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The investigation of nanostructures of magnetic recording media by TEM

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Abstract

Diverse applications of transmission electron microscopy techniques used in investigating the nanostructures of magnetic recording materials are presented. Specimen preparation methods are discussed for the specific case of magnetic thin film recording media. Investigations of the crystallographic orientation, grain size and distribution, and interfacial nanostructures are presented.

Keywords: Nanostructure; TEM; Magnetic recording media

1. Introduction

The magnetic recording media in hard drives are currently all in the form of thin films. In order to optimize the properties of the magnetic thin layer, other layers are necessary. Typical configurations of the thin films for recording are shown in Fig. 1, in which the magnetic layers are usually cobalt based hcp alloys. In Fig. 1(a) the easy magnetization axis is made to lie in the plane of the recording layer and hence the media is called longitudinal recording media. An underlayer is employed to control the crystallographic texture and the grain size of the magnetic layer. The overlayers are introduced to protect the recording layer. Fig. 1(b) shows another scheme of magnetic thin films for perpendicular recording media, which may be utilized in the future. Since the magnetization direction during the reading and writing is perpendicular to the thin layer, the soft magnetic underlayer (SUL) forms a flux closure and therefore functions as part of the head. The intermediate layer is used to decouple the SUL from the recording layer and also to control the grain and texture growth of the recording layer. The overlayers have the same function as in longitudinal media. All the layers in perpendicular recording media except the SUL are required to be as thin as possible to decrease the distance between the head and the media and SUL. Thus, microstructural control is even more challenging in the perpendicular media than in longitudinal media.

Other types of recording media are also being investigated, including high coercivity $L1_0$ thin films, multilayered media, and nanoparticles. The $L1_0$ FePt is one of the materials with the highest coercivity under investigation and thus has been

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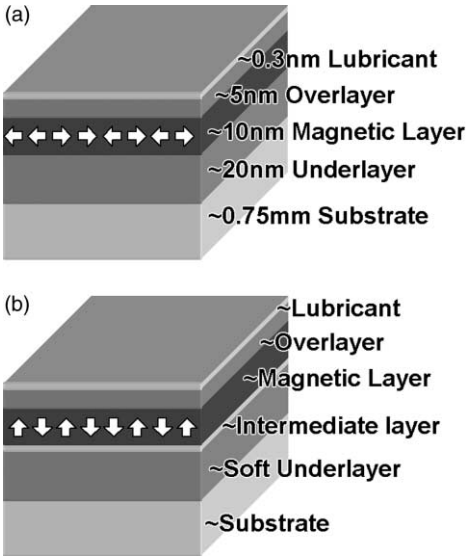

Fig. 1. Schematic of the typical configurations of longitudinal (a) and perpendicular (b) recording media.

studied intensively as a potential medium for ultrahigh recording density [1]. The higher coercivity allows for the use of smaller grains, without the problem of superparamagnetism. The smaller grains give rise to a better signal to noise ratio, which allows for higher recording density. For the same reason, self-assembled FePt nanoparticles are also among the potential media for ultrahigh density magnetic recording [2].

Since the important length scales of thin film media (film thickness, grain size, etc.) are in the nanometer range, transmission electron microscopy (TEM) with the capability of high resolution and nanobeam probe has been an important tool used to develop the thin film media. A modern TEM is usually equipped with a highly coherent source beam (field emission gun), high resolution (~0.1 nm), beam size of ~1 nm in the scanning mode, and the capability of chemical compositional analysis—X-ray energy dispersive spectroscopy (EDS), electron energy loss spectroscopy, Z-contrast imaging, etc. We report on some of our microstructural studies of various magnetic recording materials by means of modern TEM techniques.

