

PREDICTION OF SURFACE ROUGHNESS AND APPLICATION OF RESPONSE SURFACE METHODOLOGY TO DEVELOP A MATHEMATICAL MODEL

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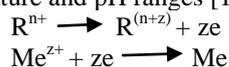
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Abstract: Ni-Cu-P coating was deposited on 99.99% pure Copper substrate electrolessly. The central composite design of experiment has been considered as the statistical analysis tool. The variation in surface roughness is studied by varying three parameters Ni-ion concentration, Cu-ion concentration and reducing agent concentration of the chemical bath. The surface roughness is considered as the response in student's t test and it has been observed that the Ni -ion source and Cu-ion source significantly influence the surface roughness at 10% level of significance. Also all the main effects (Ni-ion source, Cu-ion source, reducing ion source) and the interaction of Ni-ion and reducing agent, significantly influence the surface roughness at 20% level of significance. A mathematical model has developed considering second order response surface methodology which further considers the main effects as well as the effects of interaction and the effects of curvatures.

Keywords: Student's t test, Electroless Ni-Cu-P coating, RSM, CCD

I. INTRODUCTION

The term "Surface Roughness" refers to the deviation or undulation of a surface from a mean level on a given length of the surface. The surface roughness of electroless Ni-Cu-P coating is a very important property, as it determines the resistance to corrosion, predicts the wear phenomenon and determines the friction properties which helps to find the micro-hardness values of the coating-substrate combinations. The electroless bath typically consists of an aqueous solution of metal ions, reducing agents, complexing agents and stabilizers operating in a special ion concentration, temperature and pH ranges [12]. The basic chemical reactions for the electroless plating are as follows [12]:



Though copper have good mechanical properties and high thermal conductivity but oxidation and wear is a major problem in copper which can be solved by Ni-Cu-P coating without changing the bulk property of the substrate.

II. EXPERIMENTAL DETAILS

In this present work electroless Ni-Cu-P coating has been deposited on $(20 \times 15 \times 0.1) \text{ mm}^3$ 99.99% pure Copper samples by Electrolessly. The samples are cut from 99.99% pure Copper strips and first rinsed in distilled water for cleaning and then the samples are acid pickled in dilute HCL. Then again they are rinsed in distilled water. Then the samples are kept to dry. The electroless bath comprised of the following chemical as shown in table 1. The bath is alkaline.

TABLE I
DIFFERENT EXPERIMENTAL FACTORS WITH THEIR RANGES

Factors	Range
NiSO ₄ , 7H ₂ O	23.18 – 56.82 (gm/lit)
CuSO ₄ , %H ₂ O	0.86 – 1.54 (gm/lit)
NaH ₂ PO ₂ , H ₂ O	25.908 – 46.092 (gm/lit)
Na ₃ C ₆ H ₅ O ₇ , 2H ₂ O	55 (gm/lit)
CH ₃ COONa, 3H ₂ O	20 (gm/lit)
Temperature	85°C
Time	30 minutes

After the deposition of coating for 30 mins the samples are rinsed in distilled water and then properly dried. Then The surface roughness measurements are done by TalySurf machine by Taylor Hobson Precision Instrument Surtronic 3⁺. Both Horizontal Magnification (around 100) and Vertical Magnification (around 10000) are done by the instrument accordingly. The surface roughness measurements are taken over a standard sample-length of 25 mm.

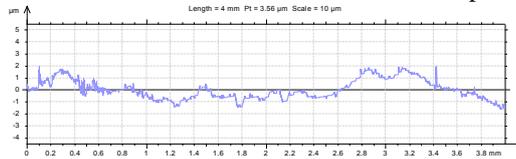


Fig. 1 A sample measurement of the surface profile by Talysurf instrument (without levelling)

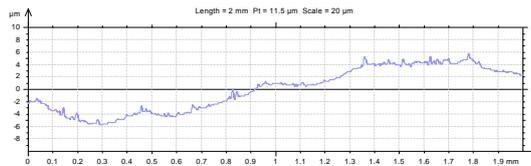


Fig. 2 Measurement of the surface profile of the above sample by Talysurf instrument (with levelling)

