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Investigation of soft magnetic properties of Ni/Cu multilayer films: Definitive screening design and response surface methodology

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ABSTRACT

Ni/Cu multilayer films have been found to be highly promising due to their applications in magnetoresistive sensors. Therefore, this study aims to find the optimum conditions for the production of Ni/Cu films with soft magnetic properties of by monitoring the maximum saturation magnetisation, M_s, and minimum coercivity, H_c. Thus, a relatively new class of experimental design called "definitive screening design (DSD)" was adopted for this investigation. Subsequently, response surface methodology (RSM) was applied to obtain an optimum deposition recipe for soft magnetic Ni/Cu multilayer films. A 13-run experiment from DSD approach was employed on the deposition of the multilayer films. Magnetic properties of the films were measured by a vibrating sample magnetometer. The influence of simultaneously varying factors of total thickness (TT), Ni deposition rate (Ndr), Cu deposition rate (Cdr), Ni layer thickness (Nlt) and rotation of the substrate (RS) on maximum M_s and minimum H_c responses was investigated. The most effective and interactive factor that was obtained from mean response characteristics and counter plots for both M_s and H_c values is Nlt. The optimum experimental conditions were obtained as TT (100 nm), Ndr (0.02 nm/s), Cdr (0.1 nm/s), Nlt (8.8 nm) and RS (20 rpm). After experimental analysis of optimisation process, the M_s of the films increased from 472.5 to 537.2 emu/cm^3, while the H_c decreased from 37 to 28 Oe. Structural analysis of the films was carried out by X-ray diffraction. All films showed face-centred cubic (fcc) structure and the preferential orientations were (111) for the films. The quadratic regression was successfully applied and shown to estimate the M_s (522.6 emu/cm^3) and H_c (31 Oe) values by using optimal conditions. It is observed that the experimental results and predicted values were very close to each other. The results demonstrated that optimal values for the production factors can be efficiently used to obtain maximum M_s and minimum H_c of the films synthesis process.

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1 Introduction

In recent years, researchers have tried to explain what exactly happens or changes in magnetic properties of materials to manufacture more qualified and useful technological devices or tools [1–3]. In the same manner, multilayers have extensively been recently studied [2, 3]. With the reason of the interest, multilayers have a wide range of applications, from simple devices to the advanced technologies [4, 5]. The multilayered structure of nickel and its alloys is very commonly used as they have useful magnetic properties for specific applications [6]. The studies of nickel and copper multilayers are also very common [7–11]. And, it is well known that Ni/Cu multilayer films are very easy to produce with many production techniques [8, 12]. Especially using the sputtering method to produce these structures is widely preferred, due to advantageous of process control [13]. For this reason, well-defined experimental steps are important to produce Ni/Cu multilayer films with soft magnetic properties. However, depending on process factors, Ni/Cu multilayer films with different properties can be formed. Therefore, it is very important to apply experimental routines with a well-defined protocol and planning practical steps can significantly improve the quality of products. Thus, to obtain Ni/Cu multilayer with the desired specifications, such as maximum saturation magnetisation, M_s, and minimum coercivity, H_c, it is important to understand the production parameters, called factors in the definitive screening design (DSD). If the process shows numerous factors, investigating the connections between factors and response, DSD method is used to determine the values of factors over a response [14, 15]. By now, the influence of limited number of factors was used to study Ni/Cu multilayers using "one factor at a time" approach [3, 16, 17]. Under study, design of experiments technique was used to evaluate all factors together to produce soft magnetic Ni/Cu multilayer films with maximum M_s and minimum coercivity H_c. In the production process, five factors affecting the values of production process, including total thickness (TT), Ni deposition rate (Ndr), Cu deposition rate (Cdr), Ni layer thickness (Nlt) and rotation of the substrate (RS), were obtained from previous studies [3, 16, 17]. The experiments were carried out based on a 13-run experiment suggested by the DSD. And, the main effects, interactions and optimisation of these factors with responses were statistically studied with the help of the response surface methodology (RSM). In addition, quadratic regression analysis was applied to predict the experimental values. This study highlights the values of DSD and RSM in optimising properties for the products and materials, and it demonstrates their role and potential application in material processing.

