni-Cr films regardless of the deposition conditions.

Figures 2(a) and 2(b) show electron microdiffraction patterns and the corresponding bright field images of the Co-Ni-Cr grains. The $[2\bar{1}\bar{1}0]$ diffraction pattern shown in Fig. 2(a) clearly shows [0001] streaks, which indicates that the planar defects lie on (0001) planes. From this microdiffraction pattern, it was also confirmed that the striations in the bright field image of the coresponding grain lie along the traces of the (0001) planes. Since the (0001) plane cannot be a twinning plane in the hcp structure, it is concluded that the planar defects are stacking faults. No evidence was found to prove the presence of $\{10\bar{1}2\}$ twins. It was noted above that some fcc phase is also present in Co-Ni-Cr films. The microdiffraction pattern shown in Fig. 2(b) is a $[110]_{fcc}$ pattern. Note that the streaks are clearly observed in the $[\bar{1}11]$ directions. This indicates that the stacking faults of this fcc phase are on the $(\bar{1}11)$ planes. The traces of the striations of the corresponding grain were also confirmed to lie along the $(\bar{1}11)$ planes. This was observed in films deposited both on glass and on the Cr underlayer.

Figure 3(a) shows a plan-view microstructure of a Co-Cr film. The details of the Co-Cr microstructure are reported elsewhere.[8,9] The grain size is on the order of 30 nm. As seen from the SAD pattern shown in Fig. 3(b), the Co-Cr film is strongly [0001] textured [cf. Fig. 1(b)]. Unlike the Co-Ni-Cr case, a $\{200\}_{fcc}$ ring was not found to be present.

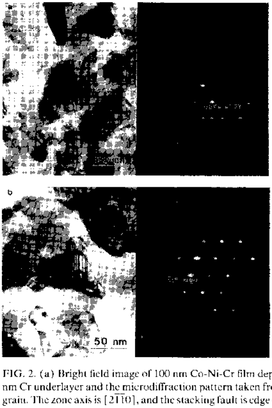

FIG. 2. (a) Bright field image of 100 nm Co-Ni-Cr film deposited on a 350 nm Cr underlayer and the microdiffraction pattern taken from the arrowed grain. The zone axis is $[2\bar{1}\bar{1}0]$, and the stacking fault is edge on. The trace of the striation is perpendicular to the [0001] direction. (b) Bright field image of $\sim 1\ \mu m$ Co-Ni-Cr film deposited on a glass substrate and the microdiffraction pattern taken from the arrowed fcc grain. The zone axis is [110]. The trace of the striation is perpendicular to the $[\bar{1}11]$ direction.

FIG. 3. Plan-view bright field image of a Co-Cr film deposited on a glass substrate and the corresponding SAD pattern displaying a strong [0001] texture.

Note also that the striations are not observed at all in this strongly (0001) textured film.

Figure 4 shows a section view of a Co-Cr column displaying defect contrast. The c-axis direction in each column is indicated. As shown in Fig. 4, the trace of the defect makes an angle of $\sim 90°$ with the c axis. If these defects were $\{10\bar{1}2\}$ twins, their traces would lie at an angle between 47° and 90° with [0001], depending on the particular twin in a $[10\bar{1}2]$ zone axis. Figure 4(b) represents a corresponding $[11\bar{2}0]$ electron microdiffraction pattern. In this $[11\bar{2}0]$ zone axis, the trace of the $(10\bar{1}2)$ twins should lie at an angle of $\sim 65°$ with [0001], which is not the case. As shown, we can clearly see streaks along [0001], which can only be due to (0001) stacking faults, since reciprocal lattice rods due to these thin planar defects lie 90° to (0001).

Once it has been established that the planar defects are (0001) stacking faults, the number of grains with striations

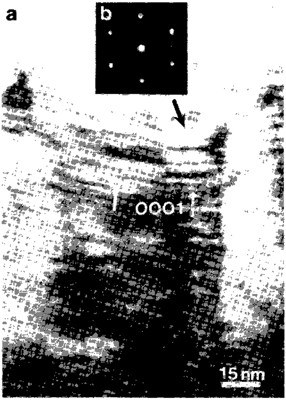

FIG. 4. (a) Cross-sectional bright field image of a Co-Cr film and (b) the corresponding $[11\bar{2}0]$ microdiffraction pattern. Note that the traces of the striations are perpendicular to the [0001] streaks.

