

Magnetization Reduction Due to Oxygen Contamination of Bias Sputtered $\text{Fe}_{35}\text{Co}_{65}$ Thin Films

Winnie Yu, James A. Bain, Yingguo Peng, and David E. Laughlin

Abstract—Obtaining high magnetization sputtered thin films with soft properties requires small grains with good exchange coupling between the grains. Contamination of grain boundaries during deposition will significantly degrade magnetic softness and reduce magnetization. In this paper, we report on such contamination induced by substrate bias during sputter deposition of $\text{Fe}_{35}\text{Co}_{65}$ films. 100-nm-thick $\text{Fe}_{35}\text{Co}_{65}$ films were sputtered at 100 W target power in 3 mtorr of Ar, with varying substrate bias from 0 to -300 V. A maximum value of magnetization of 2.35 T was observed at intermediate bias values, but the saturation magnetization of the films decreased nearly 10% for the samples at the highest bias. TEM analysis of the samples indicated an increase in oxide content and a decrease of grain diameter with increasing substrate bias. The loss of magnetization with increasing bias is quantitatively consistent with a nonmagnetic oxide shell of approximately 1 nm in thickness around each grain for all biases. The effect is simply that of increasing surface to volume ratio as the grains get smaller. A nonmagnetic shell of this thickness is expected to be sufficient to break exchange coupling between the grains and yield very poor soft properties, which were observed in these samples.

Index Terms—Co, Fe, high magnetization, oxygen contamination, substrate bias.

I. INTRODUCTION

THE AREAL density of hard disk drives increases at a rate of approximately 60% a year. As the density increases, the hard disk media requires increasingly higher values of coercive field, which makes it more difficult for today's magnetic recording heads to write on the media. As a result, improvements in recording heads are necessary for writing on future media. One method of achieving this is to change the material that is used for the recording head poles to one that has a high saturation magnetization and soft properties. One magnetic material that has potential to meet these requirements in recording heads is FeCo.

FeCo alloys have the highest induction (approximately 2.45 T) of all known magnetic materials at room temperature. This high saturation induction makes FeCo thin films desirable for use as recording head materials. Although FeCo alloys have the magnetic properties necessary for use in recording heads, the magnetic magnetization and soft properties are highly

dependent on film microstructure, which can be significantly affected by deposition conditions such as substrate biasing.

The addition of substrate biasing to the deposition of magnetic thin films is often used to manipulate microstructural features, such as stress or growth texture. Indeed, previous work has shown that substrate biasing in $\text{Fe}_{35}\text{Co}_{65}$ films can be used to obtain low stress films [1]. Additionally, in this paper we observe a reduction in grain size with increasing substrate bias, which is usually favorable for magnetic properties.

However, it was also found that the addition of biasing caused an unexpected increase in oxide contamination of the samples. This may be due to increasing bombardment of deposition chamber walls during sputtering that can dislodge water that may contribute oxygen to the depositing film. This paper presents the effects of oxide contamination on the magnetic magnetization (reduction) and coercivity (increase) of $\text{Fe}_{35}\text{Co}_{65}$ films sputtered with substrate biasing. It is reasonable that this contamination can cause reduced film magnetization (due to dilution by the oxide phase) [2] as well as high coercivity (due to the decreased exchange coupling between the grains) [3], [4]. A simple model of the dilution of the magnetization provides an estimate of the amount of the oxidation at the grain boundaries.

II. EXPERIMENT

100-nm-thick $\text{Fe}_{35}\text{Co}_{65}$ films were deposited in a dc magnetron sputtering system with an $\text{Fe}_{35}\text{Co}_{65}$ alloy target at 100-W target power in 3 mtorr of Ar. The films were deposited at a rate of 13 nm/min onto 1-in glass and Si wafers with substrate bias varying from 0 to -300 V. The magnetic properties of the samples deposited on glass were measured using a DMS 1660 vibrating sample magnetometer. TEM analysis was performed on the $\text{Fe}_{35}\text{Co}_{65}$ films on Si using a Philips EM420 and a JEOL 2000EX transmission electron microscope.