2 Experimental

2.1 Deposition of the films

Magnetic Ni/Cu multilayer films were deposited by using a sputtering system (MANTIS, Q-Prep 500). The advantages of this system for multilayer production are that allows the independent layer depositions of desired materials type due to possessing two magnetron sources. Also, the system makes it possible to easy control of each layer thickness and hereby the total thickness of sputtered multilayers. Nickel and copper targets have 99.99% purity, 50.8 mm diameter and 2 mm thick (Kurt J. Lesker Company). An acetate layer was chosen as a substrate as it can be easily cut and have no magnetic properties. Prior to deposition, the substrate was cleaned as explained in Ref. [16]. Once cleaning finished, the substrate was dried at room temperature and then placed as opposite to the targets with a stable distance at each deposition. The deposition of the films was done in the argon atmosphere of 40 sccm at ambient temperature in the vacuum system. Emerged plasma was maintained until the desired thickness was obtained onto the acetate substrate. The thicknesses of the films were monitored and determined by using a quartz crystal thickness monitor. The multilayer films can be symbolised as x[Ni(9 nm)/Cu(1 nm)], x[Ni(1 nm)/Cu(9 nm)] and x[Ni(5 nm)/Cu(5 nm)], separately. The bilayer number (x) was regulated as 10, 15 and 20 to obtain different total thicknesses of 100, 150 and 200 nm, respectively. The Ni/Cu multilayer films were preserved in a desiccator containing silica gel so that no oxidation on the films can occur. The films were deposited according to the recipes provided by the DSD. The representation factors and levels are presented in Table 1. The 13-run experimental design is also shown in Table 2.

Table 1 Factors with coded and natural levels for the DSD

Factors and units	Code	Levels		
		− 1	0	+ 1
Total thickness (nm)	TT	100	150	200
Ni deposition rate (nm/s)	Ndr	0.02	0.06	0.10
Cu deposition rate (nm/s)	Cdr	0.02	0.06	0.10
Ni layer thickness (nm)	Nlt	1	5	9
Rotation of the substrate (rpm)	RS	0	20	40

2.2 Characterisations

The magnetic properties of the multilayer films were identified using a vibrating sample magnetometer (VSM, Ade Technologies DMS-EV9). Hysteresis loops were obtained at room temperature by applying the films to a magnetic field ranging from − 20 to + 20 kOe at 1 Oe intervals. The accuracy of the magnetisation is 1×10^{-6} emu. Data received from the hysteresis loops were used to obtain the M_s and H_c values. X-ray diffraction (XRD, PANanalytics X'Pert PRO) technique was used to investigate the crystal structure of the samples. In the technique, Cu-Kα radiation was applied and the scan was performed from 30° to 90°. Surface morphology of the films was examined by scanning electron microscope (SEM, FEI Quanta 200 F). Elemental analysis of the films was also done by energy dispersive X-ray spectroscopy (EDX) which is integrated in SEM.

2.3 Design of experiments

In this study, the RSM based on DSD was applied to investigate the influence of the production parameters on the M_s and H_c and to optimise the properties of Ni/Cu multilayers using trial version of Minitab 18. The levels of factors evaluated under study are listed in Table 1. Each level was represented at three levels, namely, "low", "medium" and "high". A five-factor [total thickness (TT), Ni deposition rate (Ndr), Cu deposition rate (Cdr), Ni layer thickness (Nlt) and rotation of the substrate (RS)] and 13-run DSD was tested. Means, interactions and optimisation of the synthesis parameters cannot be specified using DSD. For that, the RSM was employed to identify them. The RSM method is a mathematical formation method which is dependent on the relation between the factors and the response [11–13]. The model is based on the examination of the RSM which is obtained from the design matrix results. These are created depending on the above defined factors. And, the significance of each factor on the response was within the 95% confidence interval.

2.4 Quadratic regression

In this study, DSD experimental design is based on a mathematical model that was developed by fitting the experimental data with a quadratic regression model as expressed in Ref. [18]:

Table 2 DSD runs, factors and responses for Ni/Cu multilayer films

Std order	Run order	Pt type	Blocks	Parameters					Responses	
				TT (nm)	Ndr (nm/s)	Cdr (nm/s)	Nlt (nm)	RS (rpm)	M_s (emu/cm^3)	H_c (Oe)
1	1	2	1	0	1	1	1	1	464.3	69
2	2	2	1	0	− 1	− 1	− 1	− 1	37.8	49
3	3	2	1	1	0	1	− 1	− 1	43.1	39
4	4	2	1	− 1	0	− 1	1	1	357.2	57
5	5	2	1	1	1	0	1	− 1	316.3	68
6	6	2	1	− 1	− 1	0	− 1	1	60.4	43
7	7	2	1	1	− 1	1	0	1	241.9	52
8	8	2	1	− 1	1	− 1	0	− 1	286.5	48
9	9	2	1	1	− 1	− 1	1	0	467.4	45
10	10	2	1	− 1	1	1	− 1	0	67.0	51
11	**11**	**1**	**1**	**1**	**1**	**− 1**	**− 1**	**1**	**46.4**	**37***
12	**12**	**1**	**1**	**− 1**	**− 1**	**1**	**1**	**− 1**	**472.5***	**43**
13	13	0	1	0	0	0	0	0	246.2	44

*Bold shows the runs with maximum M_s and minimum H_c

$$Y = \beta_0 + \sum_i \beta_i X_i + \sum_i \beta_{ii} X_i^2 + \sum_{i=1} \sum_{j=i+1} \beta_{ij} X_i X_j + \varepsilon, \tag{1}$$

where Y is the response (M_s and H_c), β_0 is the model interception coefficient, β_i is the linear coefficient effect, β_{ii} is the quadratic coefficient effect, β_{ij} is the interaction coefficient effect, X_i and X_j are the factors, and ε is the random error.

