Determination of Grain Size Distributions in Magnetic Recording Media by Grazing Incidence X-ray Diffraction

Shaoping Li, Charles Potter, Dean Palmer, Dennis D. Eberl, Tim Klemmer, Jennifer Spear, Celeste Reiss, Daniel Brown and Augusto Morrone

***Abstract*-- The grain size distribution of magnetic recording media has been measured with in-plane grazing incidence X-ray diffraction (GIXRD), without using synchrotron radiation, via the Bertaut-Warren-Averbach (BWA) technique. Lognormal-like grain size distributions are observed in all recording media. The obtained distributions are consistent with the TEM observations. In addition, the relevant measurement issues are also discussed.**

***Index Terms*—Grain sizes, grain size distributions, magnetic recording media, grazing angle X-ray diffraction (GIXRD)**

I. INTRODUCTION

THE role of grain size and its distribution in magnetic recording media cannot be understated because it directly influences both the raw signal to noise ratio (SNR) capability and the thermal stability of storage bits[1]. As magnetic recording density is further increased, the grain size distribution and its control will become extremely important, ultimately determining the recording density in thin film recording media.

Currently the measurement of grain sizes and their distribution in recording media is mainly obtained through transmission electron microscopy (TEM) characterizations. However, TEM has a few disadvantages: (i) TEM is a destructive method for grain size measurement. (ii) The procedure for preparing the plane view TEM specimens for the recording media is tedious, somewhat difficult, time consuming and thus expensive. Usually it takes few days to a week to get the grain size information from the TEM measurement. (iii) TEM can only provide local grain size information over a small spot area (0.5-5μm^2),with mediocre repeatability. In this article, we report our recent attempt to utilize the grazing incident X-ray diffraction technique to estimate the grain size distribution via a modified BWA technique[2-4]. The non-destructive method is based on in-plane scans using the grazing angle geometry, which has the capability to examine the microstructure of the surface magnetic layer without interference from either the underlayer or the substrate[5].

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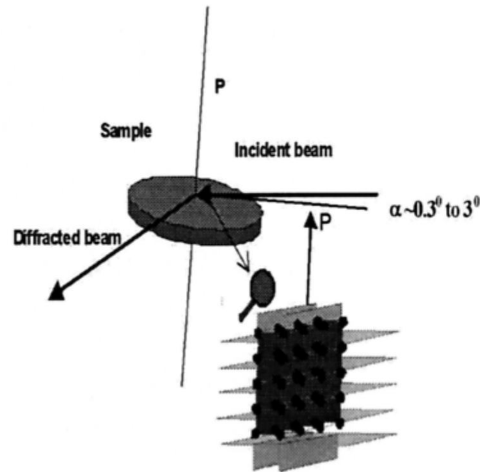

FIG. 1. Grazing-incidence x-ray diffraction geometry. The sample surface normal is **P** and the grazing incident angle α is around 0.7^0 in the measurements.

II. EXPERIMENT

In this study we have examined two types of longitudinal recording media samples with different mean grain sizes. The media compositions are primarily CoCrPtTa alloy with Cr based underlayer on the top of NiP/Al substrates textured circumferentially. The coerocivity (H_c), and the remnant moment-thickness product (Mt) between the sample A and sample B are close, and they are 3800Oe and 3420Oe; 0.4 memu, and 0.42, respectively. The recording media sample A and B were intended to support a recording linear density of 400-450 kBPI. The measured SNR difference in two media is around 2.5 dB, primarily due to the grain size difference.

The perpendicular medium examined is (Co/Pd)n superlattice (n=13). The thickness of each Pd layer in the superlattice is 1.0nm, and the thickness of each Co layer is 0.38nm. The very latest Philips laboratory X'Pert PRO MRD system with a special X-ray lens has been utilized to collect the symmetric in-plane GIXRD data (CuK_αradiation λ=1.5418 Å), allowing a large accessible

range for in-plane two theta scan without requiring advanced synchrotron source. A Ni filter is used to eliminate the beta radiation. Fig.1 illustrates the optic scattering geometry. When the magnetic layer was measured, the grazing angle between incident beam and specimen surface was around 0.7^0, which is near to the condition for total reflection. Fig.2 show in-plane 2theta-theta diffraction scan of the longitudinal medium sample. From the raw data fitting analysis, it was found that the Co fcc-(111) peak was present, indicating that there exists a small number of grains with deformation faults. Nevertheless, the Co-based magnetic thin film is mainly in the hcp-phase. The peak intensities from (101), (102) and (103) are not as abnormally large as reported previously[5].