TABLE III

AVERAGE SURFACE ROUGHNESS OF THE AS-DEPOSITED SAMPLES AS FOUND IN FACTORIAL, AXIAL AND CENTRAL POINTS

Experiment Points	Sequence No. of Experiments	Run No. of the Experiments	Coded Values of the Parameters			Average Surface Roughness, R_a μm
			NiSO ₄ . 7H ₂ O	CuSO ₄ .5H ₂ O	NaH ₂ PO ₂ . H ₂ O	
			X ₁	X ₂	X ₃	
Factorial Points (n _f = 8)	1	20	-1	-1	-1	0.4212
	2	9	+1	-1	-1	0.3888
	3	10	-1	+1	-1	0.7196
	4	7	+1	+1	-1	0.5282
	5	17	-1	-1	+1	0.524
	6	16	+1	-1	+1	0.565
	7	3	-1	+1	+1	0.5322
	8	18	+1	+1	+1	0.5832
Axial Points (n _a = 6)	9	1	-1.682	0	0	0.509
	10	11	+1.682	0	0	0.4402
	11	2	0	-1.682	0	0.4682
	12	13	0	+1.682	0	0.327
	13	6	0	0	-1.682	0.58
	14	4	0	0	+1.682	0.518
Central Points (n _c = 6)	15	14	0	0	0	0.6798
	16	12	0	0	0	0.5782
	17	8	0	0	0	0.5594
	18	19	0	0	0	0.4268
	19	5	0	0	0	0.439
	20	15	0	0	0	0.5978

III. STATISTICAL ANALYSIS

The Central Composite Design (CCD) is used for modelling a second-order response surface, A CCD consists of 1^k factorial or fractional factorial points, n_f (usually coded ± 1 rotation), 2k axial points [($\pm a, 0, 0, \dots, 0$), (0, $\pm a, 0, \dots, 0$), (0, 0, $\pm a, \dots, 0$), \dots , (0, 0, $\dots, \pm a$)] and n_c central points [(0, 0, 0, $\dots, 0$)] and central points, n_c (to make CCD orthogonal or uniform precision design). The uniform precision design means that the variance of the response at origin is equal to the variance of the response at a unit distance from the origin. A uniform precision design ensures more

protection against bias in the coefficients than an orthogonal design. A CCD can be made rotatable by selecting the appropriate value of α and for a rotatable CCD, $\alpha = \pm \sqrt[4]{n_f}$.

Therefore,

$$Z_i^0 = \text{central point of the design or the basic level } i^{\text{th}} \text{ parameter} = \frac{Z_i^{\max} + Z_i^{\min}}{2}$$

$$\Delta Z_i^0 = \text{unit or interval of variation on the } Z_i \text{ axis for the } i^{\text{th}} \text{ parameter} = \frac{Z_i^{\max} - Z_i^{\min}}{2}$$

$$X_i = i\text{-th independent variable} = \frac{Z_i - Z_i^0}{\Delta Z_i^0}$$

Z_i = actual value of the i -th process parameter

Z_i^{\max} = maximum actual value of the i -th process parameter

Z_i^{\min} = minimum actual value of the i -th process parameter

Let, k = No. of factors = 3 and l = No. of levels = 2,

$\therefore n_f$ = No. of factorial points = $l^k = 2^3 = 8$, n_a = No. of axial points = $2 \times k = 2 \times 3 = 6$ and n_c = No. of central points = 6

$\therefore Z$ = Total no. design points or total no. of observations = $n_f + n_a + n_c = 8+6+6=20$

Now, for Central Composite Design (CCD), the value of $\alpha = \sqrt[4]{n_f} = \sqrt[4]{8} = \pm 1.682$

In order to determine the significance of individual process parameters as well as their interactions, the following equation can be considered [14]:

$$R_a = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 + \beta_{123} X_1 X_2 X_3 + \epsilon$$

Where ϵ represents an error component

The corresponding fitted equation can be expressed as follows [14]:

$$\hat{R}_a = E(R_a - \epsilon) = \hat{\beta}_0 + \hat{\beta}_1 X_1 + \hat{\beta}_2 X_2 + \hat{\beta}_3 X_3 + \hat{\beta}_{12} X_1 X_2 + \hat{\beta}_{13} X_1 X_3 + \hat{\beta}_{23} X_2 X_3 + \hat{\beta}_{123} X_1 X_2 X_3$$

Where $\beta_0, \beta_1, \beta_2, \beta_3, \dots, \beta_{123}$ and $\hat{\beta}_0, \hat{\beta}_1, \hat{\beta}_2, \dots, \hat{\beta}_{12}, \dots, \hat{\beta}_{123}$, are the regression co-efficients.