3 Results and discussion

3.1 Definitive screening design

Table 2 presents 13-run experiment established by DSD and their responses. The design was applied for determination of the primary values of experimental parameters in the production process of Ni/Cu multilayer films. Five input factors with three level values were TT (100, 150 and 200 nm), Ndr (0.02, 0.06 and 0.10 nm/s), Cdr (0.02, 0.06 and 0.10 nm/s), Nlt (1, 5 and 9 nm) and RS (0, 20 and 40 rpm).

According to the DSD, the M_s and H_c values as responses were obtained from the hysteresis loops of the samples produced according to the recipes given in Tables 1 and 2. The maximum M_{sini} was obtained from run 12 (472.5 emu/cm^3) and the minimum H_{cini} was measured for run 11 (37 Oe) as depicted in bold with "*".

3.2 Magnetic and structural analysis of Ni/Cu films in DSD experiments

Figure 1 presents the hysteresis loops of the selected films among the films given in Table 2. Run 1 has the highest saturation magnetisation, M_s among the three films, whereas Run 11 has the lowest M_s in all films. The M_s of Run 7 is between these two. The M_s values are in consistence with the Ni content of the films as the M_s decreases with the decrease of total Ni thickness from Runs 1 to 11 (among three films). Coercivity, H_c of the films decreases from Runs 1 to 11. Furthermore, a shift was observed in the H_c values of Run 11. Previous studies on sputtered Ni/Cu multilayers show different magnetic properties according to the change of parameters [3, 16, 19]. M_s differentiated from 180 to 889 emu/g according to Cu layer thickness as in Ref. [19], whereas it changed from 48 to 71 emu/g with the change of deposition rate as in Ref. [16]. M_s values obtained in this study (see Table 1) are in a wide range, indicating that the optimisation scans a wide range of parameters. H_c values also show a wide range between 62 and 164 Oe in [19] and change between 48 and 71 Oe in [16]. In our study, magnetic analysis results of 13-run experiment show a narrow distribution of H_c values changing between 37 and 69 Oe. Structural characterisations of the same samples were done by XRD. The XRD patterns of the selected films and substrate are given in Fig. 2. There are no peaks in the pattern of the substrate. The patterns of the samples include (111), (200) and (220) peaks at around $2\theta \approx 43°$, 52° and 74°, respectively. The XRD analysis showed that Ni/Cu films were crystallised at the face-centred cubic (fcc) phase (according to the JCPDS cards no. 04-0836 and 88-2326) and the preferential orientation was (111). In the pattern of Run 1, a small shift to higher angles is observed since the Ni content of the film is higher than the others due to the high total Ni thickness of the film. And the peak around 43° shows separation into two peaks probably corresponding to Cu (smaller angle) and Ni (higher angle), implying the presence of starting an individual phase structure. In Refs. [3, 19], splitting of (111) peak was also observed in sputtered Ni/Cu multilayers as in the XRD pattern of Run 1. However, in Ref. [16] single phase was observed in the patterns of Ni/Cu films as Runs 7 and 11 in the present study. Crystallite sizes of the films were also estimated by using Scherrer formula [20]. In this paper, $K = 0.9$ was used. All peaks in the patterns were taken into account in the estimation. The crystallite sizes were estimated to be $\sim$ 6 nm, $\sim$ 8 nm and $\sim$ 15 nm for Runs 1, 7 and 11, respectively. Since Runs 7 and 11 has less total Ni thickness than Run 1, it is expected that Ni content of Run 1 to be higher than the others. In order to clarify the elemental compositions of the films, EDX analysis was performed. Elemental composition of Run 1 is found to be 74.5 at.% Ni and 25.5 at.% Cu, whereas Run 7 has 21.2 at.% Ni and 78.9 at.% Cu. Besides, Run 11 has the lowest Ni content with 1.9 at.% Ni and 98.1 at. % Cu. Thus, it can be concluded that the structural and magnetic properties of Run 1 was dominated by Ni content of the film rather than the crystallite size. SEM image of the film Run 7 with the EDX results are given in Fig. 3 as an example. As can be observed from the image, film surface is quite smooth with reasonable homogeneity.

