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Research Article

Analysis of Effect of Deposition Parameters on Thickness of Co-sputtered Titanium/Alumina Thin Film Using ANOVA

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Abstract

The aim of this article is to study the influence of deposition parameter on thickness of Titanium (Ti) - Alumina (Al_2O_3) thin film co-sputtered using magnetron sputtering technique on Stainless Steel grade 304(SS304) and Copper (Cu) substrates. Nanostructured thin film have been sputtered simultaneously from two targets (Ti and Al_2O_3) by Direct Current (DC) magnetron sputtering and Radio Frequency (RF) magnetron sputtering respectively without substrate heating. Though most engineering cutting tools are primarily manufactured from bulk material, thin film is often required for hardness, its adhesiveness and surface integrity control. This paper presents the result of an analysis of experimental investigation on thickness. The experiments have been carried out based on rotatable Central Composite Design (CCD) of experiments approach. The influence of deposition parameters viz. DC power, RF Power and inert gas flow rate on thickness has been analyzed statistically by ANOVA. It is established that DC power and RF power have been linearly significant. Contour plots of thickness have been developed for DC power and RF power.

Keywords: Titanium (Ti) & Alumina (Al_2O_3), CCD, Co-Sputtering, Magnetron Sputtering, Analysis of Variance

Introduction

Titanium finds over 96% of the world-wide use of titanium for potential application, thus creating a high demand for this material and it exhibits excellent corrosion resistance and good mechanical properties. Many different synthesis methods have been used to prepare thin films on the desired substrate. Among all methods, sputtering method has been extensively used to deposit thin films on conductive substrates. Sputtering involves physical ejection of atoms or molecules from a pure target and their condensation onto a substrate. Investigations have revealed that the formation of films with properties depends upon deposition conditions, such as substrate temperature, deposition rate, inert gas pressure, and power and upon the material itself [1]. It is necessary to effectively determine the impact of these deposition parameters. By the role refinement of each parameter and their potential correlation, it will be possible to control thin film characteristics. The CCD is the method based on the experimental design is certainly a way to be discovered allowing optimization of Ti- Al_2O_3 deposition by means of Physical Vapor Deposition (PVD) processes; predominantly magnetrons sputtering that are used for a thin films development onto the appropriate substrate. Further, ANOVA test has been used for analysis of the experimental results to find the significance of variables. ANOVA method is tool to explore the implications of observations or analysis of variety of experimental designs based on F-test with 95% of level of significance or the probability (p-value) less than significance level i.e. is 0.05 which justifies the significance. [2-4].

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Experiments

Ti- Al_2O_3 thin films have been so-sputtered or co-deposited by magnetron sputtering onto SS304, and Cu substrate. The substrates has been prepared to size of 10mmX10mm and polished for good surface finish, later substrates were cleaned by rinsing in ultrasonic baths of acetone, ethanol, and deionized water for 20 min to remove any contaminations. Substrate has been placed in the vaccum chamber and targets have fixed into the two magnetron heads, one head is DC powered and the other is RF powered. The co-sputtering has been carried out in presence of an Argon atmosphere. DC sputtering power is ideal for Ti deposition and RF sputtering power for Al_2O_3. All experiments have been conducted based on CCD .The details of parameters and its levels has been presented in the table 1 and table 2 indicates the experimentally deposition condition matrix based on CCD and measured thickness in nm.

Table 1.Deposition Parameters and their levels

Levels Parameters	Designation	–1.68 (extreme negative value)	–1 (low level)	0 (zero level)	1 (high level)	1.68 (extreme positive value)
DC power in watts	DC	500	601.2	750	898.8	1000
RF power in watts	RF	400	638	800	1038	1200
Gas Flow rate in Standard Cubic Centimeter	SCCM	150	239.3	300	389.3	450

Table 2.Experimental matrix based on CCD and measured Thickness of thin films

S. No.	Test Combinations	DC	RF	SCCM	Response (Thickness in nm)
1	1	-1	-1	-1	155
2	a	+1	-1	-1	175
3	b	-1	+1	-1	230
4	ab	+1	+1	-1	200
5	c	-1	-1	+1	169
6	ac	+1	-1	+1	189
7	bc	-1	+1	+1	185
8	abc	+1	+1	+1	205
9	-αa	-1.68	0	0	145
10	+αa	+1.68	0	0	225
11	-αb	0	-1.68	0	152
12	+αb	0	+1.68	0	200
13	-αc	0	0	-1.68	145.5
14	+αc	0	0	+1.68	207
15-20	At Zero level	0	0	0	172