The co-efficients of the factors of the fitted equation are obtained through the Regression analysis in MINITAB Release 13.1 statistical software.

Regression Analysis equation for R_a versus $X_1, X_2, X_3, X_1X_2, X_2X_3, X_3X_1, X_1X_2X_3$

$$\hat{R}_a = 0.519 + 0.0493X_1 + 0.0274X_2 + 0.0492X_3 - 0.0104X_1X_2 - 0.0006X_2X_3 + 0.0284X_3X_1 - 0.0170X_1X_2X_3$$

The estimated 't' values for particular process parameter can be obtained from the following equation:

$$t_{\text{estimated}} = \frac{\text{Coefficient of process parameters}}{\sigma_\beta} = \frac{|\hat{\beta}|}{\sigma_\beta}$$

If for a particular regression coefficient, $t_{\text{estimated}} > t_{\alpha, v}$, then the main effect or the interaction of the factors corresponding to that particular regression coefficient is significant, i.e. the factor or the interaction significantly affect the process response.

$$\sigma_e^2 = \text{Replication variance} = \sum_{i=1}^{n_c=6} \frac{(Ra_{acti} - Ra_{avg})^2}{(n_c - 1)} = \text{Estimate of error}$$

Where, Ra_{acti} = Actual value of the surface roughness for the i -th central point.

Ra_{avg} = Average value of surface roughness for the central points.

$$n_c = \text{No. of central points} = 6 \quad \& \quad \text{DOF} = n_c - 1 = 6 - 1 = 5$$

$$\therefore Ra_{avg} = \frac{(0.4212 + 0.597 + 0.565 + 0.4268 + 0.524 + 0.5823)}{6} = 0.5195$$

Now,

$$\sigma_e^2 = \frac{(0.00966289 + 0.00600625 + 0.00207025 + 0.00859329 + 0.002025 + 0.0039384)}{6 - 1} = 0.006059354$$

Now $\sigma_\beta^2 = \sigma_e^2 / n_f$

Where, n_f = No. of factorial points. = 8.

$$\text{So, } \sigma_\beta^2 = 0.006059354 / 8 = 0.00075741925 \rightarrow \sigma_\beta = 0.02752$$

Estimated t-values for process parameters and their interactions are calculated and given in the following tabular form.

TABLE IIIII
ESTIMATED T-VALUES OF THE PROCESS PARAMETERS AND THEIR INTERACTIONS

	Constant	X₁	X₂	X₃	X₁₂	X₂₃	X₁₃	X₁₂₃	
σ_e^2	t_0	t_1	t_2	t_3	t_{12}	t_{23}	t_{13}	t_{123}	
0.006059354	0.02752	18.859	1.7914	0.9956	1.7878	0.3779	0.0218	1.032	0.6177

TABLE IVV
FONT SIZES FOR PAPERS TABULATED VALUES OF STUDENT'S T-DISTRIBUTION

Degrees Of Freedom	Level of Significance	
	0.1	0.2
5	$t_{0.1;5} = 1.476$	$t_{0.2;5} = 0.92$

TABLE V
FONT SIZES FOR PAPERS COMPARISON OF ESTIMATED T-VALUES OF THE COEFFICIENTS WITH THE TABULATED VALUES OF STUDENT'S T-DISTRIBUTION

SI No.	Student's <i>t</i> co-efficients	Estimated <i>t</i> -values	$t_{0.1;5} = 1.476$	$t_{0.2;5} = 0.92$
1	t_1	1.7914	Significant	Significant
2	t_2	0.9956	Insignificant	Significant
3	t_3	1.7878	Significant	Significant
4	t_{12}	0.3779	Insignificant	Insignificant
5	t_{23}	0.0218	Insignificant	Insignificant
6	t_{13}	1.032	Insignificant	Significant
7	t_{123}	0.6177	Insignificant	Insignificant

From the above table it can be concluded that (X₁, X₃) two of the main effects are significant at 10% & all the main effects (X₁, X₂, X₃) at 20% level of significance. The interactions X₁₃ is also significant at 20% level of significance. The other interactions are insignificant at both 10% & 20% level of significance.