III. RESULTS AND DISCUSSION

A. Magnetic Characteristics

The behavior of the magnetization and the coercivity of the deposited $\text{Fe}_{35}\text{Co}_{65}$ films as a function of substrate bias are shown in Fig. 1. A maximum magnetization value of 2.35 T was observed at -100 -V bias, but the magnetization of the films decreased nearly 10% for the samples at the highest bias. The coercivity increased with increasing substrate bias, with a sharp change at -200 V. The sudden increase in coercivity at -200 -V bias is caused by the formation of stripe domains in the high

Manuscript received February 14, 2002; revised April 28, 2002. This work was supported by the National Science Foundation under Grant ECD-8907068 and by the National Storage Industry Consortium.

The authors are with the Data Storage Systems Center, Carnegie-Mellon University, Pittsburgh, PA 15213 USA (e-mail: wyu@andrew.cmu.edu).

Digital Object Identifier 10.1109/TMAG.2002.802126.

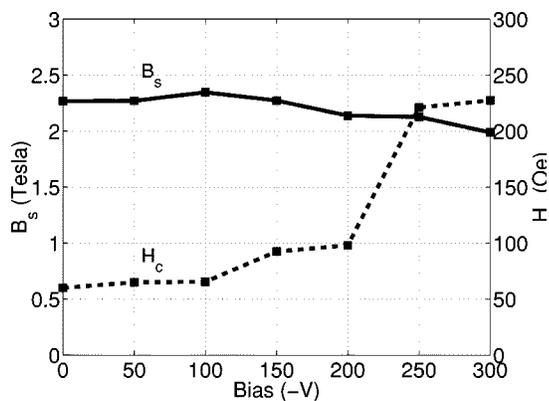


Fig. 1. Magnetization and coercivity versus substrate bias for Fe₃₅Co₆₅ deposited at 100 W and 3 mtorr.

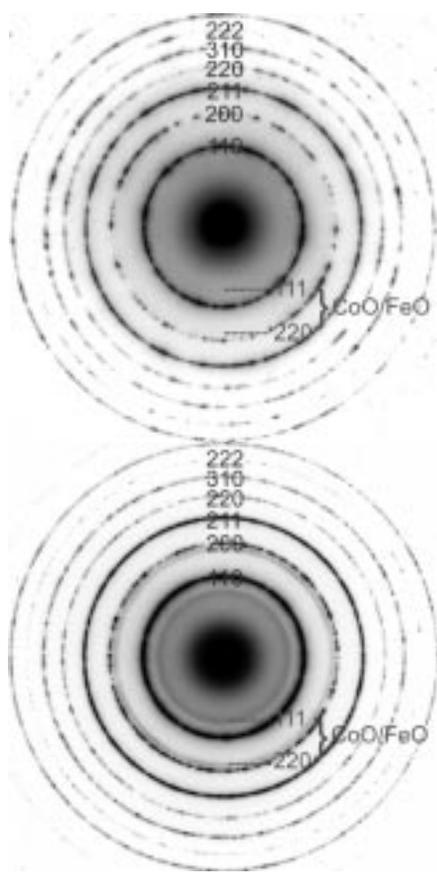


Fig. 2. TEM diffraction images of Fe₃₅Co₆₅ deposited at 0 and -300 V.

bias samples, which were determined through the observation of characteristic hysteresis loops. It is the increase in coercivity between 0 and -200 V that we focus on in this paper. The stripe domains occur due to the stress in the film, not the oxygen contamination.

In order to determine the cause of the decrease in magnetization and increase in coercivity, TEM analysis was performed on the Fe₃₅Co₆₅ films deposited at 0-, -150-, and -300-V bias.

B. TEM Analysis

The TEM diffraction images and ring intensity plots for Fe₃₅Co₆₅ deposited at 0- and -300-V bias are shown in Figs. 2

