





#### Conference Paper

# Transmission Electron Microscopy Study of Magnetic Domain of Cobalt-Samarium Thin Films Fabricated Using DC Magnetron Sputtering Technique

#### Erwin Amiruddin<sup>1</sup> and Adhy Prayitno<sup>2</sup>

<sup>1</sup>Department of Physics Faculty of Mathematics and Natural Sciences Riau University, KampusBinaWidyaSimpangBaru, Tampan, Pekanbaru, Riau - Indonesia 28293 <sup>2</sup>Department of Mechanical Engineering Faculty of Engineering Riau University, KampusBinaWidyaSimpangBaru, Tampan, Pekanbaru, Riau - Indonesia 28293

**Abstract** Alloys of cobalt samarium (Co-Sm) in the form of thin films were fabricated using dc magnetron sputtering technique. The films were fabricated as a function of samarium concentration ranging from o to 28 at.% in order to investigate the relationship between microstructure, coercivity and magnetic domain structure. Magnetic domain structures in the films have been studied by Lorentz microscopy using transmission electron microscopy (TEM). In this technique, the TEM was operated in the defocused mode. The results show that the magnetic image of Co<sub>90</sub>Sm<sub>10</sub> film has fairly coarse structure with magnetization ripple and the domains ranging over 200-300 nm. The domain size is much larger than the grain size of Co<sub>90</sub>Sm<sub>10</sub> film. The "multiparticle" or interaction domains suggested that there is strong exchange coupling between the magnetization of the neighbouring grains inside each of them. The hysteresis loop for this film shows a small coercivity with high magnetization value and high loop squareness, indicating a greater proportion of magnetic material.

Received: 1 August 2016 Accepted: 18 August 2016 Published: 6 September 2016

Publishing services provided by Knowledge E

Corresponding Author: Erwin

erwin\_amiruddin@yahoo.

Amiruddin; email:

com

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Selection and Peer-review under the responsibility of the ICoSE Conference Committee.



Keywords: CoSm thin films, coercivity, magnetization, domain, and loop squareness

## 1. Introduction

The relationship between microstructure and magnetic anisotropy or coercivity in magnetic materials with magnetic domain configuration especially when the materials in the form of thin films has long been a subject of interest for their potential applications such as high density magnetic recording media. According to previous researcher [1] magnetic recording media for high-density magnetic data storage with low noise require a material consisting of small and magnetically isolated grains. In small grain sizes of about 10 nm or below, high magnetocrystalline anisotropy is needed in order to avoid thermal fluctuation that tends to destabilize the magnetization of the recorded bits [2]. Early attempts to grow CoSm alloys in the form of thin films exhibiting large coercivity values and could be used as a high-density magnetic recording medium were carried out by some researchers [3]. Some recording experiments have been investigated, for example, by Velu and Lambeth [4] and Velu *et al.* [5]. However,



the growth characteristics of CoSm alloys including the concentration range [6], the epitaxial relation between CoSm alloy films and underlayer materials [7], interaction effects [8], and the magnetic switching volume [9] suggest that improved magnetic properties of CoSm films could be obtained by utilization and optimisation of appropriate deposition conditions.

It is well known that magnetic properties of thin magnetic layers have predominantly affected by several parameters such as preparation conditions, films fabrication methods and utilization of underlayer materials. In order to obtain high density magnetic recording with low noise media it is necessary to have thin films with high coercivityandloop squareness and reduced magnetic domain size. Therefore, understanding domain size relationship with coercivity is one of the important steps in further development of high density magnetic recording media. The morphology of rich non magnetic phase and magnetic domain configuration are important since these phase can act as the nucleation sites of reverse domains. This phenomenon should be understood in order to obtain the best method in developing higher coercivity values of magnetic thin films. Observed magnetic domain structure is one of the methods to studythe mechanisms of magnetic reversal in magnetic thin films. Previous researcher [10] have studied and observed magnetic domains of some ferromagnetic alloys in the form of thin films. Observations and analyses of magnetic domain structure for thin films have been studied by variety of method [11,12].