IV. MATHEMATICAL MODEL FOR SECOND ORDER RESPONSE SURFACE

Response Surface Methodology (RSM) consists of statistical and mathematical techniques which are useful for developing, improving, and optimizing processes. The most extensive applications of RSM can be found in situations when several input variables potentially influence some performance measure or quality characteristic of a process. Thus performance measure or quality characteristic is called the response. This input variables are independent variables. Response Surface method develops a model that describes a continuous surface which connects the measured data taken at strategically important places in the experimental window. It also models the effects of parameters at different levels. RSM uses a least square curve-fit to develop a mathematical model tests its validity and analyze the developed model. RSM has the ability to predict, with statistical limits, the behaviour of the process at any point within the design window. The independent variables (denoted by X₁, X₂...X_k) are assumed to be continuous and can be controlled with negligible error. The response is postulated to be a random variable. The response is postulated to be a random variable. For three independent variables X₁, X₂ and X₃, the response R_a can be represented as a function of X₁, X₂ and X₃ as follows [14]:

$$R_a = f(X_1, X_2, X_3) + \epsilon$$

If the expected response is denoted by $\hat{R}_a = E(R_a - \epsilon)$, then the response surface is represented by $\hat{R}_a = f(X_1, X_2, X_3)$.

A second or higher order RSM Model is necessary to approximate the surface around a curvature. In most cases, a second order RSM model is adequate which can be represented by the following equation:

$$R_a = \beta_0 + \sum_{i=1}^k \beta_i X_i + \sum_{i=1}^k \beta_i X_i^2 + \sum_{i=1}^k \sum_{i<j} \beta_{ij(i<j)} X_i X_j + \epsilon$$

The fitted equation is represented by [14]:

$$\begin{aligned} \hat{R}_a = E(R_a - \epsilon) &= \hat{\beta}_0 + \sum_{i=1}^k \hat{\beta}_i X_i + \sum_{i=1}^k \hat{\beta}_i X_i^2 + \sum_{i=1}^k \sum_{i<j} \hat{\beta}_{ij(i<j)} X_i X_j + \epsilon \\ &= \hat{\beta}_0 + \hat{\beta}_1 X_1 + \hat{\beta}_2 X_2 + \hat{\beta}_3 X_3 + \hat{\beta}_{11} X_1 X_1 + \hat{\beta}_{22} X_2 X_2 + \hat{\beta}_{33} X_3 X_3 + \hat{\beta}_{12} X_1 X_2 + \hat{\beta}_{23} X_2 X_3 + \hat{\beta}_{13} X_1 X_3 \end{aligned}$$

Where, ϵ = error component.

E = a mathematical expectation

The co-efficients of the fitted equations are obtained through the Response Surface Analysis in MINITAB Release 13.1 statistical software. The Second Order Response Surface Equation for Surface Roughness ($R_a \mu\text{m}$) is as follows:

$$\widehat{R}_a = 0.51985 + 0.04932X_1 + 0.02736X_2 + 0.04925X_3 + 0.01101X_1X_1 - 0.00490X_2X_2 - 0.00695X_3X_3 + 0.01040X_1X_2 + 0.02840X_1X_3 - 0.00055X_2X_3$$

