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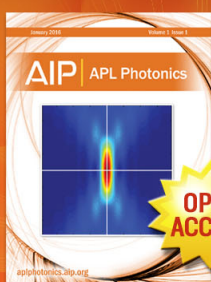
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# Physical factors affecting the corrosion resistance of CoCr thin-film media

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Various factors such as film porosity, stress, type of underlayer, and the presence of an overcoat were found to affect the corrosion resistance of CoCr and CoCrTa thin-film magnetic disks exposed to high-humidity, corrosive environments. It was found that corrosion products form preferentially along the polishing and texture lines of the NiP substrate. Relieving internal stresses in the disk layers using a low-temperature annealing process improved the corrosion resistance of these Co alloy films. Additionally, eliminating the carbon overcoat allowed the formation of an oxide layer which was more protective in chlorine-containing environments.

## INTRODUCTION

Cobalt-chromium alloys are widely used as magnetic media in high-capacity disk drives. Earlier work has shown that these alloys, which generally contain at least 14% chromium, have better corrosion resistance than cobalt-nickel alloys.<sup>1,2</sup> In this work, a number of factors which influence the corrosion behavior of Co-16% Cr and Co-14% Cr-4% Ta alloys were investigated. Specifically, the effect of film stress on corrosion resistance has been evaluated. It is well known that stressed metals are more susceptible to corrosion than unstressed metals.<sup>3</sup> Internal stress in sputtered films can be either compressive or tensile, depending on the processing conditions during sputtering. Typical values of internal stress in metal or alloy films are of the order of  $5\text{--}7 \times 10^9$  dyn/cm<sup>2</sup>.<sup>4</sup>

Other factors considered include the effect of the carbon overcoat, as well as the underlayer, on disk corrosion resistance. In this study, disks were utilized in order to better relate the corrosion properties observed in accelerated testing to processing variations.

## EXPERIMENT

The thin-film disks were dc magnetron sputtered Co-16 at. % Cr and Co-14% Cr-4% Ta, with a nominal thickness of 1200 Å on an 1800-Å Cr underlayer. The substrates used were either glass or NiP plated onto aluminum. The accelerated corrosion test was carried out in an atmosphere consisting of 10 ppb Cl<sub>2</sub>, 120 ppb H<sub>2</sub>S, and 200 ppb NO<sub>2</sub> at 30 °C and 75% RH. The degree of corrosion was monitored by measuring the change in light scattering at various exposure times. Measurements were made at nine locations on each disk, at specific inner, middle, and outer diameter positions at 120° angles around the disks, using a constant spot size of 250 mm<sup>2</sup>. This technique has been described previously,<sup>1</sup> and the time at which the media reflectivity shows a marked increase (induction time) has been correlated to the point where the disk corrosion products would likely cause an increase in data errors or a head crash.<sup>5</sup> In this study, the reflectivity measurements were taken at regular intervals until the change in reflectivity was greater than 0.1 μW.

Disks were evaluated both with and without a nominal 350-Å carbon overcoat. In addition, some disks were annealed at 110 °C and 0.1 Torr for 6 days to partially relieve the internal stress in the metal layers. Qualitative determina-

tions of film stress were made by comparing the relative amount of film deflection after chemically etching away the aluminum substrate in a 25% potassium hydroxide solution. Further media characterization was carried out using both optical and electron microscopy techniques. Auger depth profiles were obtained for some of the samples to determine if differences in chemical composition influenced the disk corrosion resistance. Finally, the student's *t*-test was used to determine if the differences in corrosion resistance for various types of disks were significant.

## RESULTS AND DISCUSSION

Corrosion products were found to form initially along the polishing and texture lines of the NiP substrate layer as shown in Fig. 1. If the aluminum and NiP layer are replaced by glass, the corrosion resistance is improved and corrosion products form first at the disk edges. In this case, two sets of disks were prepared simultaneously, with one set of disks having the standard substrate, NiP plated onto aluminum, while the other set was made by sputtering the Cr, CoCrTa,

