Scatter Diagram Analysis of Cr Segregation in Co–Cr Based Recording Media

Werner Grogger and Kannan M. Krishnan

***Abstract*—The segregation of Cr in Co–Cr based thin film recording media was measured using energy-filtered TEM images and quantitatively evaluated by the scatter diagram (SD) method. Following a description of such SD analysis it is applied to the measurement of Cr segregation in two CoCrPtB thin film recording media with different Cr contents. Taking the entire data sets into account, the two dimensional distribution of the nonmagnetic, high Cr phase can be visualized and determined quantitatively. Cr segregates to the intergranular regions and the sample with the higher Cr content shows a significantly higher and more uniform distribution of the nonmagnetic phase at the boundaries. This leads to better magnetic isolation between the grains and correlates with the better recording performance observed in the same sample.**

***Index Terms*—Cr segregation, energy-filtered TEM, recording media, scatter diagrams.**

I. INTRODUCTION

IN Co–Cr ALLOYS used for longitudinal recording media, enhancement in recording performance due to intergranular isolation is achieved by the segregation of Cr to the grain boundaries. An ongoing challenge is the development of a suitable analytical technique to measure the segregation of Cr at the required resolution (around 1 nm) and statistical significance. Transmission electron microscopy (TEM) techniques using energy dispersive X-ray spectrometry and electron energy-loss spectrometry line-scans across the grain boundaries, as well as energy-filtering TEM (EFTEM), have been used for such measurements [1]–[4]. These investigations have shown that Cr enrichment at grain boundaries can reach the onset of paramagnetism (Cr content $>\sim$24 at%) providing the necessary intergranular isolation, i.e., exchange-decoupling. However, reliable quantification from line profiles (with or without integration widths) is not straightforward, as signals from real media (film thickness around 15 nm) are usually small and rapidly approach the detection limits. In addition, these methods also require straight interfaces aligned normal to the surface.

Manuscript received October 13, 2000.

The work of W. Grogger was supported during his stay at Berkeley by the Max Kade Foundation. Work at LBNL/NCEM was supported by the Director, Office of Energy Research, Office of Basic Energy Sciences, Materials Sciences Division of the U.S. Department of Energy under Contract DE-AC03-76SF00098.

W. Grogger is with the Materials Sciences Division, National Center for Electron Microscopy, Lawrence Berkeley National Laboratory, Berkeley, CA 94720 USA, on leave from the Research Institute for Electron Microscopy, Graz University of Technology, Graz, Austria (e-mail: Werner.Grogger@TUGraz.at).

K. M. Krishnan is with the Materials Sciences Division, National Center for Electron Microscopy, Lawrence Berkeley National Laboratory, Berkeley, CA 94720 USA (e-mail: Krishnan@LBL.gov).

Publisher Item Identifier S 0018-9464(01)07322-8.

In this work, we present an alternative method of analysis using two-dimensional histograms calculated from element-specific EFTEM images [5]. This technique, which is called scatter diagram analysis, makes it possible to obtain reliable, quantitative information on the segregation, globally over the entire image area. Application of the scatter diagram analysis of EFTEM elemental maps is demonstrated by the measurement of Cr segregation in two CoCrPtB alloys with different Cr contents and recording performances. The higher Cr content alloy has a better signal to noise ratio (SNR) in recording measurements, which is in good agreement with the EFTEM results.

II. EXPERIMENTAL

The samples investigated were two commercial $Co_{0.8-x}Cr_xPt_{0.1}B_{0.1}$ magnetic thin film media, with the same layer/underlayer structure, but different Cr contents of 10 and 16 at% Cr respectively. Planview specimens of both samples were prepared using the conventional TEM preparation method of polishing, dimpling and ion-milling.

All EFTEM work was done on a Philips CM200/FEG microscope operated at 200 kV and equipped with a Gatan Imaging Filter (GIF). Elemental maps of Co and Cr were acquired using the three-window technique [6]. However, small amounts of oxygen, due to the surface oxidation of the alloy thin film samples, made it necessary to apply a four-window technique for the Cr maps in order to perform a reliable quantification [7]. Due to the uniform thickness and in order to keep additional generation of noise to a minimum, the elemental maps were quantified by normalizing them to the nominal composition values.

The computation of the scatter diagrams was performed within Gatan's DigitalMicrograph software; the script package is available upon request from the authors.

