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Optimization of sputtering parameters for Sm—Co thin films using design of experiments

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ABSTRACT

A design of experiments (DOE) study on the optimization of DC magnetron sputtering parameters for Sm—Co films was carried out using a Taguchi-fractional factorial, L8 (2^{4-1}) design methodology. Four important sputtering parameters, viz., sputtering pressure, DC power, substrate—target distance and sputtering time were considered in their upper, standard and lower levels of their predefined range in order to investigate the range of processing conditions and their effect on the film quality. The attributes of Sm—Co thin films were quantified with respect to surface roughness, thickness, crystallite size, phase composition and coercivity. The significance of each process parameter as well as the optimal combination of sputtering parameters to achieve the desired film characteristics such as finer crystallite size, low surface roughness and high coercivity was obtained using statistical analysis of the experimental results by the analysis of variance (ANOVA) method.

1. Introduction

Permanent magnetic films play an important role in the realization of many micro-electromechanical system (MEMS) devices [1,2]. One of the major requirements for such applications is the need to deposit magnetic films with high coercivity and large energy product on silicon substrates. Sm—Co system which exhibits a variety of hard magnetic phases such as $SmCo_3$, Sm_2Co_7, $SmCo_5$, $SmCo_7$ and Sm_2Co_{17} is an ideal system for such applications in thin film form [3]. Sm–Co films can be deposited by various physical methods such as sputtering [4–7], molecular beam epitaxy [8], pulsed laser deposition [9] and electron beam evaporation [10]. Among all these techniques, sputtering has been recognized as the most promising route for depositing Sm—Co films [4–7] and the desired Sm—Co hard magnetic phases such as $SmCo_5$, Sm_2Co_{17} and Sm_2Co_7, as well as thickness can be achieved by controlling the sputtering parameters such as pressure, power, time and substrate—target distance. Recently, considerable efforts have been made on investigating the effect of individual process parameters on the structure and phase composition of Sm—Co films during DC magnetron sputtering [11–13]. However, studies on the simultaneous effect and interaction of two or more sputtering parameters on the characteristics of Sm—Co thin films are still lacking. Such an understanding would be essential for these films to find applications in MEMS devices.

Design of experiments (DOE) techniques have been shown through many studies to be an efficient method in optimizing the process parameters for deposition of coatings as well as for understanding the effects of deposition parameter on coating properties [14,15]. DOE techniques have also been applied to optimize sputtering parameters for various thin films as listed in Table 1 [16–21]. In this study, we further explore the potential of DOE for optimization of sputtering parameters for Sm—Co thin films using Taguchi-type fractional-factorial design. A ‘two-level four-factor fractional-factorial analysis’ model was constructed to study the effect of simultaneous interaction of sputtering parameters on the characteristics of Sm—Co films. Analysis of variance (ANOVA) was carried out to understand the influence of sputtering process parameters on the properties of the Sm—Co thin films. The present work can be distinguished from the earlier work of Xu et al. [16,17]; wherein the effect of sputtering parameters such as target—substrate distance, DC power, sputtering pressure and time were studied exclusively on the coercivity of Sm—Co thin films. In contrast, in the present study, surface roughness, crystallite size, phase formation, microstructure and coercivity of the Sm—Co film were taken into account for studying the influence of sputtering

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Table 1
Design of experiments strategies applied to various thin films for optimization of sputtering process parameters.

Type of film deposited	Type of DOE method	No. of experimental runs (l^{f-p})	Ref.
Sm—Co/Cr	Fractional-factorial	3^{4-2}	[16,17]
TiSiN	Fractional-factorial	3^{4-2}	[18]
ZnO:Al	Fractional-factorial	3^{4-2}	[19]
In—Sn—O	Fractional-factorial	3^{4-2}	[20]
AlN	Fractional-factorial	4^{5-3}	[21]

l denotes no. of levels of each factor investigated, *f* denotes no. of factors investigated and *p* denotes the size of the fraction of the full factorial used.

parameters and these attributes of Sm—Co thin film are correlated with the changes in operating parameters. DOE was also used in optimizing sputtering processing parameters to produce Sm—Co films of desired characteristics. In fact, such a study is an essential requirement toward fabricating micro-magnetic devices; as these devices demand not only high coercivity Sm—Co films but also films of low surface roughness and fine crystallite size for a given thickness.

