Comparative Study of Surface Roughness of Tungsten and Alumina co-sputtered on Stainless Steel and Copper substrates

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Abstract

A comparative study **of** the effect of co-sputtering specifically magnetron sputtering process input factors on surface roughness of W and Al_2O_3 thin film developed on the SS304 and Copper(Cu) substrates in the argon atmosphere. The input parameters have been varied based on the Design of Experiments specifically Center Composite Design experimental plan. Atomic Force microscope has been used to determine the average surface roughness in nanometer of all the samples as per experimental combinations. By using ANOVA method, the effect of factors on roughness and regression model have been developed, which express degree to input parameter can effect on the surface roughness. Analysis of surface roughness of W and Al_2O_3 thin film developed above said different substrates have been presented.

Introduction

Surface finish is the most important output responses, the quality or state of having an uneven or irregular surface is a component of surface texture. It is quantified by the deviations from the mean line. The average roughness (Ra) and Root mean square (Rq) are the most commonly used parameter in surface finish measurement, which usually helps to determine the quality of your deposition technique. Reduction in roughness implies surface diffusion, which plays active role in growth process and surface roughness is also attributed to the change of sputtering conditions [1]. For deposition, Magnetron sputtering machine has been used to develop Tungsten (W) /Alumina (Al_2O_3) on different substrates. The development of W /Al_2O_3 coatings produced by magnetron co-sputtering with the same deposition conditions on SS304 and Copper substrates at room temperature[2]. Direct current (DC) technique is used to sputter W and Radio Frequency (RF) Technique is used to Al_2O_3. DC and RF are in Watts and the gas flow rate as medium in Standard Cubic centimeter (SCCM), being the input parameters (factors) for the sputtering conditions. Even small changes in any of the mentioned factors may have a significant effect on the produced surface. Number of levels of each parameter with different configurations and number of experimental runs have been determined by Design of experiments approach [3]. Response Surface Methodology (RSM) is a technique that has been adopted to develop, improved and optimized process by using a statistical data and mathematical techniques. Statistical analysis in research activities helps in improving our knowledge on how to control the process parameter and quality of the thin film. In this article, the effect of process input parameters on surface roughness of the thin film developed on the SS304 and Cu have been effectively studied using ANOVA. Gangshi Hu et al have focused on control of thickness and surface roughness through deposition process and its model via kinetic Monte Carlo Simulation and also referred the study of linear (Ni and Christofides, 2005) and nonlinear (and nonlinear (Lou et al., 2008))Stochastic differential equations (SDEs) on surface roughness [4].

Experimental Details

The W/Al2O3 thin films were deposited onto SS304 and Copper substrates by using magnetron sputtering system, after polishing and successive cleaning of the substrates with acetone and ethanol then dried. Two targets of W and Al_2O_3 with purities of 99.99% and 99.95%, respectively, were employed. After loading substrates and targets, the process chamber was evacuated to a base pressure of 4×10^{-5}.Co-deposition carried out in presence of inert gas (Argon) at room temperature for about 20minutes. This process has been repeated after each experiment. The numbers of experimental runs or test combinations have been determined to 20runs. Below table 1 provides complete details of different levels of input parameters varied. Experimental Configuration of levels of Parameters has been presented in earlier paper [7] and each experiment has been carried out. The best known parameter in characterizing the surface roughness of a surface is average roughness and the root mean square (rms) roughness. The surface roughness measurement carried out using Atomic Force Microscope (AFM) of all the samples of a developed thin film. An area of size 10mm X10mm has been selected for measurement of roughness of the coatings. Results are tabulated in the in table 2.

Table 1
Sputtering Parameter and Levels

Input Parameters	Units	Designated Code	5 Levels				
			-α 1.68	-1	0	+1	+α 1.68
DC power	Watt	a	500	601.2	750	898.8	1000
RF Power	Watt	b	400	638	800	1038	1200
Gas Flow rate	SCCM	c	150	239.3	300	389.3	450

Table 2
Experimental results

Sample nos	SS 304 Substrate		Copper Substrate	
	R_a	R_q	R_a	R_q
1	4.468	5.549	11.320	16.332
2	10.871	12.76	15.947	22.36
3	5.850	7.36	18.169	23.51
4	10.21	12.40	27.746	35.61
5	6.823	8.73	15.927	20.24
6	9.303	11.37	14.32	18.24
7	8.028	10.36	22.831	27.07
8	6.901	8.46	6.119	7.68
9	7.766	7.766	10.722	15.480
10	5.406	7.22	41.368	49.869
11	5.557	6.78	11.275	14.061
12	4.082	5.316	13.38	16.718
13	8.633	10.55	10.648	14.264
14	67.763	81.66	31.403	41.843
15-20	9.725	12.03	10.843	13.567