2. TEM specimen preparation for magnetic recording material

Since magnetic recording media usually consists of multiple thin layers on a glass substrate, some

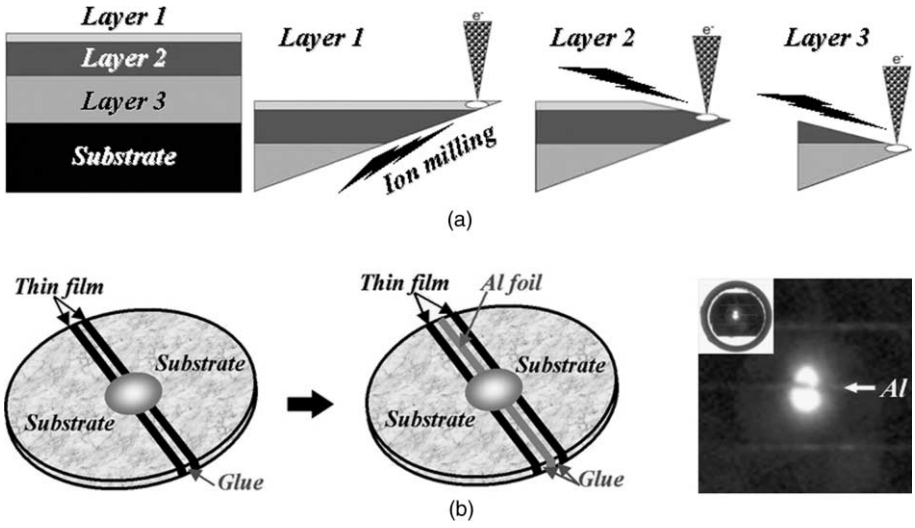

Fig. 2. (a) The step-by-step ion milling of plan-view TEM specimen of multiple layered sample [3]; (b) schematic of the cross-sectional TEM specimen with and without an Al line in the middle and an image of the specimen with an Al middle line after perforation by ion milling. Note the hole shape is not a regular circle.

specific techniques have been developed and applied to the preparation of both plan-view and cross-section TEM specimens. As shown in Fig. 2(a), the step-by-step ion milling procedure can produce an electron transparent area for plan-view examination for each layer in a sample with several layers. The milling time, incident angle, and ion energy should be carefully controlled to insure that the regions to be investigated are not milled away. A few seconds of milling at 3–5° with ~2/3 of the normal voltage is a good way to begin the thinning process. Once the desired layer is reached, the microstructures can be studied individually, even for very thin (tens of nm) films. To prepare a cross-sectional specimen, the conventional method is to bond two pieces of the sample together face to face, slice it perpendicular to the surface, grind it and ion mill it to obtain a perforation near the interface. At the edge of the hole, there are four opportunities to obtain thin area of the magnetic films. However, this procedure typically does not yield good results for magnetic media samples because the films are very thin and are more readily thinned than the substrates. Thus they are quickly removed by the ion beam after perforation, making it difficult to obtain a large enough area for TEM investigation. Since aluminum is a "tough" material in terms of ion milling rate, we place an Al foil between the two surfaces when bonding them together. During ion milling the Al foil acts as a shield to the thin films on both sides and protects them from the on-coming ion bombardment even after the sample is perforated. Fig. 2(b) shows a schematic of the sample structure and the image of a specimen prepared by the above procedure. The Al foil is still present after enough thin area is obtained, which also improves the electrical conductivity of the samples that have glass substrates.

3. Texture determination by electron diffraction

The microscopes employed for our studies are a Tecnai F20 from Philips (FEI now) equipped with a Field Emission Gun, Gantan's Imaging Filter system, and EDS for high resolution observation and chemical analysis, and a JEM-2010 from JEOL for conventional studies. TEM specimen preparation were all performed on a Gatan's Precision Ion Polishing System, on which a small milling angle can be easily achieved.

One of the advantages of TEM is the combination of electron diffraction with a real space image. The information of crystallographic parameters and orientations can be easily obtained and associated with any specific region by means of selected area electron diffraction (SAD). SAD is therefore often used to determine the identity of phases and grain textures in recording media.