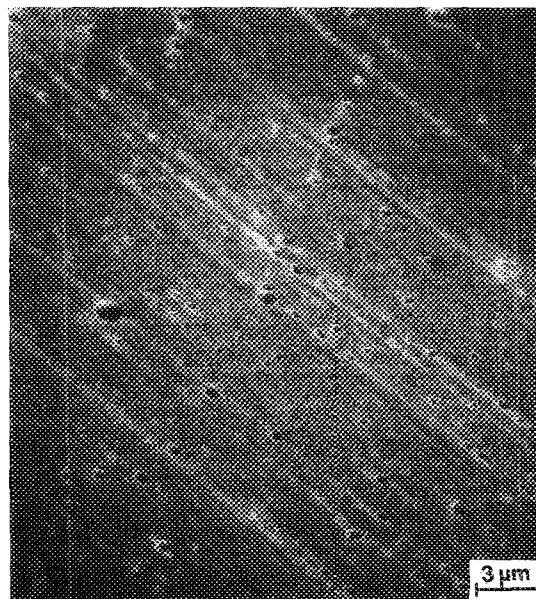


FIG. 1. Corrosion product formation along polishing and texture lines of the NiP layer for a CoCr disk with a carbon overcoat.

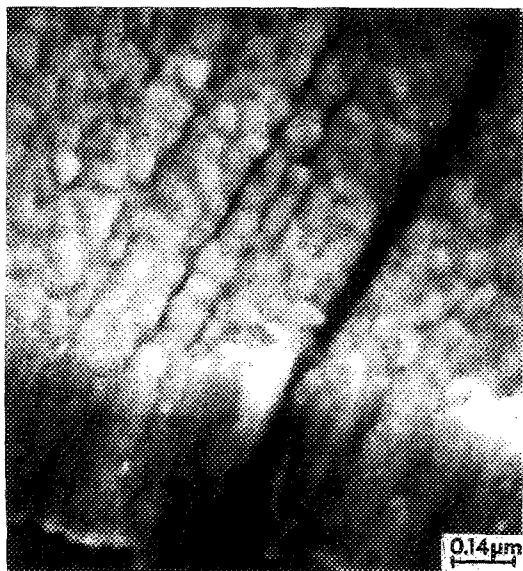


FIG. 2. Electron micrograph showing porosity at the NiP texture lines.

and C layers directly onto glass. The induction time for the disks on the NiP layer averaged 17 h, while the disks fabricated with glass substrates exhibited signs of corrosion at the edges after an average of 42 h. A high-resolution electron micrograph of a carbon-coated CoCrTa film, on a NiP substrate, is shown in Fig. 2. It is apparent that the magnetic coating is more porous along the NiP polishing/texture lines, allowing water vapor, along with dissolved gases, more access to the thin-film layers. Also, in these areas of higher porosity, an electrochemical cell is set up between the magnetic layers and the NiP substrate.<sup>6</sup> After these typical films are exposed to the accelerated atmosphere, the corrosion products can be removed from the surface and analyzed further. The EDX spectrum shown in Fig. 3 indicates that the main corrosion products contain nickel and chlorine, which confirms that the main corrosion reaction is occurring at the sputtered film/NiP interface. Four sets of two disks each were prepared together to investigate the effects of the

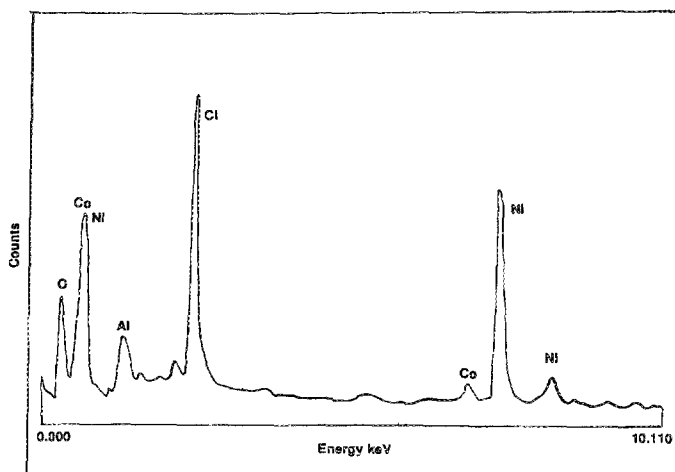


FIG. 3. Nickel-rich corrosion products removed from a CoCr disk with a carbon overcoat after the induction time was reached.

TABLE I. Average corrosion induction times.