2. Experimental procedures

Sm—Co alloy target prepared by powder metallurgy route and having a nominal composition of $Sm_{21}Co_{79}$ was utilized for the sputtering experiments. All the experimental runs of Sm—Co films were carried out on Si(1 0 0) substrates using a DC magnetron sputtering system under a typical base pressure of 1×10^{-6} Torr. A Taguchi-style design of experiment [22], i.e. L8 (2^{4-1})-eight run, fractional-factorial, studying four process parameters, each at two levels, was employed to study the effect of selected process parameters on the quantitatively measured thin film characteristics. Prior experience was used to identify the major sputtering parameters and their respective operational ranges in the DC magnetron sputtering process. Care was taken to avoid overwhelming the matrix because of one parameter having an unusually broader range than the others. The main process parameters for DC magnetron sputtering process are sputtering pressure (S1), DC power (S2), substrate—target distance (S3) and sputtering time (S4). Two levels, viz., low and high were selected for each variable around a standard setting. The natural and coded values of main variables are given in Table 2. Sputtering experiments D01 through D09-representing the nine runs evaluated (including the standard settings) with the Taguchi L8 approach are shown in Table 3. The experimental runs of a given design were performed in a random order to reduce the influence of potential systematic errors.

After the experiments were conducted as per the designed factors, the films were characterized for the following attributes, viz., surface roughness, thickness, crystallite size, phase composition and coercivity. The film thickness and average surface roughness were measured using a stylus profiler (Model XP-1, Ambious

Table 2
Design of experiments with main variables.

Process parameter/ factor	Levels			Symbol
	Lower (−1)	Standard (0)	Higher (+1)	
Gas pressure (mTorr)	06	10	14	S1
DC power (W)	30	50	70	S2
Substrate—target distance (mm)	75	100	125	S3
Sputtering time (min)	30	45	60	S4

technology, USA). A Philips X-ray diffractometer was used to identify the various phases present in the films. Microstructural features of the films were observed using field emission scanning electron microscopy (FESEM, ZEISS); while magnetic measurements were carried out by vibrating sample magnetometer (VSM, DMS model 1600).

The experimental data was subjected to ANOVA for each specific film attribute to select the significant level of the process parameter and to understand the magnitude of influence that each variable had on the film properties. In addition, the *F*-test was also used to determine which process parameters have a significant effect on the film characteristics. The *F*-test (*F*-ratio) for a given/known degrees of freedom corresponding to any process parameter is defined as the quotient of the mean square of factor (MS_{factor}) and mean square of error (MS_{error}) [23]. From the ANOVA calculations, percent contribution (ρ%) was calculated as follows [23]:

$$\%\text{Contribution factor} = \frac{\text{Sum of Squares for the factor}\,(SS_{factor})}{\text{Sum of Squares for the total}\,(SS_{total})} \quad (1)$$

$$\%\text{Contribution error} = \frac{\text{Sum of Squares for the error}\,(SS_{error})}{\text{Sum of Squares for the total}\,(SS_{total})} \quad (2)$$

The ρ value indicates the influence of the process parameter on the measured film attribute, with a larger value indicating a stronger influence. The description of statistical terms used in this study as well as their significance is given in Refs. [23–25].

3. Results and discussion

3.1. Characterization of sputtered Sm—Co thin films

The Sm—Co film characterization results for the experiments D01 through D09 are given in Table 4. The ANOVA results for the individual process parameters on the film characteristics such as thickness, surface roughness, crystallite size, phase composition and coercivity are enumerated in Table 5.

3.1.1. Thickness

The thickness of the Sm—Co films was measured by surface profiler and the values ranged from 155 to 1086 nm as shown in Table 4. From the *F*-test, it was found that for lowering the film thickness the substrate—target distance and sputtering time were the most important parameters at 5% significance level whereas

Table 3
Taguchi experimental design test matrix (L_8) for sputtering of Sm—Co films.

Run no.	Order no.	Gas pressure S1 (mTorr)	DC power S2 (W)	Substrate—target distance S3 (mm)	Sputtering time S4 (min)
D01	03	14/+1	70/+1	125/+1	60/+1
D02	09	14/+1	70/+1	75/−1	30/−1
D03	02	14/+1	30/−1	125/+1	30/−1
D04	08	14/+1	30/−1	75/−1	60/+1
D05	04	06/−1	70/+1	125/+1	30/−1
D06	06	06/−1	70/+1	75/−1	60/+1
D07	05	06/−1	30/−1	125/+1	60/+1
D08	07	06/−1	30/−1	75/−1	30/−1
D09	01	10/0	50/0	100/0	45/0