in the plan view can be correlated with the development of the [0001] texture. We now consider the origin of the striation contrast. If an electron wave front propagates through a faulted region of a crystal, fringes will appear in the image due to the phase change introduced on passing from the unfaulted to the faulted region.[14] Such fringe contrast can be observed only when the stacking fault is inclined in the foil. The observed striation contrast is formed due to the superposition of the fringes at multiple stacking faults [denoted *A* in Fig. 1(a)]. Hence, the striations are not always well-defined fringes as is generally observed in the case of a single stacking fault. When the *c* axis is perpendicular to the incident beam, fringes do not appear, and we simply observe the traces of the stacking faults on the foil surface as denoted *B* in Fig. 1(a). The striations due to these traces look sharp since there is no fringe interference from multiple stacking faults. This contrast is observed only when faults are edge-on to the electron beam. The striations are not observed when the *c* axis is parallel to the incident beam. Therefore, we can estimate the degree of [0001] zone axis texture by measuring the fraction of the grains which do not show striations in the plan view, assuming that all the grains contain stacking faults, which seems to be a reasonable assumption from Fig. 3. In order to confirm this, we obtained microdiffraction patterns in plane view from grains displaying striation contrast in Fig. 1(b). None of the grains displayed a [0001] zone axis. On the other hand, the grains which did not show striations were *all* of [0001] orientation.

It has been shown here that the planar faults observed in the Co-Cr and Co-Ni-Cr are principally stacking faults on $(0001)_{hcp}$ or $\{111\}_{fcc}$. No evidence to support the presence of twins was found. We now consider why (0001) stacking faults are introduced during the deposition process of the Co-Ni-Cr and the Co-Cr films instead of twins. One possible reason is that they might be formed during deposition as a result of mis-stacking of the newly deposited layer. But this mechanism is not likely to be applicable in the present case, because the stacking faults are present even when there is no *c*-axis texture, as in the case of Co-Ni-Cr thin films deposited on Cr underlayers [Fig. 1(a)]. The films deposited on the Cr underlayer have been reported to have the $\{10\bar{1}1\}$ texture so that the Co-Ni-Cr layer grown epitaxially on the Cr underlayer.[15] If the *c* axis is not perpendicular to the plane surface, (0001) stacking faults cannot be formed as a result of the mis-stacking of a newly deposited surface layer. Then, the most probable reason for the introduction of stacking faults is to relieve the internal stress which is built up during the film growth. The built-up stress in Co-Cr thin films is reported to be extremely high,[9] so the grains must plastically deform to relieve this stress during the growth process. The plastic deformation occurs either by twinning or by slip.

Slip deformation will occur if the resolved shear stress on the (001) plane in the $\langle 11\bar{2}0\rangle$ exceeds the critical resolved shear stress for slip. If the stacking fault energy on the (0001) planes is low enough, slip deformation will occur by the passage of partial dislocations. Unless another partial dislocation follows on the same (0001) plane, a stacking fault will remain in the grain after the passage of the first partial dislocation. The stacking fault energies of Co and Co-based alloys are, in general, much lower than those of other hcp systems such as Cd, Zn, Mg, Ti, and Be.[16] Thus, the stacking fault width on the basal plane of Co-based alloys will be large. In the case of a Co-Fe alloy, the stacking fault width is reported to be in the range of 100 nm.[17] The average grain size of the present films was less than this stacking fault width. Since the grain size is smaller than this stacking fault width, the first partial may annihilate at the grain boundary before the second partial starts to follow the first one. Once the first partial is annihilated, the driving force for the movement of the second partial is substantially decreased. This mechanism would be particularly applicable in the case of a Co-Ni-Cr thin film, since the stacking fault energy is thought to be essentially zero from the fact that both hcp and fcc are present. Hence, the basal slip would occur by the passage of partial dislocations instead of total dislocations. So, whenever slip deformation occurs, stacking faults may be formed. Since we could not find any evidence of twinning in the present alloy thin films, it is highly likely that the deformation occurs by basal slip, which causes the formation of an extremely high density of stacking faults in the grains.

In summary, the planar faults which are observed in both Co-Cr and Co-Ni-Cr films were unambiguously determined to be (0001) stacking faults. In Co-Ni-Cr thin films, $\{111\}_{fcc}$ stacking faults were also present. These stacking faults are thought to be formed in order to reduce the growth stress which is built up during the deposition process.

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