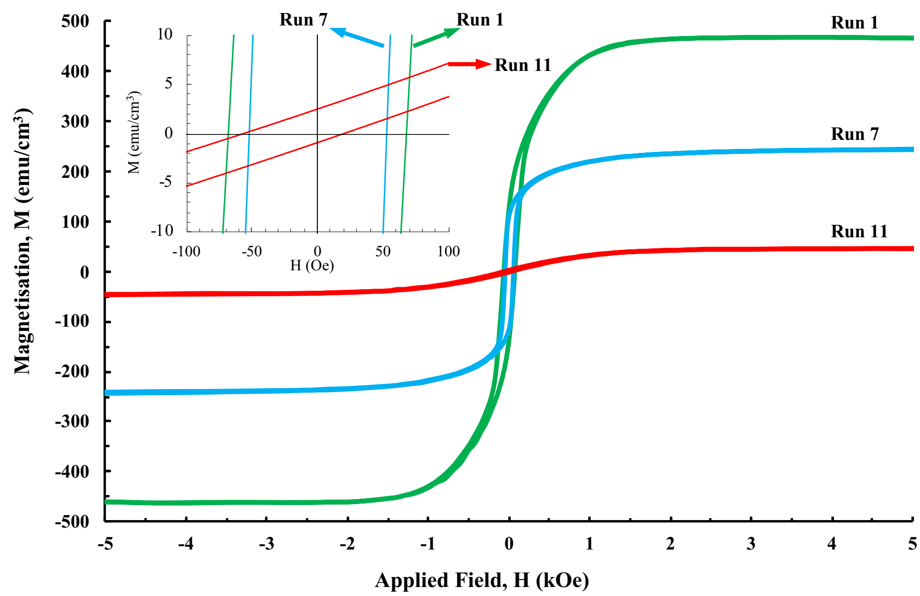

Fig. 1 Hysteresis loops of the selected Ni/Cu multilayer films according to the DSD. Inset shows the curves at ± 100 Oe (for interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article) (Color figure online)

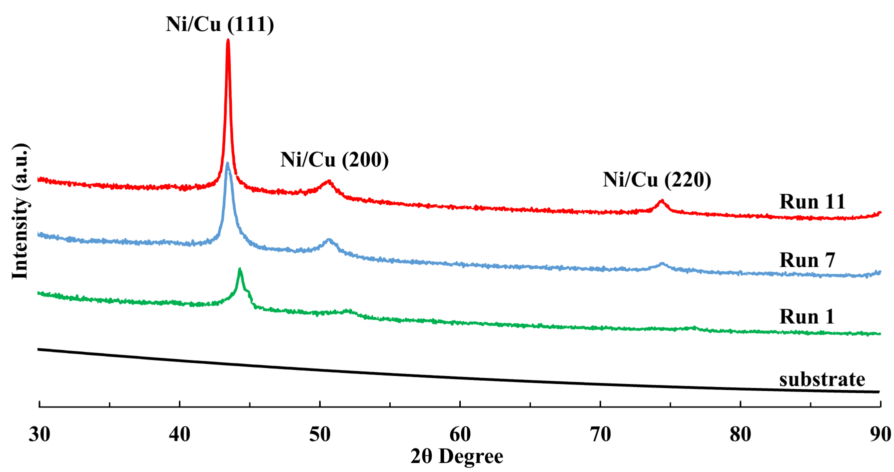

Fig. 2 XRD patterns of the selected Ni/Cu multilayer films and substrate (for interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article) (Color figure online)

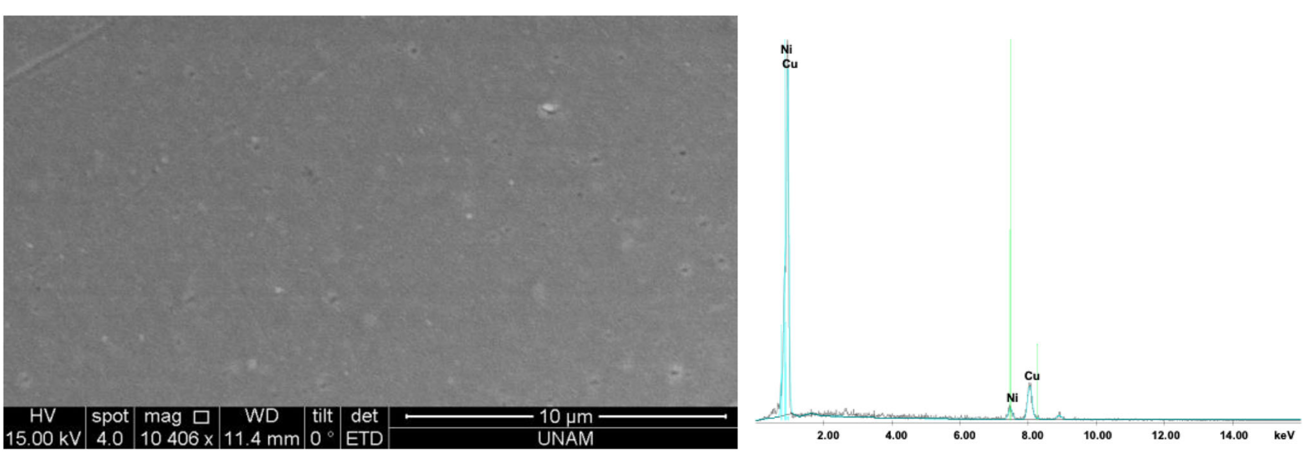

Fig. 3 SEM image and elemental analysis results of the selected Ni/Cu multilayer film (Run 7)