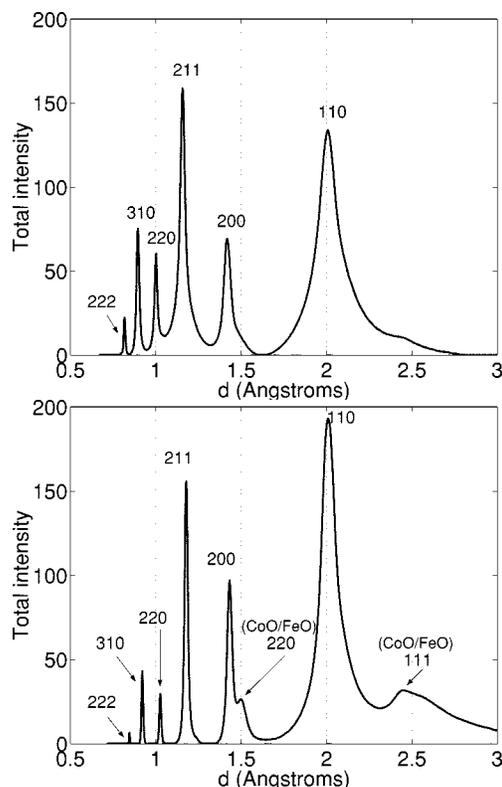


Fig. 3. TEM diffraction intensity plots of Fe₃₅Co₆₅ deposited at 0 and -300 V.

and 3, respectively. The TEM analysis indicated the presence of oxide (CoO/FeO) with a significant increase in the intensity of the oxide diffraction ring between 0- and -300-V bias. The TEM results also indicated a decrease in grain diameter from 75 to 25 nm with increasing substrate bias, as shown in Fig. 4. The decrease in grain size was unexpected since an increase in substrate bias typically increases atomic mobility on the surface of the developing film, which should increase the overall grain size. However, the amount of oxide contamination increases with increasing bias, which reduces atomic mobility and grain size.

Examination of the magnetic characteristics and the TEM results of the Fe₃₅Co₆₅ films suggest that although the decrease in grain size with increasing bias is usually beneficial for coercivity, an increase in coercivity was caused in the present case by grain decoupling caused by bias induced oxide contamination. The cause of decrease in magnetization may also be attributed to the increase in oxide contamination. In order to quantitatively determine the effect of contamination on magnetization, a simplified cubic grain morphology was used to model the behavior of magnetization as a function of oxide contamination.

C. Estimation of Oxidation

The grain morphology used to model the magnetization as a function of contamination is shown in Fig. 5 where a grain is approximated as a cube of length *d* with a nonmagnetic grain boundary of thickness *t*. The magnetization can be considered proportional to the ratio of the grain volume to the total volume of the grain plus the nonmagnetic grain boundary.

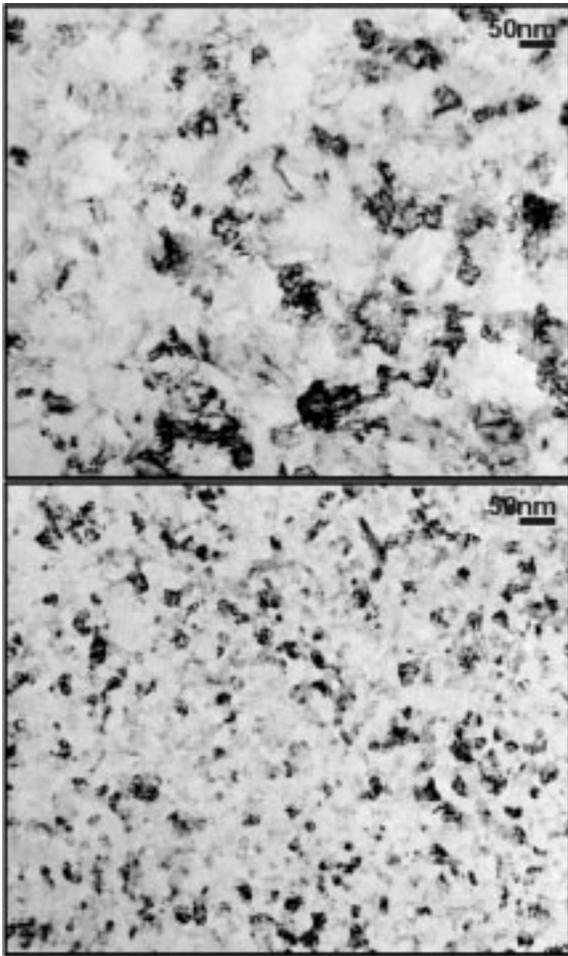


Fig. 4. TEM images of $\text{Fe}_{35}\text{Co}_{65}$ deposited at 0 and -300 V.