Analysis Of Results And Discussions

The results obtained were analyzed by Analysis of Variance (ANOVA) using newest software MINITAB 18.To find the influence or effect of each process parameters (Factors) on the thin film, Results are analyzed through statistical analysis using response surface regression. To determine whether a factor has a significant effect or not, F test of significance has been used. Here, since the experiments are conducted at two levels factorial, there is only one degree of freedom for each effect and at zero level five trials were conducted in order to determine the experimental error, for which the degree of freedom is 5. Accordingly F critical value is 5.05 with a confidence limit of 95% or P-value of 0.05. If any factor's linear or quadratic or two way interactions are significant, respective F value should be equal to or more

than F critical. In other words P-value should be equal to or less than 0.05. Table 3 depicts the details of results of analysis of factors and its significance on roughness of thin film developed on SS304 and copper, which indicates adjusted sum of squares, F values and P values of factor's linear or quadratic or two way interactions terms. The table 4 shows the percentage of the each term that contributes for the surface roughness; it is computed by dividing adjusted SS value by total adjusted SS. The significance of a factor can be confirmed by the main effects plot and normal probability plot. For nonlinear effect on the response, the quadratic terms of the factors are included in the analysis to incorporate the results of the composite part of CCD.

Table 3
ANOVA results of Surface roughness of W/Al2O3 thin film developed on SS304 and copper

Analysis of Variance										
	Roughness on SS304					Roughness on Copper				
Source	**DF**	**Adj SS**	**Adj MS**	**F-Value**	**P-Value**	**DF**	**Adj SS**	**Adj MS**	**F-Value**	**P-Value**
DC	1	7.64	7.638	0.05	0.821	1	252.59	252.593	5.02	**0.049**
RF	1	0.00	0.000	0.00	0.999	1	47.30	47.298	0.94	0.355
SCCM	1	585.89	585.889	4.13	0.070	1	49.58	49.583	0.99	0.344
DC*DC	1	124.37	124.368	0.88	0.371	1	221.60	221.602	4.41	0.062
RF*RF	1	116.33	116.327	0.82	0.387	1	2.84	2.845	0.06	0.817
SCCM*SCCM	1	754.35	754.352	5.31	0.044	1	51.48	51.481	1.02	0.335
DC*RF	1	2.11	2.109	0.01	0.905	1	15.99	15.992	0.32	0.585
DC*SCCM	1	7.79	7.786	0.05	0.820	1	143.27	143.269	2.85	0.122
RF*SCCM	1	23.27	23.269	0.16	0.694	1	45.91	45.905	0.91	0.362
Lack-of-Fit	5	1417.76	283.551	781.6	0.000	5	495.08	99.017	65.13	0.000
Pure Error	5	1.81	0.363			5	7.60	1.520		
Total	19	3475.17				19	1277.62			

Table 4
Percentage contribution of significant of linear or quadratic or two way interactions of main factors

Substrate	**Factors**	**DC**	**RF**	**SCCM**	**DC*DC**	**RF*RF**	**SCCM*SCCM**	**DC*RF**	**DC* SCCM**	**RF* SCCM**
SS304	Percentage %	0.22	-	16.86	3.58	3.35	21.71	0.06	0.22	0.67
Cu	Percentage %	19.77	3.7	3.88	17.34	0.22	4.03	1.25	11.21	3.59

The factors and the interactions which are found to be statistically significant can be identified from the above Tables 3 and 4. Factors A (DC power in watts), B (RF power in watts) and C (Gas flow rate in SCCM) are the deposition

parameters, Thin film developed on SS304, only one Quadratic term of argon gas flow rate (SCCM*SCCM) is marginally significant for surface roughness as its P-value is less than and very closed to 0.05 wiz 0.044. At same time, the quadratic term of DC power (DC*DC) is more than 0.05 but also near to it. The percentage contribution of the SCCM*SCCM is 21.71and it is highest followed by linear term of SCCM. Similarly, on copper substrate, linear term of DC found to be statistically significant and its P value is 0.049 and the percentage contribution of the DC is 19.77 which is the highest followed by quadratic term of DC power (DC*DC).