Crystallographic texture is an important issue in magnetic recording thin films [3]. One of the important roles of the underlayer in longitudinal recording media is to induce the desired crystallographic texture in the magnetic layer. The texture of the underlayer itself is critical to accomplish this function. With the sample preparation method described above, the texture for each layer can be determined by the electron diffraction technique developed by Tang and Laughlin [4]. Here we illustrate a simple way to characterize the texture by means of pattern matching. An electron diffraction pattern can be considered to be the intersection of the Ewald sphere and the reciprocal structure of the material. For a single crystal, the reciprocal structure is simply the intensity weighted reciprocal lattice and the diffraction pattern is thus an array of spots. For a larger number of randomly oriented grains of a certain material, the reciprocal structure becomes concentric spheres formed by the rotation of the intensity weighted reciprocal lattice about the original of reciprocal space. When there is a fiber texture, the reciprocal structure is formed by the rotation of each intensity weighted reciprocal lattice point around the fiber axis and thus consists of a series of circles on each Laue zone. At some specific tilting angles, the Ewald sphere touches a circle in a high order Laue zone and a characteristic arc pattern forms. Fig. 3(a) shows schematics of the arc patterns of hcp-Co with $(1\,0\,\bar{1}\,0)$ texture at the tilting angles of 0°, 30°, and 55°, respectively. The tilting angles are chosen at the position that the Ewald sphere meets the innermost ring of each Laue zone. Fig. 3(b) shows the experimental SAD patterns taken at the three angles from the

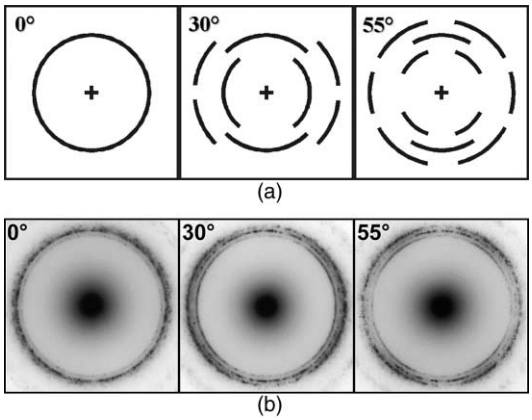

Fig. 3. (a) The characteristic arc patterns of hcp-Co at 0°, 30°, and 55°, and (b) experimental SAD patterns taken at same tilting angles.

top CoCrPt layer of the antiferromagnetically coupled (AFC) media [5]. The media has a structure of CoCrPt/Co/Ru/Co/CoCrPt/Cr/NiAl/glass-substrate. The bottom CoCrPt layer directly above the Cr/NiAl underlayer is expected to have the correct $(1\,0\,\bar{1}\,0)$ texture due to the established epitaxial relationship. We were unsure if the texture of the top CoCrPt layer returns to $(1\,0\,\bar{1}\,0)$ because of the possible lattice mismatch with the Co/Ru/Co nanolayers. The SAD patterns were found to fit very well with the characteristic arc patterns so it was concluded that the top CoCrPt layer also has the $(1\,0\,\bar{1}\,0)$ texture. The texture was actually retained due to deformation that occurred in the Co/Ru/Co nanolayers, which will be discussed below.

4. Measurement of grain size and distribution

The grain size of current magnetic recording media is about 10 nm. Thus, bright and dark field imaging are standard methods of investigation of the important microstructural features.

In order to achieve higher recording density, the grain size has been decreased to retain a certain number of grains within one recording bit. The grain size distribution is also very important. The narrower the distribution is, the better are the magnetization decay properties of the media. To measure the grain size and distribution it is necessary to first image the grains. Dark field images are usually taken for this purpose for the higher resolution. The condenser aperture is placed on a diffraction ring in the diffraction mode and all the grains contributing to the diffraction intensity within the aperture are imaged after switching to the imaging mode of the microscope. However, this dark field image is not necessarily representative of the grain size and distribution of the area, since only a small fraction of the grains are imaged. Also the selection is probably biased if the aperture is placed on the strongest part of the ring. This is especially true for the case of mechanically textured media. Only those grains with a particular orientation will be imaged in that way, while the grains are actually orientated periodically with the grooves on the substrate. The SAD pattern of the plan-view sample without tilting of the mechanically textured CoCrPtB thin film is shown in Fig. 4(a) and displays discrete rings (arcs) due to the mechanical texture. In order to collect as many