## 2. Experimental Procedures

Alloys of cobalt samarium in the form of thin films were fabricated by depositing Sm and Co onto Si (100) substrates using dc magnetron sputtering technique at JOULE laboratory Salford University UK. The magnetron sputtering system that has been used in this work was an Ion Tech 2000 UHV Deposition System available at Salford University. A pneumatically actuated gate valve isolates these two chambers. The sputtering system has a rotating carousel and shutters controlled by computer and able to fabricate the films with varying input power for Co and Sm targets. The pressure inside the sputtering chamber prior to deposition was 5 x  $10^{-8}$  mbar. The films were fabricated as a function of samarium concentration ranging from o to 40 at. % under the sputtering condition of  $12 \times 10^{-3}$  mbar argon gas pressure. The base pressure inside the sputtering chamber prior to deposition was  $5 \times 10^{-8}$  mbar. Magnetic domain structures in the films have been studied by Lorentz microscopy using transmission electron microscopy (TEM, JEOL 3010, with in situ ion beam bombardment for radiation damage studies). In this technique, the TEM was operated in the defocused mode. The compositions of CoSm films were analysed by energy dispersive spectroscopy (EDS). Magnetic properties such as coercivity (Hc), loop squareness (S) of the films were measured using alternating gradient force magnetometer (AGFM).

## 3. Result and Discussion



**Figure** 1: Magnetic domain structure of (a)  $Co_{90}Sm_{10}$  and (b)  $Co_{80}Sm_{20}$  films observed using Lorentz microscopy.

#### 3.1. Magnetic Domain Structure

The effects of the CoSm film composition on magnetic domain structure are analysed through the Lorentz microscopy technique using electron transmission microscopy (TEM). In this technique, the TEM was operated in the defocused mode. Figure 1A and (B) show Lorentz microscopy images for  $Co_{90}Sm_{10}$  and  $Co_{80}Sm_{20}$  thin films respectively. The magnetic image of  $Co_{90}Sm_{10}$  film shows a fairly coarse structure with magnetization ripple and the domains ranging over 200-300 nm. Here the domain size is much larger than the grain size of  $Co_{90}Sm_{10}$  film (Fig. 2). The "multiparticle" or interaction domains shown in Fig.1a suggest there is strong exchange coupling between the magnetization of the neighbouring grains inside each of them. The hysteresis loop for this film (Fig. 4A) shows a small coercivity with high magnetization value and high loop squareness, indicating a greater proportion of magnetic material. The film with increased Sm concentration exhibits a very fine micromagnetic structure with less well defined magnetic domains and domain walls. This can be seen in Fig. 1B.

#### 3.2. Microstructural Properties of Samples

The TEM image shown in Fig 2A reveals the detailed microstructure of a  $Co_{90}Sm_{10}$  film. The grains are non-uniform with irregular shapes and are identified by similar lattice fringes observed in the HRTEM image. Each grain is separated from the adjacent grain by grain boundaries labelled by letters GB. These grain boundaries are several nmin width, have darker contrast and are supposed to contain amorphous material without an obvious crystal structure. This film contains small crystals (nanocrystals) with lattice fringes as indicated by arrows.Co<sub>80</sub>Sm<sub>20</sub> films show remarkably different grain morphology as shown in Fig. 2B. The film contains smaller (3-5 nm) size nanocrystals with distorted lattice fringes surrounded by disordered material as indicated by arrows. The electron diffraction pattern from this film shown diffuse rings characteristic of amorphous material. Moreover, it is clear that lattice fringes become less visible as Sm concentration is increased, indicating a highly defective increasingly amorphous state. Further increase of Sm concentration leads the distorted lattice fringes to completely disappear as shown in Fig.2C. This indicates that at higher Sm concentration the film becomes almost completely amorphous. This result is supported by the selected area diffraction patterns inserted in Fig. 2C showing extremely diffuse rings. The decrease



**Figure** 2: TEM image of (A)  $Co_{90}Sm_{10}$  film showing the crystallites in the amorphous matrix with grain boundaries labelled by letters GB, (B)  $Co_{80}Sm_{20}$  film showing more uniform grains which are well separated from their neighbours and (C)  $Co_{72}Sm_{28}$  thin film. Arrow indicates nanocrystals.

in the coercivity of CoSm films for Sm concentration beyond 20 at. % is attributed to the inception and completion of the amorphous state.

### 3.3. Magnetic Properties

The magnetic properties such as coercivity of the CoSm thin films were obtained from alternating gradient force magnetometry (AGFM) measurement. Fig. 3 shows the important effect of Sm concentration on the coercivity of CoSm thin films. As seen with an initial increase of samarium concentration, the coercivity increases and reaches a maximum value at around 19 - 22 at. % Sm, followed by a decrease with further increase in Sm concentration. The coercivity maximum at 19 - 22 at. % Sm was found to lie between the crystalline  $Co_5Sm$  and  $Co_7Sm_2$  compound compositions.