III. SCATTER DIAGRAM ANALYSIS

For the statistical analysis of the quantified elemental maps scatter diagrams were used [8], [9]. Scatter diagrams have proven to be an ideal method for determining and visualizing the correlation between images. However, in this work their main purpose was to provide an appropriate means to segment the images according to their gray values (i.e., elemental concentrations) and to find image areas within a certain compositional range.

A scatter diagram can be considered as a two-dimensional histogram showing the frequency of occurrence of gray value combinations of two images. In the case of a correlation between the two, one or more clusters can be seen in the scatter diagram,

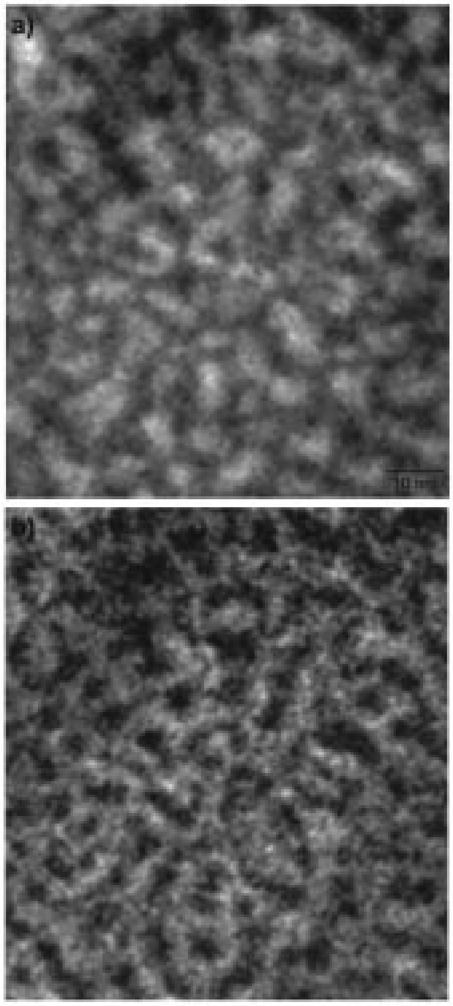

Fig. 1. EFTEM images of a CoCrPtB planview sample (16 at% Cr). (a) Co elemental map, (b) Cr elemental map. The Cr enriched grain boundaries can be seen in (b), whereas the same areas are depleted in Co (a).

each representing a different gray value combination. If maps showing elemental concentrations are used, each point in the scatter diagram corresponds to specific concentrations of both elements (i.e., chemical phases). Furthermore, the clusters can be mapped back to find the image areas where they originate from to create chemical phase images. The resulting trace backs do not necessarily correspond to real chemical phases, but, as in this work, show various compositional ranges.

IV. Results and Discussion

Fig. 1 shows a typical set of elemental maps for the 16 at% Cr sample. Due to segregation, an enrichment of Cr in between the grains, as well as Co depletion at the same areas can be clearly seen. Using these images as input, a Co–Cr scatter diagram is calculated as shown in Fig. 2 for the 16 at% Cr sample. One cluster is recognized in the scatter diagram, which indicates a continuous variation in composition from low Cr to high Cr regions. Additionally, the cluster is elongated along the diagonal showing an inverse correlation in composition (high Cr areas have low Co and vice versa) as expected.

In the following processing step, lines representing a specific Co/Cr ratio are used to segment the scatter diagram into different

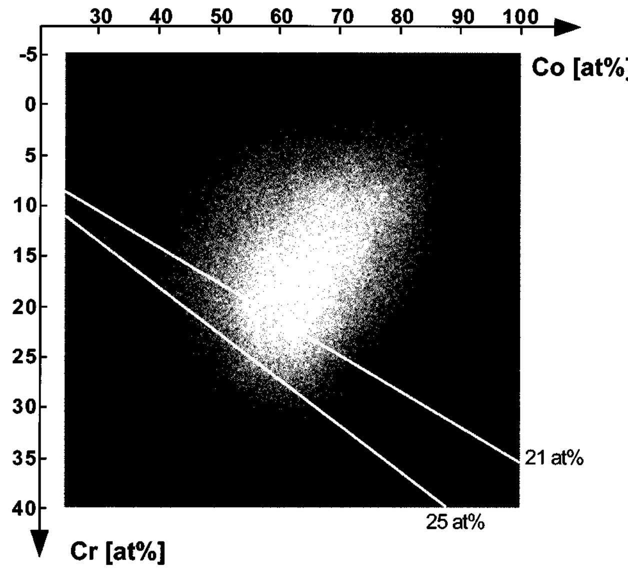

Fig. 2. Scatter diagram calculated from the elemental maps shown in Fig. 1. Lines corresponding to 21 and 25 at% Cr respectively were used to divide the scatter diagram into three different parts: <21 at% Cr, $21-25$ at% Cr, and >25 at% Cr.