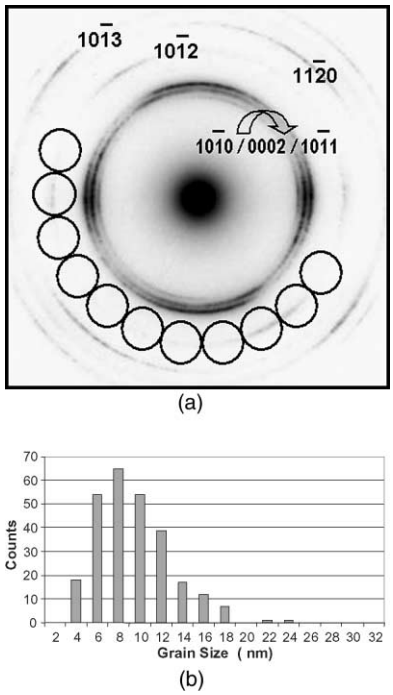

Fig. 4. (a) SAD pattern of the mechanically textured Co-alloy thin film, on which the method of placing aperture is illustrated, and (b) the plot of the grain size distribution.

grains as possible, we developed a method of producing images. The aperture is placed adjacently along half a circumference of the ring and dark field images are taken in succession. All the images are included in the calculation of the grain size and distribution after being taken from a number of different regions. The plot of the grain size distribution is shown in Fig. 4(b) for this sample, and shows the average grain size to be about 8 nm.

5. Interfacial strain analysis by high resolution TEM

High resolution TEM (HRTEM) is a powerful tool to study crystalline nanostructures, especially of interfaces, defects, etc., which play a dominant role in determining the properties of magnetic recording media.

A nanolayer of pure Co was added to both sides of the Ru layer in AFC media to enhance the exchange coupling effect [5]. The perfection of the interfacial structure determines the exchange coupling strength. Electron diffraction revealed that the top CoCrPt layer possessed the $(1\,0\,\bar{1}\,0)$ texture despite the expected lattice mismatch between Ru and Co. The HRTEM cross-section image also showed the crystal planes going through the Co/Ru/Co interfaces. In order to study the strain at the interface, images were taken at an orientation to show only the $(1\,0\,\bar{1}\,0)$ planes (Fig. 5(a)). These are the planes parallel to the film plane. Split spots are seen in the fast Fourier transformation (FFT) of the $(1\,0\,\bar{1}\,0)$ lattice image in Fig. 5(b). The main spot in the middle is from CoCrPt, and the inside

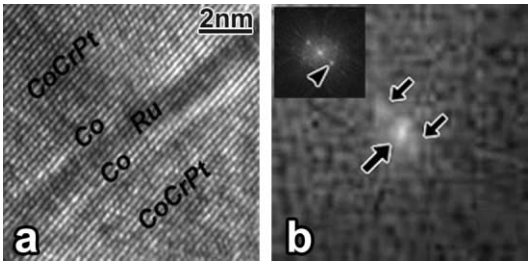

Fig. 5. $(1\,0\,\bar{1}\,0)$ lattice and FFT images of the interfacial area of AFC media.

Table 1
Comparison of crystallographic parameters of the nanolayers

	a (nm)	c (nm)	d (nm)	d' (nm)	S (nm^2)	S' (nm^2)
Ru	0.271	0.428	0.234	0.254	0.1159	0.1069
Co	0.250	0.406	0.216	0.206	0.1016	0.1068
CoCrPt	0.256	0.414	0.222	–	0.1061	–

a and c are the lattice parameters; d is the $(1\,0\,\bar{1}\,0)$ plane spacing in bulk, d' is in nanolayers; $S = ac$, $S' = acd/d'$.

satellite corresponds to the region with a larger $d_{(1\,0\,\bar{1}\,0)}$ which is Ru, and the outside satellite is from Co. The $d_{(1\,0\,\bar{1}\,0)}$ of CoCrPt is measured to be 0.222 nm by X-ray diffraction. With CoCrPt as reference, the $d_{(1\,0\,\bar{1}\,0)}$ of Co and Ru nanolayers are measured from the FFT image to be 0.206 and 0.254 nm, respectively. They are obviously different from the d-spacings of bulk samples, which implies that deformation has occurred in the Ru and Co nanolayers to retain coherency. Assuming the deformation in the nanolayers is elastic, the unit area of the $(1\,0\,\bar{1}\,0)$ plane can be calculated for both Ru and Co before (S) and after (S') the deformation. The results are summarized in Table 1. It can be seen that the S' of Ru and Co are very close to each other, which means that there was a good lattice match between the planes perpendicular to the interface.