The convex dependence of coercivity on the samarium concentration is governed by several mechanisms depending on samarium concentration of the films. In the range between o and 20 at. % Sm, the average grain size increases slightly as shown in the TEM micrographs in Fig. 2, while the grain separation also increases with the addition of Sm concentration from o to 20 at.%. The grain distribution for the film with Sm concentration of 20 at. % is more uniform than that of film with lower Sm concentrations i.e., 10 at. %. Moreover, the degree of crystallinity decreases as Sm concentration is increased. Thus the increase of coercivity in this range can be qualitatively understood in terms of the combined effect of initial increase in size of relatively tightly coupled grains followed by the increase in grain separation, which ultimately reduces exchange coupling between weakly coupled grains or clusters and uniformly sized grains. However, in the rangebetween 20 and 40 at. % Sm, the size continues to decrease. This result is supported by the selected area diffraction (SAD) pattern where the diffraction rings become broader inserted in Fig.2, suggesting that the degree of crystallinity decreases as Sm concentration is increased. This effect could certainly lead to the decrease in the coercivity for the films with Sm concentration of more than 20 at. %. Moreover, the convex dependence of coercivity on Sm concentration could be related to retained regions of the Co<sub>17</sub>Sm<sub>2</sub>, Co<sub>5</sub>Sm and Co<sub>7</sub>Sm<sub>2</sub>phases. These are marked on Fig. 3. The highest value of the coercivity of CoSm thin films which is obtained in the region around  $Co_{80}Sm_{20}$  is close to the putative  $Co_5Sm$  and  $Co_7Sm_2$ phase boundary.





**Figure** 3: The coercivity of  $Co_{100-x}Sm_x$  thin film as a function of Sm concentration (at. %).

Figure 4 shows typical hysteresis loops measured by AGFM for three representative compositions of  $Co_{100-x}Sm_x$  alloy thin films; for x =10 the film exhibits low coercivity, x = 20 gives high coercivity and x = 40 much lower coercivity. For the film with low samarium concentration (10 at. % Sm) shown in Fig. 4 (a), the coercivity is small (340 Oe). The hysteresis loop is almost square with squareness or remanenceratio  $(M_r/M_s)$  of nearly unity. As already stated, when the concentration of samarium is increased to 20 at.%, the coercivity is at its highest (1254 Oe) with a remanence ratio less than unity as shown in Fig. 4(b). Fig. 4(c) shows the hysteresis loop for a film with samarium concentration of about 40 at.% which corresponds to a coercivity of 160 Oe. It is worth noting that in this hysteresis loop the squareness is much less then unity and contrasts strongly with that in Fig 4(a).

The variation in hysteresis loop squareness (*S*) as a function of the Sm concentration of the films is shown in Fig. 5. The loop squareness is found to decrease as the Sm concentration is added. One possible mechanism for the higher *S* values in low Sm concentrations is a greater exchange coupling interaction resulting from a small grain separation as evident in TEM images (Fig. 2). This is supported by the large domain size as shown in Fig. 1(a). The generally lower loop squareness (*S*) observed in the films with higher Sm concentration can be attributed to less interaction between grains. This was supported by the small domain size as shown in TEM micrograph (Fig.1b) obtained for these films, which is typical of more isolated micromagnetic units. This result is in agreement with the observation by previous researcher [13].

### 4. Conclusion

The domain size of cobalt samarium alloy  $(Co_{90}Sm_{10})$  is much larger than the grain size of that alloy in film. The "multiparticle" or interaction domains suggest there is





**Figure** 4: Hysteresis loops for  $Co_{100-x}Sm_x$  thin film with varying samarium concentration (a) x=10, (b) x=20 and (c) x=40.



**Figure** 5: Variation of squareness (*S*) as a function of samarium concentration.

a strong exchange coupling between the magnetization of the neighbouring grains inside each of them. The hysteresis loop for this film shows a small coercivity with high magnetization value and high loop squareness, indicating a greater proportion of magnetic material. It is probable that the domain boundaries occur at grain boundaries



where the intergranular exchange coupling is not strong. The film with increased Sm concentration exhibits a very fine micromagnetic structure with less well defined magnetic domains and domain walls. For films with Sm concentration of 28 at. % and more, no Lorentz image could be observed, implying that the film has very small domains and/or for very low magnetization both of which make it difficult to resolve by any contrast Lorentz microscopy. The maximum value of the film's coercivity lies between the Co<sub>5</sub>Sm and Co<sub>7</sub>Sm<sub>2</sub> equilibrium compound compositions. This behaviour could be due to a contribution of high magnetic anisotropy from small regions of Co<sub>5</sub>Sm; no evidence of this compound was found in the electron diffraction patterns.

# 5. Acknowledgments

The author would like to thank P.J. Grundy for his valuable discussions of the manuscript. C.A Faunce provided helpful technical advice and assistance.

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