Disk composition	Treatment	Average induction time
350 Å C/1200 Å CoCr/1800 Å Cr	none	$9 \pm 2$ h
350 Å C/1200 Å CoCr/1800 Å Cr	110 °C/144 h	$17 \pm 3$ h
1200 Å CoCr/1800 Å Cr	none	$13 \pm 3$ h
1200 Å CoCr/1800 Å Cr	110 °C/144 h	$37 \pm 12$ h

carbon overcoat and annealing for stress relief on the corrosion resistance of the disks. The control group of disks had the nominal 350-Å sputtered carbon overcoat, while another set of disks was removed from the sputtering system before any carbon was deposited. Half of the disks from each of these groups were then annealed according to the procedure described previously.

Table I shows the average corrosion induction times for the four combinations of CoCr disks evaluated. When the student's *t*-test was carried out for all of the disk combinations, it was found that the four sets of disks had significantly different distributions of induction times. In all cases there was a less than 0.001% chance that the distributions were the same at a 95% confidence level. The best corrosion resistance was obtained for the annealed disks without carbon overcoats, with average induction times of 37 h, compared to 9 h for the unannealed disks with the carbon overcoat. The effect of eliminating the carbon overcoat without the annealing treatment led to a smaller, but still significant increase in induction time from 9 to 13 h. This is in contrast to earlier work which showed that a carbon overcoat offered improved corrosion resistance in high-relative-humidity atmospheres containing only 5 ppm SO<sub>2</sub> as an added pollutant.<sup>1</sup> The Auger depth profile in Fig. 4 shows that an oxide layer about 100 Å deep is present on the magnetic surface of the disk without the carbon overcoat. The presence of this oxide layer must provide the improved corrosion resistance in chlorine-containing atmospheres.

The Auger depth profile taken from the annealed disk without the carbon overcoat is also shown in Fig. 4, revealing no changes in the oxygen concentration between the an-

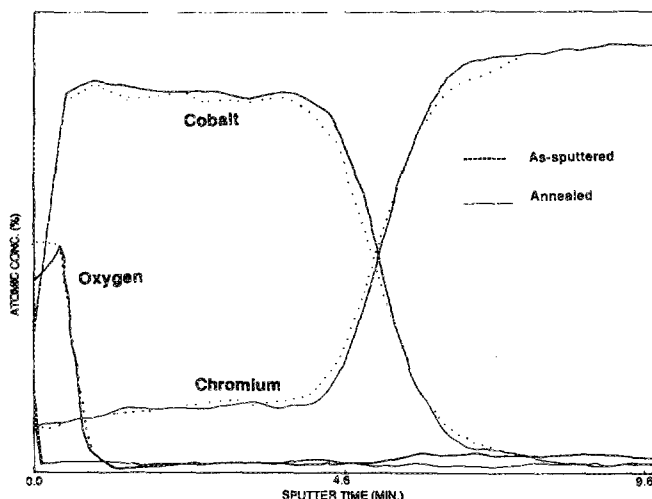


FIG. 4. Auger depth profiles of annealed and unannealed disks showing no differences in oxygen between the disks before exposure.

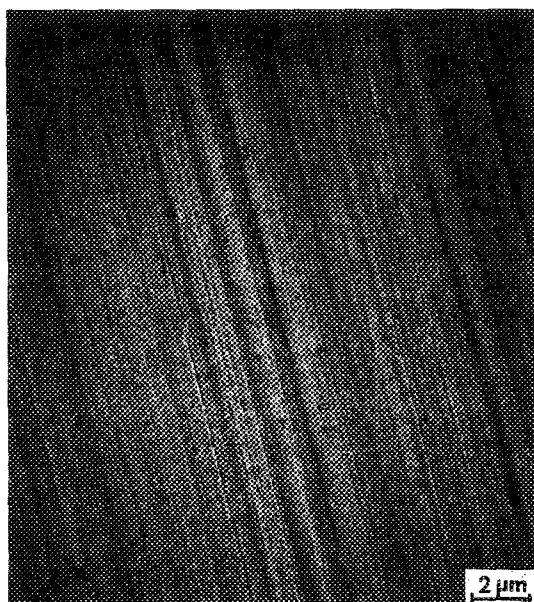


FIG. 5. Corrosion product morphology as observed at the induction time for the annealed disk without a C overcoat.