Mathematical Models

Regression Equation in Uncoded Units for Surface roughness on SS304

$$Ra\_S = -99 + 0.248\,DC + 0.131\,RF - 0.336\,SCCM - 0.000135\,DC*DC - 0.000053\,RF*RF + 0.000956\,SCCM*SCCM - 0.000017\,DC*RF - 0.000088\,DC*SCCM - 0.000112\,RF*SCCM$$

Regression Equation in Uncoded Units for Surface roughness on copper

$$Ra\_C = -55 - 0.091\,DC + 0.1044\,RF + 0.282\,SCCM + 0.000181\,DC*DC - 0.000008\,RF*RF + 0.000250\,SCCM*SCCM - 0.000047\,DC*RF - 0.000376\,DC*SCCM - 0.000157\,RF*SCCM$$

The normal probability plot of the effects is a graphical technique to determine magnitude, direction and the importance of the effects. On the normal probability plot of the effects, effects that are further from 0 are statistically significant. The color and shape of the points differ between statistically significant and statistically insignificant effects. Fig 1 and fig 3 shows the normal probability plot with standardized effects of SS304 and Copper substrates. It can be observed from the plot that in fig 1, red color and square shape clearly indicates the quadratic term of C that is (SCCM*SCCM) is significant on SS304 substrate and in fig 3, linear term of A (DC) is significant on copper substrate. If red and square shaped point on the right side of normal line indicates impact is positive and its furthest distance indicates the level of particular factor. Fig 2 and fig 4 shows that the normal plots of residuals are normally distributed. A few points lying away from the line implies a distribution with outliers. If the residuals do not follow a

normal distribution, the confidence intervals and p-values can be inaccurate.

Conclusions

Experiments are conducted based on CCD to determine the surface roughness

a) The effect of factors and its significant have been analyzed using ANOVA. Results have confirmed by verifying with normal probability and Main effects plots.

b) The factor C, its quadratic (SCCM*SCCM) affects the surface roughness on SS304 substrate, it contributes about 16.86 % at higher level.

c) Similarly for Copper substrate , The linear factor of DC influences the roughness