3.3 Response surface method

3.3.1 Main effects

The influences of factors were separated out in terms of the mean response characteristics since the experimental design was orthogonal. It is known from previous studies that deposition parameters have effect on the properties of Ni/Cu multilayers [3, 16, 19]. However, the most effective parameters in whole process can be presented through optimisation. Besides, interactions between the parameters can also be determined by optimisation (see Sect. 3.3.2). The maximum and minimum levels of the mean response of the M_s and H_c values at all factors and levels are given in Fig. 4a and b, respectively. The maximum and minimum mean value of each factors supply the best experimental finding for M_s and H_c, respectively. Figure 4a and b shows the plots of mean parameter effects of Ni/Cu multilayer films on the M_s and H_c, respectively. In the figures, the distance between the maximum and minimum dots measures the importance of the individual parameters, and the line is edge of relevance, which is correlated by the angle. From Fig. 4a, it can be observed that mean of M_s increases from around 50 emu/cm^3 to over 400 emu/cm^3 with the increase of Ni layer thickness (Nlt) from 1 to 9 nm. However, mean M_s values hardly change (around 240 emu/cm^3) with the change of other parameters. Nlt seems to be the most effective parameter since the minimum and maximum values strongly affect the M_s of the film. For the maximum M_s the Nlt is the most relevant parameter, and the rest of the parameters for M_s have almost the same magnitude of influence. By using similar approach most effective parameter for H_c can be obtained as Nlt (Fig. 4b). The highest order values of all parameters for the H_c from the highest to lowest order were Nlt (8.8 nm), Cdr (0.1 nm/s), Ndr (0.02 nm/s) and TT (100 nm). It is observed that the parameter Nlt was the most effective parameter for the maximum M_s and the minimum H_c, and the TT was the least effective parameter for both responses.

3.3.2 Interactions of synthesis parameters

In order to see the interactions between the synthesis parameters, the counter plots of the M_s and H_c values obtained from the experimental study against factors are shown in Fig. 5a and b, respectively. In Fig. 5a, depending on the differences of the Nlt, there was significant changes in the M_s values. It can be said that the M_s values significantly increased and decreased with Nlt. The degree of the effect exists but lower for the rest of the parameters for H_c values.

3.3.3 Optimum values of parameters and the prediction

The optimum process parameters for the maximum M_s and the minimum H_c of Ni/Cu multilayer films obtained from Fig. 6 as TT (100 nm), Ndr (0.02 nm/s), Cdr (0.1 nm/s), Nlt (8.8 nm) and RS (20 rpm). These factors were used to predict optimal Ni/Cu films which has M_{spred} = 523.4 emu/cm^3 and H_{cpred} = 31 Oe.

3.4 Confirmation experiment

For validation of the optimised results, the experimental process has been conducted using these optimal setting of the process parameters presented in Fig. 6, and the M_{sexp} and H_{cexp} values were obtained as 537.2 emu/cm^3 and 28 Oe. Here, the system optimisation for M_s and H_c was achieved using the RSM at the predicted 95% confidence interval. It is observed that the optimised experimental results are consistent and much higher than the DSD primary experimental results, which are M_{sini} = 472.5 emu/cm^3 and H_{cini} = 37 Oe obtained from Table 1.

3.5 Regression analysis

In the modelling and analysing of factors, there is a relationship between a dependent variable and one or more independent variables [21]. Under study, the dependent variables are saturation magnetisation (M_s) and coercivity (H_c), whereas the independent variables are total thickness (TT), Ni deposition rate (Ndr), Cu deposition rate (Cdr), Ni layer thickness (Nlt) and rotation of the substrate (RS). In obtaining predictive equations for the M_s and H_c, regression analysis was used. These predictive equations were made for quadratic regression models. The predictive equations which were obtained by the regression model of M_s and H_c are given below:

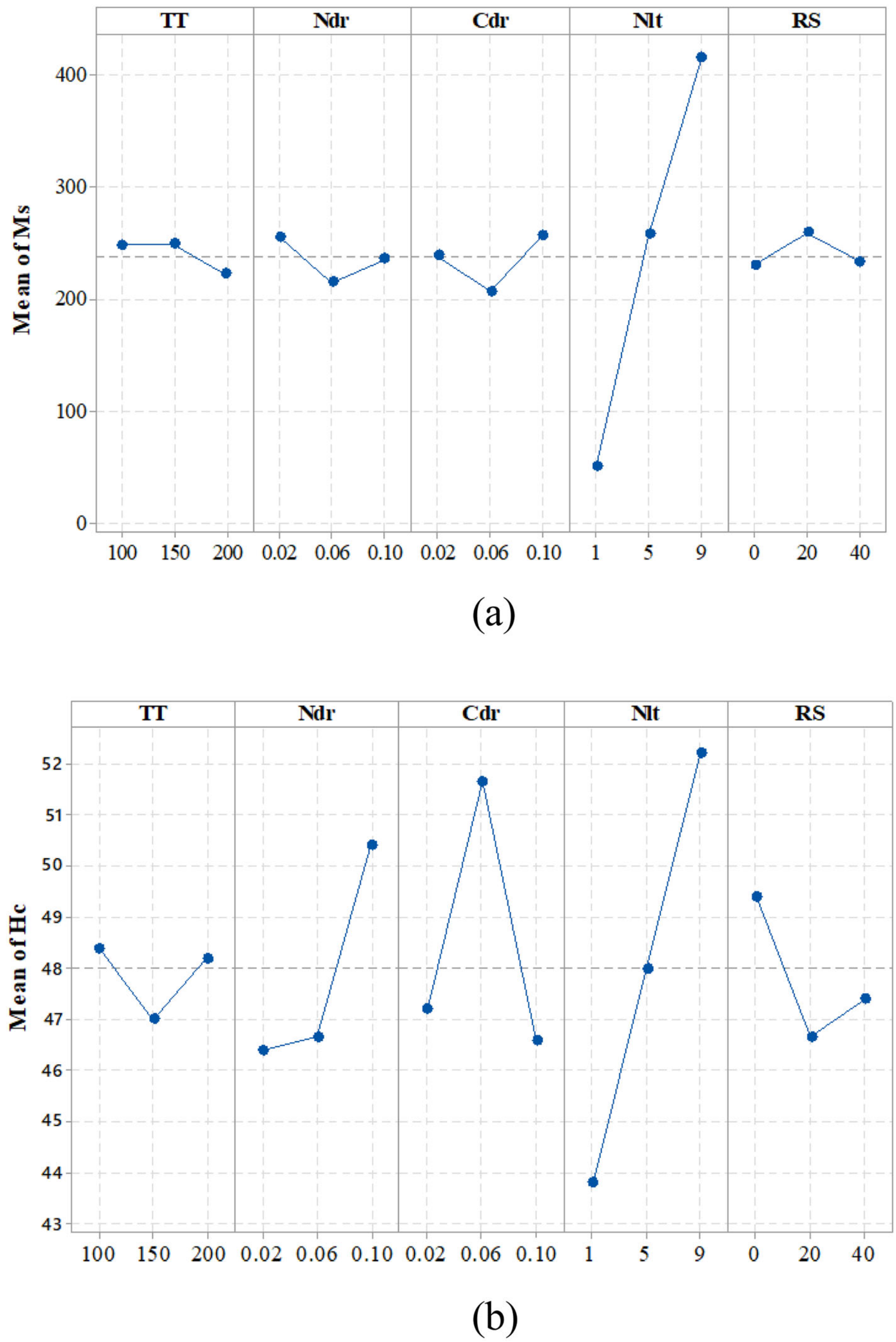

Fig. 4 Main effects of synthesis factors over Ni/Cu multilayer films characteristics **a** M_s and **b** H_c

$$M_s\ (\mathrm{emu/cm^3}) = 142 + 0.77\mathrm{TT} - 4696\mathrm{Ndr} - 5099\mathrm{Cdr} + 76.8\mathrm{Nlt} + 5.37\mathrm{RS} - 0.0057\mathrm{TT} * \mathrm{TT} + 22{,}931\mathrm{Ndr} * \mathrm{Ndr} + 44{,}438\mathrm{Cdr} * \mathrm{Cdr} - 3.12\mathrm{Nlt} * \mathrm{Nlt} - 0.132\mathrm{RS} * \mathrm{RS} + 11.3\mathrm{TT} * \mathrm{Ndr} \quad (R^2 = 97.5\%), \tag{2}$$

$$H_c\ (\mathrm{Oe}) = 6 + 0.19\mathrm{TT} + 650\mathrm{Ndr} + 1155\mathrm{Cdr} - 4.20\mathrm{Nlt} - 1.09\mathrm{RS} + 0.00028\mathrm{TT} * \mathrm{TT} + 750\mathrm{Ndr} * \mathrm{Ndr} - 9688\mathrm{Cdr} * \mathrm{Cdr} + 0.525\mathrm{Nlt} * \mathrm{Nlt} + 0.0260\mathrm{RS} * \mathrm{RS} - 4.60\mathrm{TT} * \mathrm{Nd}r \quad (R^2 = 89.8\%). \tag{3}$$

The quadratic regression model provided a very good statistical performance with high correlation