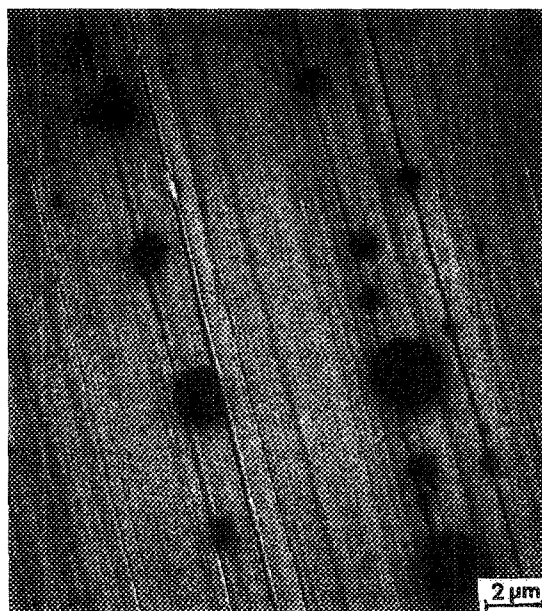


FIG. 6. Larger corrosion products are found at the induction time for the unannealed disk without a C overcoat.

nealed and unannealed disks. The corrosion induction time for the annealed disks is improved by approximately a factor of 3 over the unannealed disk, suggesting that another factor besides the presence of the oxide layer is influencing the corrosion resistance. Figures 5 and 6 are electron micrographs taken of the surfaces of the annealed and unannealed disks, respectively. It is apparent from these micrographs that the morphology of the corrosion products is different on the two disks. In both cases, the photos were taken when the corrosion induction time was reached, i.e., when the surface reflectivity changed by  $0.1 \mu W$ . The Auger profiles showed no chemical differences between the two disks before the corrosion test, and the grain size was unaffected by the annealing process. Analysis of the corrosion products scraped from the unannealed film show mostly nickel and chlorine in the EDX spectrum, similar to that seen in Fig. 3. The EDX spectrum of the smaller corrosion products from the annealed disk shown in Fig. 5, however, do not contain appreciable nickel. The EDX analysis shows that they are composed of cobalt and chlorine, along with oxygen. This result confirms that a different mechanism is responsible for the corrosion which occurs on the stress-relieved disks.

A comparison of the carbon-coated disks with and without the annealing treatment indicates that the annealing step also improves the corrosion resistance of the carbon-overcoated disks. When an annealed film with a carbon overcoat was removed from the substrate via the chemical etching technique, it was very flat relative to a similar piece removed from the unannealed film which exhibited a compressive stress. This unannealed film was found to have a radius of curvature equal to 30 mm.

## CONCLUSIONS

These results suggest that, in addition to film porosity and an electrochemical cell between the sputtered layers and

the NiP layer, the presence of internal stress at the thin film and NiP interface strongly influences the mechanism of corrosion for these disks. The average corrosion induction time was increased from 9 to 37 h for a set of disks by eliminating the carbon overcoat and relieving the internal stress. As stresses are generally higher at areas such as polishing and texturing lines, corrosion is most likely to occur at these spots when stress is dominating the corrosion mechanism. In the stress-relieved disks, corrosion occurs more randomly on the surface. The natural air-formed oxide was found to be more protective than carbon overcoats for CoCr disks exposed to chlorine-containing atmospheres. Since this is the opposite from the result obtained in  $SO_2$  atmospheres, it further indicates that there is not a single corrosion test which can determine an absolute corrosion resistance for different magnetic recording alloys. Various corrosion reactions are possible on thin-film magnetic disks, and factors such as disk-processing conditions, type of corrosives expected in the atmosphere, and the relative rates for the various reactions need to be evaluated in designing atmospheric corrosion tests.

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<sup>3</sup>U. R. Evans, *An Introduction to Metallic Corrosion* (Arnold, London, 1948), pp. 114–122.

<sup>4</sup>D. W. Hoffman and J. A. Thornton, *Thin Solid Films* **40**, 355 (1977).

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<sup>6</sup>V. Novotny, G. Itnyre, A. Homola, and L. Franco, *IEEE Trans. Magn.* **MAG-23**, 3645 (1987).