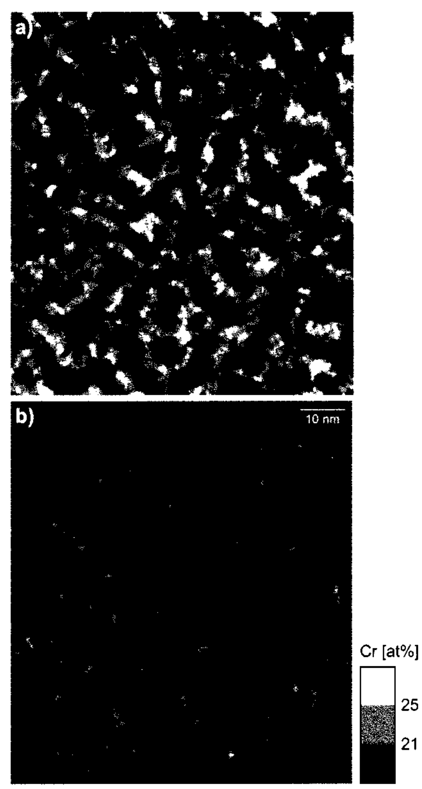

Fig. 3. Trace back of the Co–Cr scatter diagrams according to the segmentation shown in Fig. 2. (a) for the 16 at% Cr sample, (b) for the 10 at% Cr sample.

regions. Assuming constant Pt and B concentrations, a certain Co/Cr ratio corresponds to constant values for both Co and Cr. The good reason for drawing the lines at constant ratios instead of constant Cr values is to suppress thickness and diffraction effects and noise as much as possible. Unfortunately, the paramagnetic limit for the quarternary Co–Cr–Pt–B thin film media as a function of Cr content is not well known but it is expected to

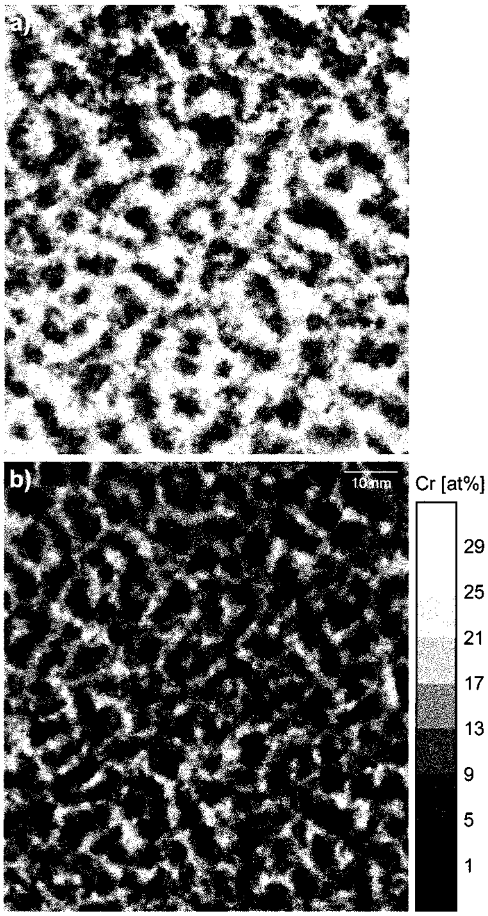

Fig. 4. Trace back of the Co–Cr scatter diagrams for a segmentation with nine different ranges.

be somewhere in the vicinity of 23 or 24 at% Cr (well known in binary Co–Cr alloys). Hence, we decided to segment the cluster into three regions: <21 at% Cr, $21-25$ at% Cr, and >25 at% Cr.

Doing this segmentation in an identical manner for both samples, trace backs are calculated in order to find the specimen areas which correspond to the three different concentration ranges. The resulting maps show areas within the same compositional range in the same shade of gray (Fig. 3).