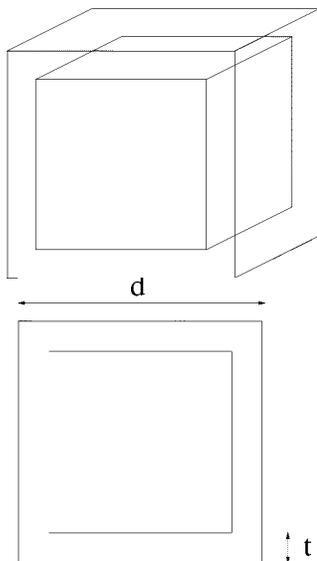


Fig. 5. Simplified cubic grain used to model magnetic magnetization.

The calculation of the theoretical behavior of the magnetization was performed using this model under the assumption that oxygen contamination in the grain boundaries produced a nonmagnetic shell of constant thickness t for varying grain sizes.

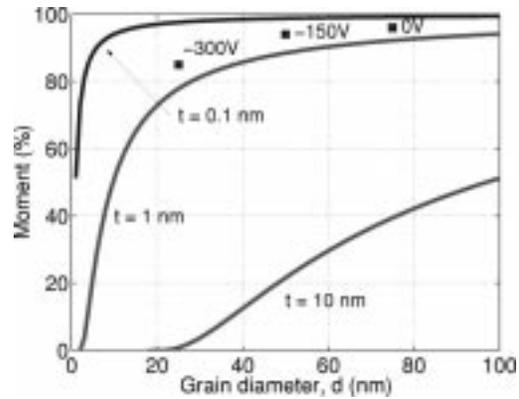


Fig. 6. Theoretical calculation of magnetic magnetization of a film composed of cubic grains, with varying oxide contamination.

The results for this calculation are shown in Fig. 6 for three different values of t . Also shown are the experimentally observed magnetization values (normalized) plotted as a function of grain size for the $\text{Fe}_{35}\text{Co}_{65}$ films deposited at 0, -150 , and -300 V.

From Fig. 6, it can be seen that for a constant t , the magnetization decreases with decreasing grain size. A comparison of the magnetization and grain size results for $\text{Fe}_{35}\text{Co}_{65}$ with the magnetization curves in Fig. 6 shows that the magnetic behavior of the experimental film is consistent with nonmagnetic grain boundaries approximately 1-nm thick.

IV. CONCLUSION

The results presented indicated that increasing the substrate biasing during the deposition of $\text{Fe}_{35}\text{Co}_{65}$ films leads to an increase in oxide contamination of the grain boundaries and a subsequent increase in coercivity and decrease in magnetization. A simple cubic grain morphology was used to model the behavior of magnetization with changing grain size and oxide contamination. The magnetization was found to decrease with increasing contamination and grain size. There was good agreement between the calculated magnetization and the behavior of experimental results for the deposited $\text{Fe}_{35}\text{Co}_{65}$ films, when a 1-nm-thick oxide shell was assumed to exist around each grain.

ACKNOWLEDGMENT

The authors would like to thank N. T. Nuhfer of the Materials Science and Engineering Department, Carnegie–Mellon University, in the TEM analysis.

REFERENCES

- [1] P. Zou, W. Yu, and J. A. Bain, "Magnetism of high moment soft magnetic thin films under stress," *IEEE Trans. Magn.*, vol. 35, Sept. 2002.
- [2] V. M. Pusekov, K. Moskalev, N. Pleshanov, A. Schebetov, V. Syromyatnikov, V. Ul'yanov, A. Kobev, and O. Nikonov, "The effect of oxygen presence in sputtering chamber on magnetization of thin FeCo films," *Physica B*, vol. 276–278, pp. 654–655, 2000.
- [3] F. J. Cadieu, M. A. Russak, and R. G. Pirich, "Magnetic properties of iron rich sputtered Fe–Al films," *J. Magn. Magn. Mater.*, vol. 54–57, pp. 1598–1600, 1986.
- [4] T. Hikosaka, T. Komai, and Y. Tanaka, "Oxygen effect on the microstructure and magnetic properties of binary CoPt thin films for perpendicular recording," *IEEE Trans. Magn.*, vol. 30, pp. 4026–4028, Nov. 1994.