Analysis of Experimental Results

The influence of various process parameters such as DC power, RF power and Argon gas pressure on thickness of the Ti-Al_2O_3 thin films needs to be understood in order to use it for better performance in actual applications. ANOVA provides a strong statistical analysis; Analysis of variances uses F tests with confidence limit 95%, in which the p value defines the significance of variable. The experimental results have been analyzed by ANOVA test using software package MINITAB 18.If p value for variable is 0.05 and lesser than that indicates respective variable is significant in contribution towards thickness. Using ANOVA, linear, square and two interactions of variables have been analyzed and the same has been validated using standardized residual plots. The ANOVA test using regression model has carried out and results have been tabulated in the table 3.Accordingly,linear model of variables DC and RF power used to sputter Ti and Al_2O_3 are significant in contributing to thickness since "p" value of both the variables (DC &RF)are 0.024 and 0.006 respectively are lesser than 0.05. In addition “p” value of DC variable is more significant. Also, ANOVA results shows the unusual observations on results of three test combinations viz. serial no. 3,6 and 9 because if its large residuals. These unusual observations have presented in the table 4.

Table 3.Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	9	8291.0	921.22	3.25	0.040
Linear	3	6447.7	2149.22	7.57	0.006
DC	**1**	**2011.5**	**2011.53**	**7.09**	**0.024**
RF	**1**	**3521.9**	**3521.86**	**12.41**	**0.006**
SCCM	1	980.3	980.28	3.45	0.093
Square	3	473.6	157.87	0.56	0.656
DC*DC	1	446.9	446.90	1.57	0.238
RF*RF	1	76.8	76.77	0.27	0.614
SCCM*SCCM	1	15.8	15.78	0.06	0.818
2-Way Interaction	3	1398.9	466.30	1.64	0.241
DC*RF	1	491.5	491.49	1.73	0.218
DC*SCCM	1	180.5	180.53	0.64	0.444
RF*SCCM	1	735.5	735.51	2.59	0.138
Error	10	2837.8	283.78		
Lack-of-Fit	5	2837.8	567.55	*	*
Pure Error	5	0.0	0.00		
Total	19	11128.7			
S=16.8457 R-sq=74.50% R-sq(adj)=51.55%					

Table 4.Fits and Diagnostics for Unusual Observations

Observations	Thickness(Ti)	Fit	Resid	Std Resid	
3	230.0	203.1	26.9	2.66	R
6	189.0	211.8	-22.8	-2.25	R
9	145.0	168.4	-23.4	-2.18	R
R Large residual					

Table 5.Coded regression co-efficient

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	173.20	6.79	25.52	0.000	
DC	***20.81***	***7.82***	***2.66***	***0.024***	1.04
RF	***30.21***	***8.58***	***3.52***	***0.006***	1.05
SCCM	15.94	8.58	1.86	0.093	1.05
DC*DC	16.0	12.8	1.25	0.238	1.06
RF*RF	6.9	13.2	0.52	0.614	1.09
SCCM*SCCM	3.1	13.2	0.24	0.818	1.09
DC*RF	-26.1	19.9	-1.32	0.218	1.02
DC*SCCM	15.8	19.9	0.80	0.444	1.02
RF*SCCM	-37.7	23.4	-1.61	0.138	1.04

Regression Equation in Uncoded Units

$T_{Ti\ \&Al2O3}$ = 10 - 0.219 DC + 0.391 RF + 0.209 SCCM + 0.000257 DC*DC + 0.000043 RF*RF + 0.000138 SCCM*SCCM - 0.000261 DC*RF + 0.000423 DC*SCCM - 0.000628 RF*SCCM------- (1)

color square shaped dot lie on the right side of normal line it indicates positive influence of the variable[ ]. Accordingly both DC and RF have positive effect on the thickness. Figure 1(b) indicates the residuals which are distributed around the line are normal. Figure 2 is a scattered plot of residuals on the Y axis and estimated values or fitted value on the