6. Growth of FePt perpendicular thin films

FePt thin films have been sputter deposited onto Si substrate with cubic MgO as underlayer [6]. The ordered $L1_0$ FePt grains with the size of 12–15 nm are oriented with the c-axis perpendicular to the substrate surface. HRTEM images were taken from the cross-section samples and are represented in Fig. 6. Both the nanobeam diffraction and HRTEM image show that the FePt is ordered with the (0 0 1) texture. It is also seen from Fig. 6(a) that the sputtered MgO was initially amorphous and subsequently crystallized with [0 0 1] normal the substrate. FePt was then epitaxially grown on the top of [0 0 1] MgO crystal grains. The lattice mismatch between MgO and FePt was accommodated by misfit dislocations, which can be seen from Fourier filtering image of the (2 0 0) reflections as shown in Fig. 6(b) and (c).

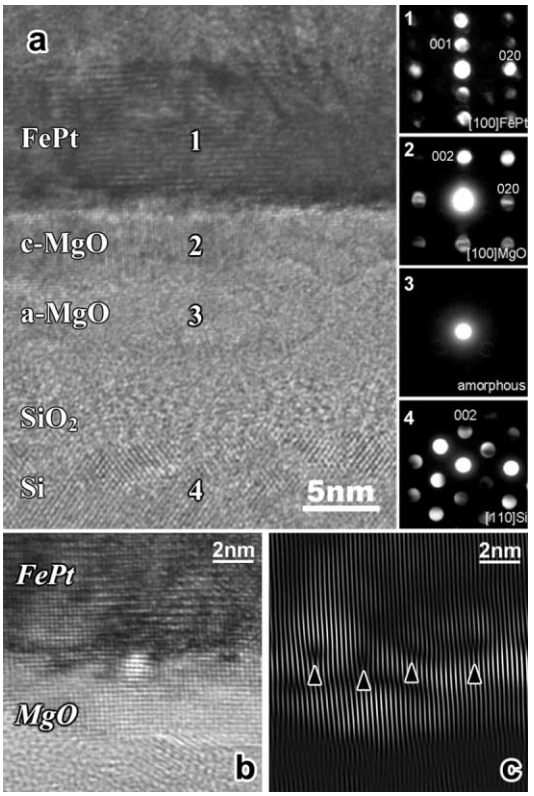

Fig. 6. Cross-sectional HRTEM image and FFT of $L1_0$ FePt thin films and the nanodiffraction patterns for each layer.

7. Summary

Various important microstructural features of magnetic recording thin films investigated by TEM have been presented in this paper. We first presented some techniques used in sample preparation. Specific applications have been discussed in texture determination, grain size and distribution measurement, and interfacial nanostructures. The investigations presented include categories of longitudinal (AFC), perpendicular, and $L1_0$ media. We have demonstrated that TEM is a powerful tool in the nanostructural investigation in magnetic recording.

Acknowledgements

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References

[1] Weller D, Moser A, Folks L, Best ME, Lee W, Roney MF, Schwickert M, Thiele J, Doerner MF. IEEE Trans Magn 2000;36:10.
[2] Sun S, Murray CB, Weller D, Folks L, Moser A. Science 2000;287:1989.
[3] Lu B, Laughlin DE, Microstructure of longitudinal media in the physics of ultrahigh-density magnetic recording. Springer Series in Surface Sciences, vol. 41. p. 34.
[4] Tang L, Laughlin DE. J Appl Cryst 1996;29:411.
[5] Peng Y, Zhu J-G, Laughlin DE. J Appl Phys 2002;91:7676.
[6] Jeong S, Ohkubo T, Roy AG, Laughlin DE, McHenry ME. J Appl Phys 2002;91:6863.