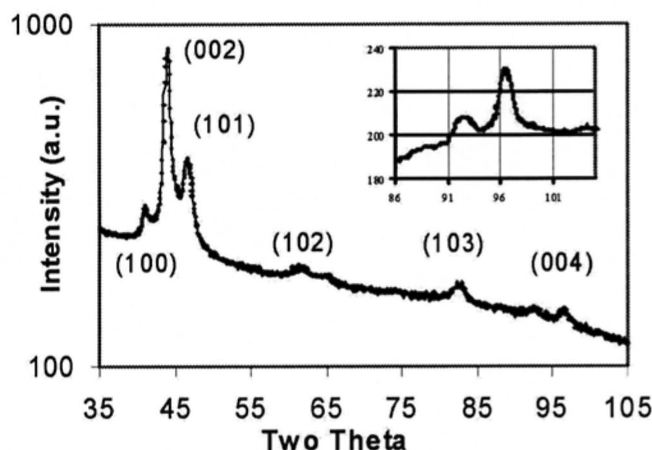

FIG.2. The in-plane GIXRD scan profile of the longitudinal medium A in the circumferential direction. The insert is the (004) peak obtained at a slow scan rate and small scan step in order to increase the resolution.

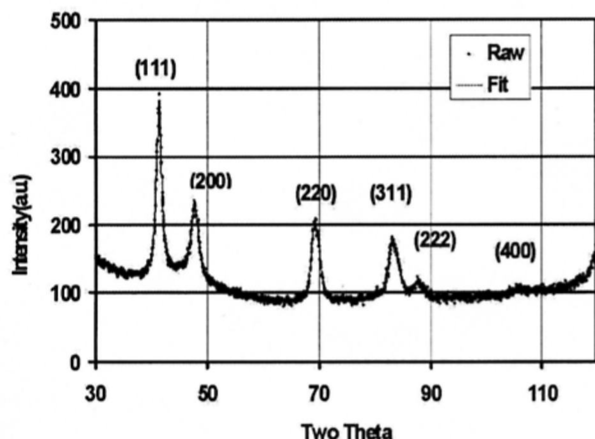

FIG.3. The in-plane GIXRD scan profile for the perpendicular recording medium: (Co/Pd)n superlattice.

III. GRAIN SIZE DISTRIBUTION

The theory of X-ray diffraction line-broadening analysis was developed more than 5 decades ago. It identifies two main types of broadening: the grain size and strain components. The former depends on the size of coherent domains which is not limited to the grains but may include effects of stacking and twin faults and subgrain structures; the latter is caused by any lattice imperfection. Therefore, in principle, the XRD peaks' shape and width allow an estimation of grain sizes and their distributions[4]. The crystal grain sizes are the extent of the coherent scattering regions, which can be determined directly by XRD technique. In pratice, the size of crystal core grain may be likely different from the physical grain size primarily due to compositional segregation and edge dislocations. However, the magnetic grain sizes should be closely associated with the size of crystal core grain.

The mean grain size and the grain size distributions are calculated by using the BWA method[2-4] without adopting Lorentzian shape correction. We have tacitly assumed that the column-like grains were grown epitaxially with the preferred orientation.

The GIXRD scan data were smoothed (or fitted) by using a finite impulse response (FIR) equiripple lowpass filter in order to retain the original diffraction shapes while reducing the noise. A zero-phase forward and reverse filter scheme was utilized to avoid distorting the diffraction peak shapes and their positions. Fig.4 shows the normalized grain size distribution from the (002) peak in sample A. Apparently the grain size distribution obtained from XRD indeed is close to a lognormal distribution, as illustrated in Fig.5. In comparison with the grain size information from the TEM measurement, the differences between the mean grain size measured by XRD and TEM is about 1.6nm. The measurement differences could reside in the following two aspects: (1) the instrumental broadening, which can be calibrated by using the standard sample, (2) the error of TEM measurements. The calibration procedure of instrumental broadening for GIXRD measurement will be discussed in detail in a forthcoming paper [6].

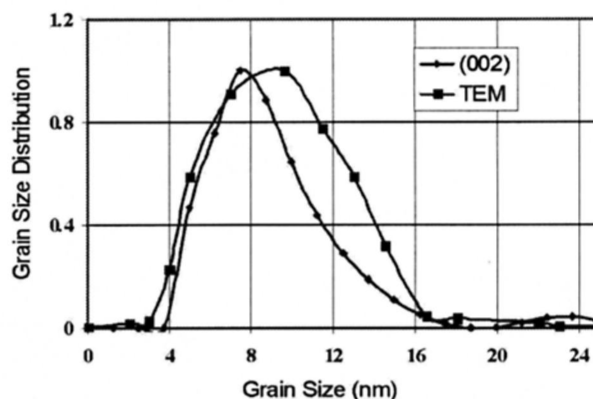

FIG.4. The normalized grain size distributions measured by using XRD and TEM in sample A.