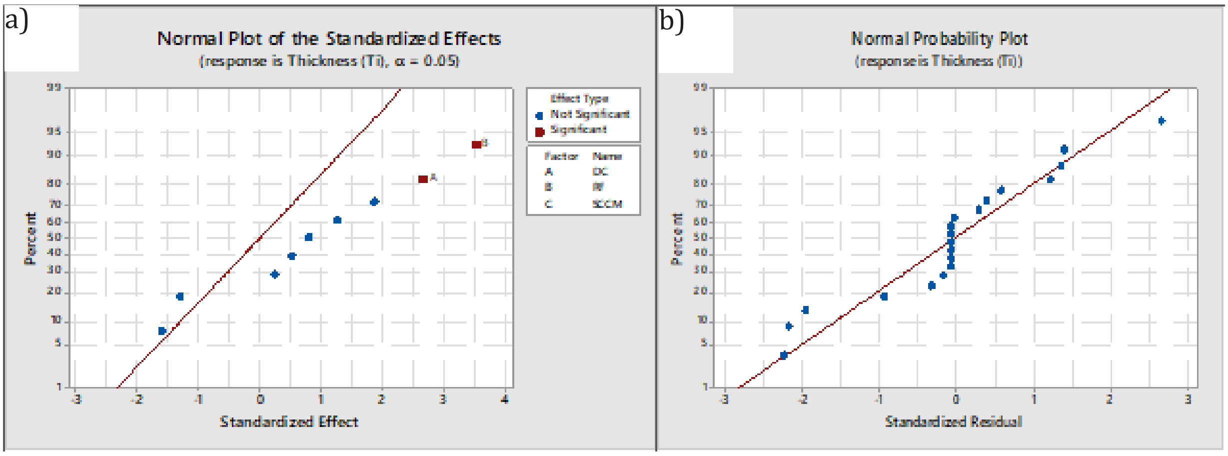

Figure 1, a) Normal plots of standardized effects of factors on Thickness Figure (b) Probability plot of distribution of Residual

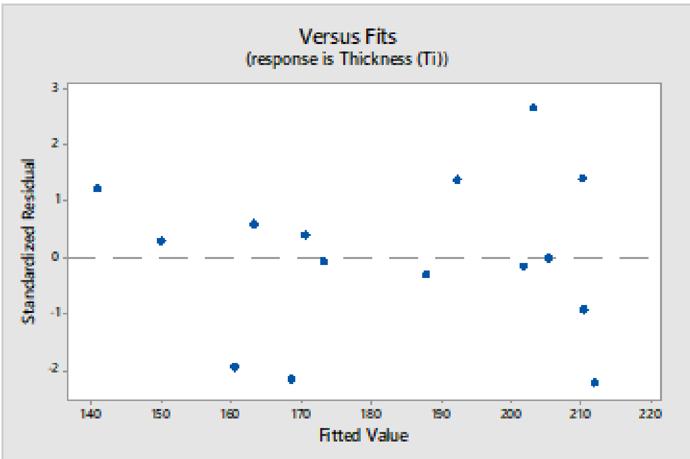

Figure 2.Plot of Residual versus Fits

Table 4 depicts the results of regression analysis, which is used to find the significant relationship in the form linear, square and two way interaction using T test value. "p" value of respective variable indicates is significance. the tabulated results confirms the that the variables DC and RF power used to deposit on to different substrates have been significant in contributing to thickness of Ti &Al_2O_3 since p value is lesser than level of significance[2]. Regression Equation 1 is a mathematical model presented in an un-coded form to predict thickness of Ti&Al_2O_3 thin film. Prediction can be done based on the deposition variables value such as the value of DC power, RF Power and Gas flow rate in SCCM. Figure 1(a) represents standardized effects of parameters on thickness which validates the ANOVA results that the significant variables are DC power and RF power. Since over the normal line or fit line, the distribution of red color square shaped dots indicates is significance. If red

x axis. Distribution doesn't show any particular pattern, it is randomly scattered around the estimated regression line that means the relationship is reasonable. There are two points which lie on estimated line has "0" residual.

Conclusion

In summary, Ti-Al2O3 thin film has been successfully co-sputtered on SS304 and Cu substrate using magnetron sputtering technique based on CCD and measured thickness has been analyzed by ANOVA method based on F-test at 95% level of significance were studied.

Analysis on thickness of thin film has been done using ANOVA; the factors which influence the response during deposition time were identified. The percentage of contribution of two factors DC and RF both has positive influence comparatively DC has more.

There is two ways interaction has been found which contributes significantly neither has quadratic effect on thickness and regression model of equation is very much needed to predict the values based on the value of variable.

References

1. Motahareh Einollahzadeh-Samadi and Reza Sabet Dariani , "Characterization of nanocrystalline Ti films deposited by DC magnetron sputtering onto FTO glass substrate" *Journal of Materials Research Society* , Vol. 30, Oct 28, 2015, pp 3093 -3103.
2. Douglas C Montgomery "Design and Analysis of Experiments", fourth edition: Wiley India (P) Ltd.
3. Kauko Leiviska "Introduction to Experiment Design" University of Oulu, Control Engineering Laboratory, 2013.
4. Thomas B Barker "Quality by experimental design". New York :Marcel Deckker Inc.
5. Kuehl Robert O. "Design of experiments" USA: Duxbury; 2000.
6. MasahikoNaoe,Satoshi Ono, Toyoaki Hirata "Crystal orientation in titanium thin films deposited by the sputtering method without plasma damage" *Material Science and Engineering*: A, Volume 134, 25 March 1991, Pages 1292-1295.
7. Nishat Arshi, Junqing Lu, Chan Gyu Lee, Jae Hong Yoon, Bon Heun Koo And Faheem Ahmed "Thickness effect on properties of titanium film deposited by d.c.magnetron sputtering and electron beam evaporation techniques" Bull. Mater. Sci., Vol. 36, No. 5, October 2013, pp. 807–812.

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