d) None of the interactions were found to contribute significantly

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Figures

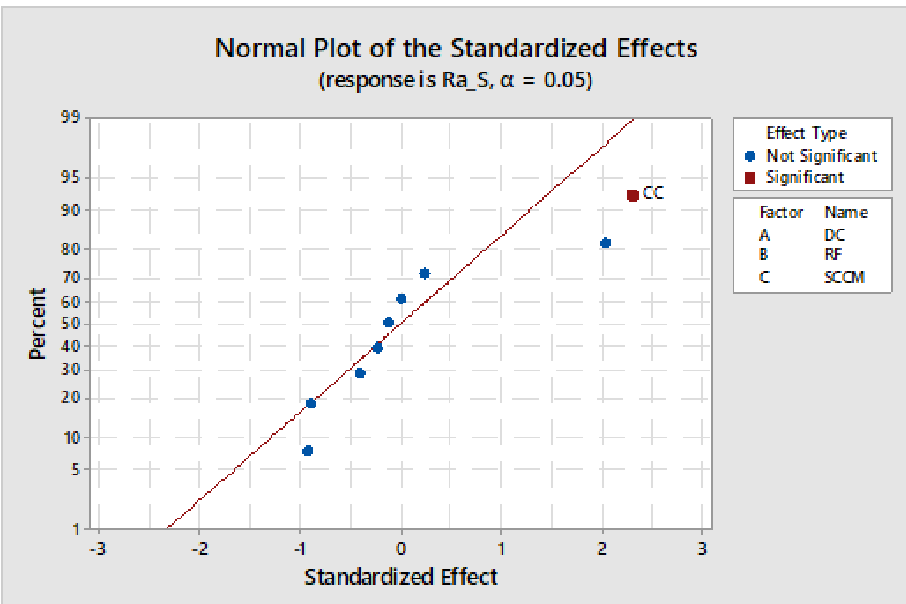

Figure 1

Normal probability plot of effects of factors- Ra S

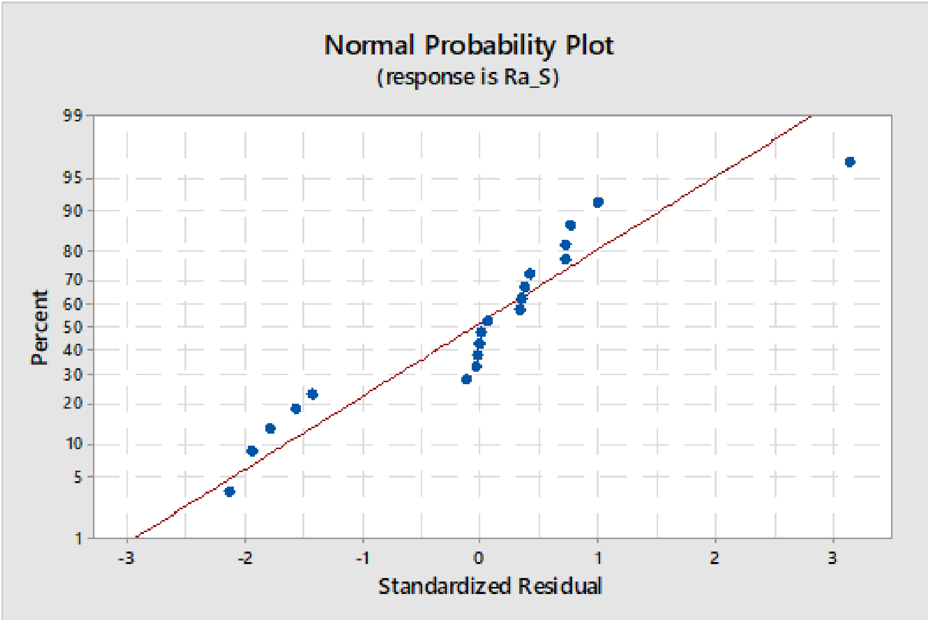

Figure 2

Normal Probability plot of residuals – Ra S

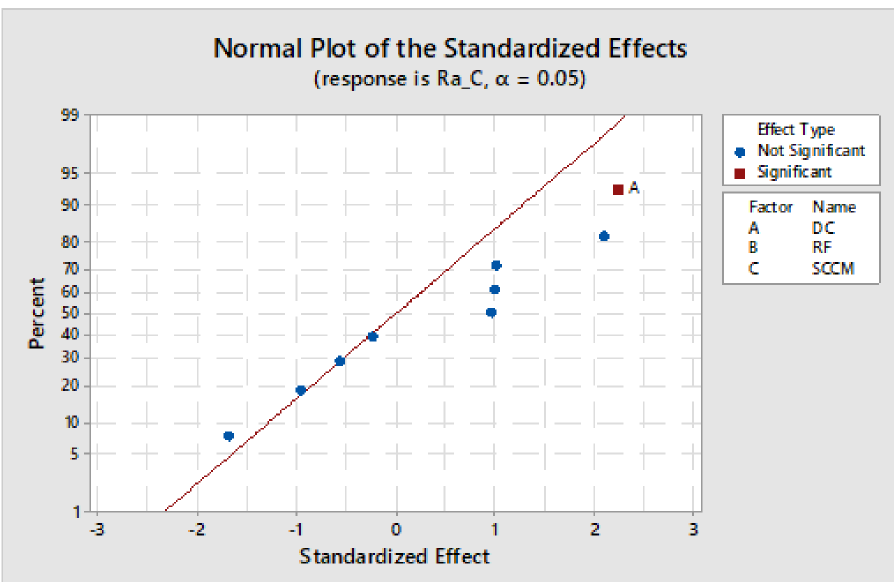

Figure 3

Normal probability plot of effects of factors RaC

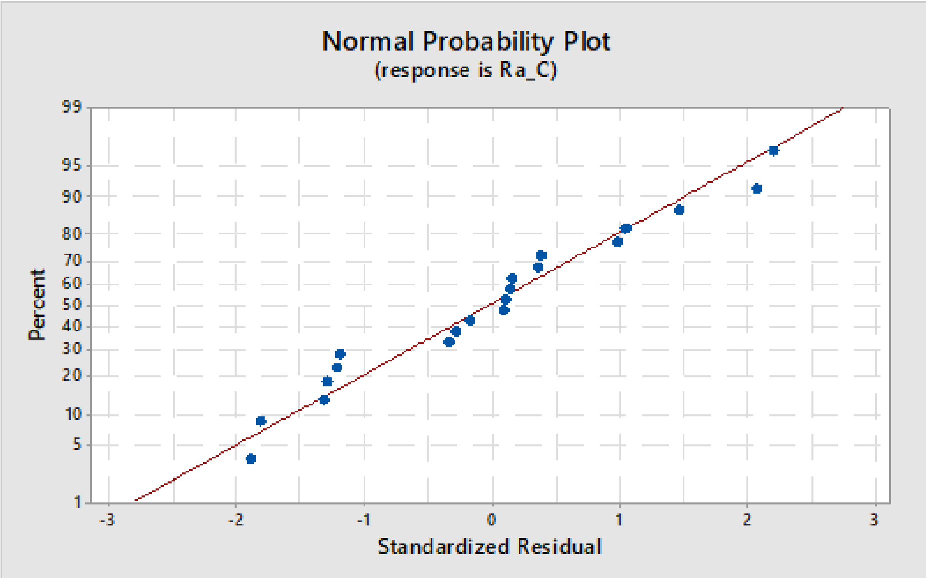

Figure 4

Normal Probability Plot of residuals - RaC

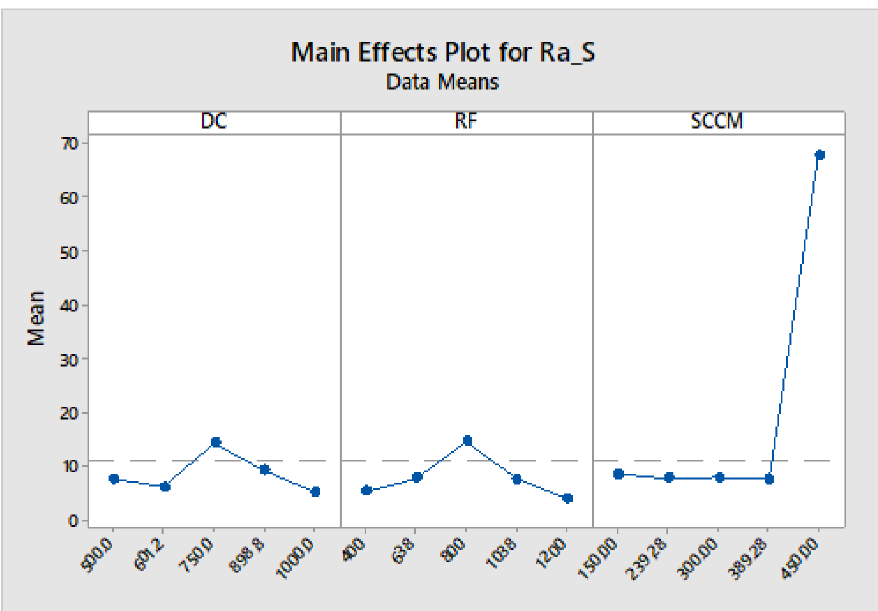