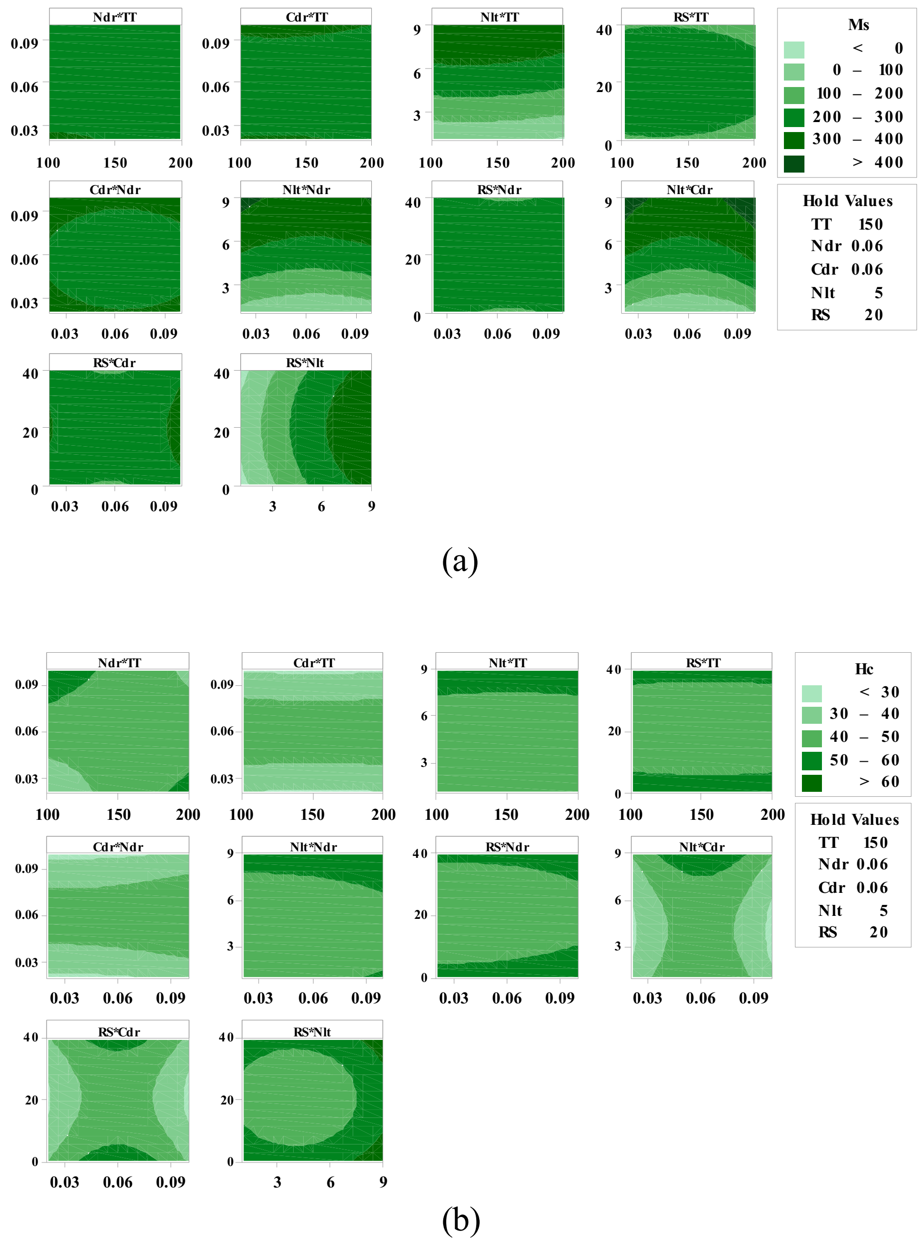

Fig. 5 Response counter plots showing the effects of two variables on **a** M_s and **b** H_c values

coefficients of 97.5% and 89.8% between the actual and predicted values of M_s and H_c, respectively. Thus, quadratic regression equations can be successfully applied to obtain the predicted values of M_s and H_c. The regression model was shown to be successfully estimate the M_s value (522.6 emu/cm^3) using optimal conditions of TT (100 nm), Ndr (0.02 nm/s), Cdr (0.1 nm/s), Nlt (8.8 nm) and RS (20 rpm). And, the model also displayed a successful estimation of H_c value (31 Oe) using the same optimal conditions. In Fig. 7a, b, the experimental results and the predicted values which were obtained by the quadratic regression model are given. It is observed that the results are consistent with each other.

3.6 Comparison of test results

Table 3 displays the error percentages between experimental results, predicted values and regression equation at optimum levels of the M_s and H_c of Ni/Cu films. The experimental and predicted values at optimum experimental conditions are very close to each other. For reliable statistical analyses, error values must be less than 20% [22]. For RSM of M_s, the error percentage of optimised sample was the

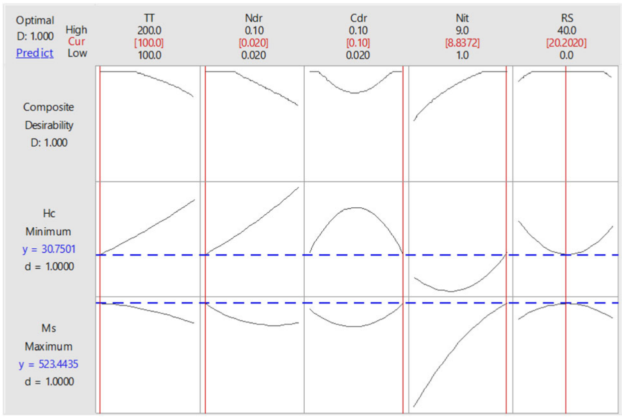

Fig. 6 The optimum process parameters for the maximum M_s and the minimum H_c of Ni/Cu multilayer films