For the higher Cr-content sample [Fig. 3(a)] a substantial fraction of the sample area shows a Cr distribution >25 at% Cr. Moreover, the higher Cr-content regions which are nonmagnetic show strong segregation and almost all grains appear to be at least partially surrounded by a nonmagnetic layer (magnetic isolation). The segregation in the 10 at% Cr sample looks very different. Only tiny spots of the nonmagnetic phase can be seen, indicating that the grains are far from being completely exchange-decoupled.

Fig. 4 is similar to Fig. 3 but used nine concentration ranges instead. It can be seen that Cr segregates to the grain boundaries in both samples. However, the local concentration of Cr at the grain boundaries is significantly different.

Almost the whole image of the 10 at% Cr sample shows Cr concentrations of 21 at% or less (99% of the image area), whereas a significant amount (21%) of the image area has a Cr content of 21 at% or higher for the 16 at% Cr sample. As discussed earlier, most of this 21% image area is localized at the intergranular boundaries.

The better grain isolation and therefore less exchange-coupling for the 16 at% Cr sample agrees well with magnetic and recording measurements (e.g., 2.5 dB better SNR) [5].

V. Conclusions

In this paper an efficient method is described for the evaluation of Cr segregation in CoCrPtB recording media. Quantitative information about Cr enrichment is extracted from the complete two-dimensional data sets of EFTEM images by scatter diagram analysis. Using this technique two thin film media with different Cr contents were investigated. The absolute difference in Cr content in this study was 6 at%. No generally valid value can be given on the concentration resolution, however we estimate that 1–2 at% should be easily detectable. The scatter diagram analysis yields a clear picture of the amount of Cr enrichment between the grains, which is significantly different for both samples. The higher Cr content sample shows a much better grain isolation which is in good agreement with the better recording characteristics.

Acknowledgment

The authors would like to thank R. A. Ristau (Seagate Recording Media) for helping with the acquisition and processing of the images, and R. Ranjan (Seagate Recording Media) for helpful discussions.

References

[1] Y. Hirayama, M. Futamoto, K. Kimoto, and K. Usami, “Compositional microstructures of CoCr-alloy perpendicular magnetic recording media,” *IEEE Trans. Magn.*, vol. 32, pp. 3807–3809, 1996.

[2] J. E. Wittig, T. P. Nolan, R. A. Ross, M. E. Schabes, K. Tang, R. Sinclair, and J. Bentley, “Chromium segregation in CoCrTa/Cr and CoCrPt/Cr thin films for longitudinal recording media,” *IEEE Trans. Magn.*, vol. 34, pp. 1564–1566, 1998.

[3] M. Futamoto, N. Inaba, Y. Hirayama, K. Ito, and Y. Honda, “Microstructure and micromagnetics of future thin-film media,” *J. Magn. Magn. Mater.*, vol. 193, pp. 36–43, 1999.

[4] N. Inaba and M. Futamoto, “Effects of Pt and Ta addition on compositional microstructure of CoCr-alloy thin film media,” *J. Appl. Phys.*, vol. 87, pp. 6863–6865, 2000.

[5] W. Grogger, R. A. Ristau, T. Thomson, S. D. Harkness, R. Ranjan, and K. M. Krishnan, “Quantitative measurement of Cr segregation in Co-Cr based magnetic recording media using energy-filtering transmission electron microscopy and scatter diagram analysis,” *Appl. Phys. Lett.*, submitted for publication.

[6] A. J. Gubbens and O. L. Krivanek, “Application of a post-column imaging filter in biology and materials science,” *Ultramicroscopy*, vol. 51, pp. 146–159, 1993.

[7] J. Bentley, J. E. Wittig, and T. P. Nolan, “Quantitative measurements of segregation in Co–Cr–X magnetic recording media by energy-filtered transmission electron microscopy,” in *Mater. Res. Soc. Symp. Proc.*, vol. 517, 1998, pp. 205–210.

[8] F. Hofer, W. Grogger, G. Kothleitner, and P. Warbichler, “Quantitative analysis of EFTEM elemental distribution images,” *Ultramicroscopy*, vol. 67, pp. 83–103, 1997.

[9] W. Grogger, F. Hofer, and G. Kothleitner, “Quantitative chemical phase imaging by means of energy-filtering transmission electron microscopy,” *Microchim. Acta*, vol. 125, pp. 13–19, 1997.