The normalized grain size distributions for both peak (100) and peak (002) in the sample B are plotted in Fig.6, in comparison with the result from the TEM measurement. It should be pointed out that the fcc-(111) peak is located between the hcp-(100) peak and the hcp-(002) peak. We neglect the influence of the fcc-(111) peak on the hcp-(002)

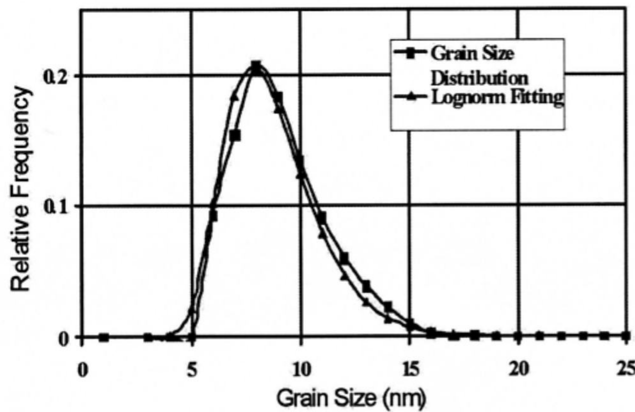

FIG.5. The measured grain size distribution in sample A is compared with a lognormal distribution (α=2.13 and β^2=0.09).

peak because the intensity of the fcc-(111) peak is far smaller compared with the intensity of the hcp-(002) peak. However, the peak intensity of the fcc-(111) may be comparable to the intensity of the hcp-(100) in some degree. We assume that the shape of the peak (100) is symmetric and peak flipping from left to right [2-3] has been performed for the grain size calculation.

Clearly, the grain size obtained from the peak (100) and peak (002) are quite close. In other words, in this recording medium, the shapes of grain are isotropic. However, the mean grain size for the (002) peak from XRD measurement is 10.8nm and the mean grain size from the (100) peak is 9.8nm, but the mean grain size from TEM is 12.5 nm. The discrepancy between TEM result and XRD results here seem to be larger than that in sample A, which may indicate the difference of the compositional segregation at grain boundaries. Overall, the conclusion is that the results from XRD measurements are consistent with the TEM results.

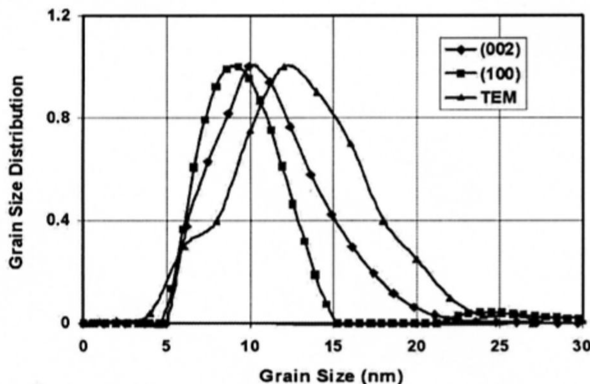

FIG.6. The normalized grain size distributions in sample B measured by both XRD and TEM characterization.

We also examined the grain size distribution in the (Co/Pd)n superlattice, which could have a potential for perpendicular recording. The measured mean grain size is 5.6 nm prior the strain correction. After the strain correction, the obtained grain size is around 7nm, which is still smaller than 10nm of TEM measured results. A possible explanation is that the interface roughness could be quite large, which leads to the relatively small coherent scattering domains in each Co layer. This picture is consistent with our grazing angle reflectivity measurement.

IV. SUMMARY

We have utilized GIXRD technique to determine not merely the mean grain size, but the entire grain size distributions in magnetic recording media via the BWA technique. In principle, XRD method has several advantages in terms of determining the grain size distributions. (i) Firstly XRD is a non-destructive analysis method. (ii) Secondly it can be used to estimate the grain size information over a relatively large area of recording media (in range of mm^2). (iii) Moreover, the precision of XRD measurements is quite high so that it could provide an effective way for monitoring recording media fabrication and grain size control. (iv) It only takes a few hours to examine the grain size information by using XRD technique, which makes this technique attractive to the recording media fabrication. Further studies are needed to calibrate GIXRD system to improve its accuracy. Although BWA technique is an old fashion line-broadening analysis method with certain drawbacks, it has remained the least constrained method for analyzing diffraction profile line broadening. Of course, another attractive attempt is to utilize the Rietveld refinement technique to conduct similar grain size evaluation for magnetic recording media.

V. ACKNOWLEDGEMENT

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