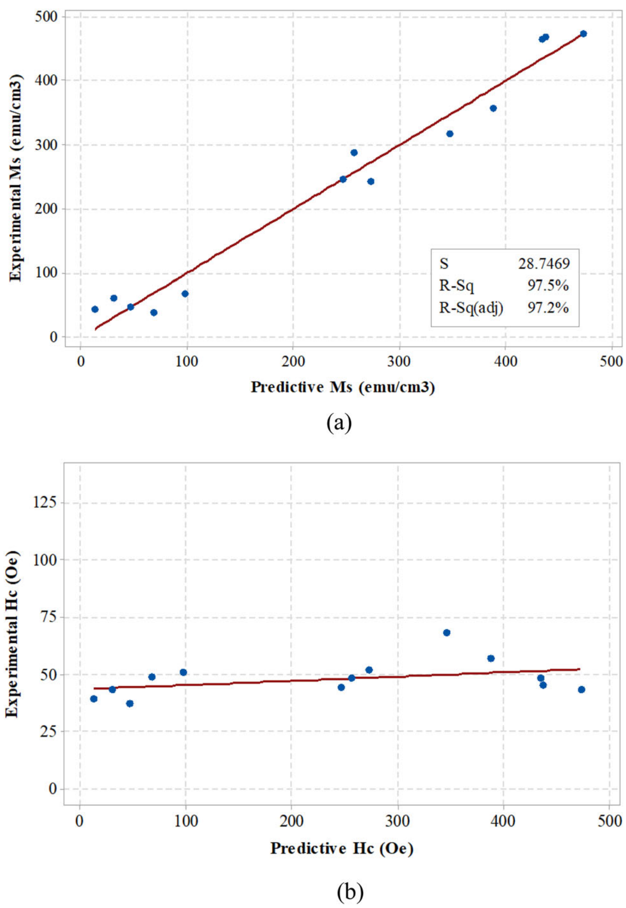

Fig. 7 Comparison of the quadratic regression model with experimental results for **a** M_s and **b** H_c

smallest one (2.6%) and this is followed for quadratic regression with the error percentage of 2.7%. For H_c, the error percentage of optimised and quadratic regression of the sample is 9.7%. The error percentages of M_s and H_c are within acceptable limits. Therefore, the results obtained from the confirmation tests reflect successful optimization. It is observed that optimised samples can be much more successfully applied to obtain the predicted and experimental values of maximum M_s and minimum H_c.

4 Conclusions

Under study, the relationships between production parameters and the maximum M_s and minimum H_c values of Ni/Cu multilayer films produced by DC sputtering method using 13 conditions planned with DSD design of experiment were investigated. Consequently, RSM was adopted for further analyses with respect to characterise the materials to a desired range of magnetic properties. And the optimum parameters were observed as TT (100 nm), Ndr (0.02 nm/s), Cdr (0.1 nm/s), Nlt (8.8 nm) and RS (20 rpm) to obtain the Ni/Cu films with the maximum M_s values and minimum H_c. With RSM, the main effects data is an efficient step in the initial stages of a process to evaluate most important effects in final characteristics of Ni/Cu films. From mean response characteristics, the most effective factor is Nlt for both cases. And, the most interactive factor is also Nlt according to the counter plots. The M_s and H_c of Ni/Cu films increased from initial measurement (472.5 emu/cm^3 and 37 Oe) to optimised (537.2 emu/cm^3 and 28 Oe) values, respectively. On the other hand, quadratic regressions were applied to predict the outcomes of the experiment. Developed regression model demonstrated a very good relationship for M_s (522.6 emu/cm^3) and H_c (31 Oe) and the best suited quadratic polynomial models for M_s and H_c with high coefficient of determination (R^2 = 97.5% and 89.8%) values were obtained, respectively. According to the confirmation test results, measured values were within the 95% confidence interval. The films displayed fcc structure and were preferentially oriented at the (111) direction. With design of experiments methods applied to production factors of sputtering technique, it is possible to produce Ni/Cu multilayer films with maximum M_s and minimum H_c responses and to choose future production factors with more reliability.

Table 3 Error percentage of the experiments and the predicted results according to design of experiments and quadratic regression analysis

Factor combinations	Responses	RSM			Quadratic regression	
		Experimental	Prediction	Error (%)	Prediction	Error (%)
Optimised: TT (100 nm), Ndr (0.02 nm/s), Cdr (0.1 nm/s), Nlt (8.8 nm) and RS (20 rpm)	M_s (emu/cm^3)	**537.2**	523.4	2.6	522.6	2.7
	H_c (Oe)	**28**	31	9.7	31	9.7

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