Figure 5

the main effects plot, which is the graphical representation of the main factors affecting the surface roughness .It can be observed from the plot that increase in level of DC power results in increase and then decrease of roughness on SS304. Similar effect can be noticed for RF power but its effect of SCCM at higher level increases the surface roughness.

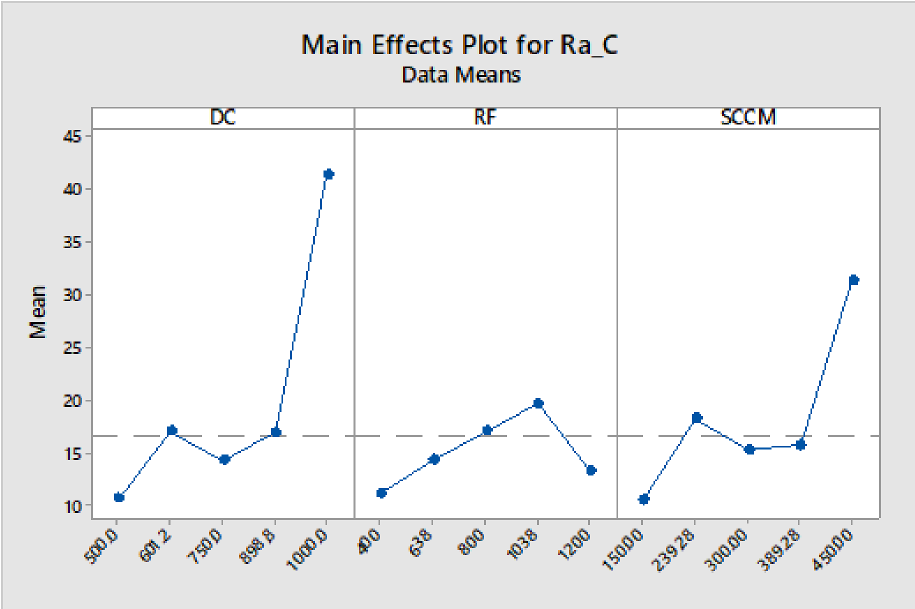

Figure 6

on Copper substrate the effect of RF is following same trend but as DC increases, roughness shows alternative signs, finally it affects at higher level.