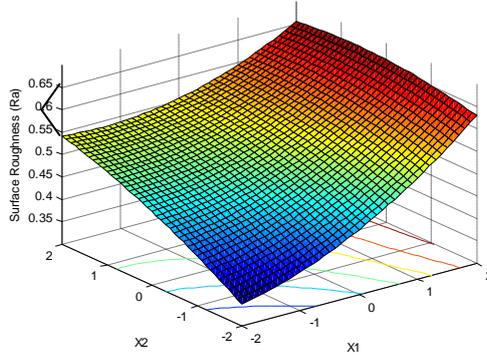


Fig. 3 Surface Contour plots for $\widehat{R}_a = f(X_1, X_2)$

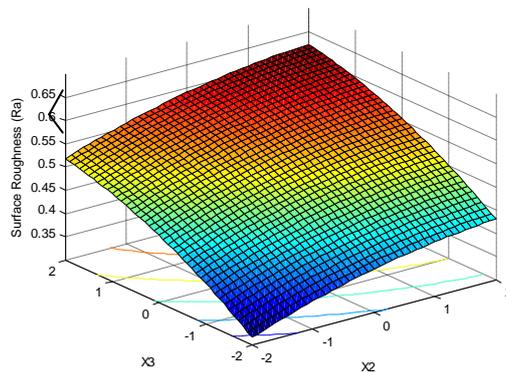


Fig. 4 Surface Contour plots for $\widehat{R}_a = f(X_2, X_3)$

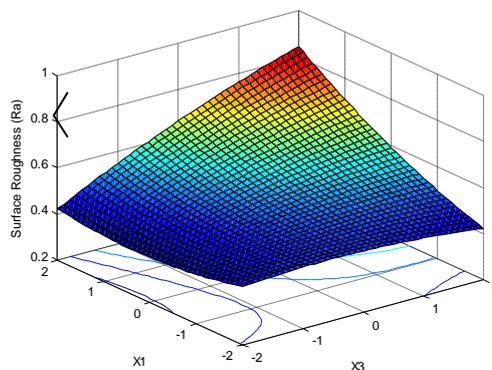


Fig. 5 Surface Contour plots for $\widehat{R}_a = f(X_3, X_1)$

The test of reliability for the predicting response surface equations has been carried out by Fisher's Variance Ratio known as the *F*-Test. The *F*-ratio is given by the following equation [14]:

$$F = \sigma_{res}^2 / \sigma_e^2$$

Where,

$$\sigma_{res}^2 = \text{Residual variance} = \sum_{i=1}^{N=20} \frac{(Ra_{acti} - Ra_{esti})^2}{(Z - n)}$$

$$\sigma_e^2 = \text{Replication variance} = \sum_{i=1}^{n_c=6} \frac{(Ra_{acti} - Ra_{avg})^2}{(n_c - 1)}$$

n = number of coefficients in the regression equation

Ra_{esti} = estimated value of Surface Roughness for the *i*-th observation

The following table gives the values of σ_{res}^2 , σ_e^2 and the *F*-values for the for the predicting response surface equation:

TABLE VV
 FONT SIZES FOR PAPERS ESTIMATION OF FISHER'S F-RATIO (FOR SURFACE ROUGHNESS)

Predicting Response Surface Equation	Residual Variance	Replication Variance	Estimated F-value
$R_a = f(X_1, X_2, X_3)$	0.0088	0.006059354	1.4523

The upper degrees of freedom ($v_1=Z - n$) and lower degrees of freedom ($v_2 = n_c-1$) are 10 and 5 respectively. The *F*-value for 1% level of significance (for $v_1=10, v_2=5$) is $F_{0.01;10,5} = 10.05$. The estimated *F*-value (1.4523) for the prediction of response surface equation, $R_a = f(X_1, X_2, X_3)$, is less than 10.05. Hence it can be concluded that the established response surface predicting equation give a good fitting to the observed data.

V. CONCLUSION

The surface roughness is considered as the response in student's test and it can be concluded that the Ni -ion source and Cu-ion source significantly influence the surface roughness at 10% level of significance. Also all the main effects (Ni-ion source, Cu-ion source, reducing ion source) and the interaction of Ni-ion and reducing agent, significantly influence the surface roughness at 20% level of significance.

Fig 3-5 shows the surface plots and contour plots for $\hat{R}_a = f(X_1, X_2)$, $\hat{R}_a = f(X_2, X_3)$, and $\hat{R}_a = f(X_1, X_3)$. From fig 3 it can be observed that, keeping $X_3=0$, for constant values of Copper source concentration (X_2), the Surface Roughness of the samples increases with the increase in Nickel source concentration (X_1). For constant values of X_1 , the Surface Roughness of the samples increases with the increase in copper concentration (X_2)

From fig 4 it can be observed that, keeping $X_1=0$, for constant values of Copper source (X_3), the Surface Roughness of the samples increases with the increase in copper concentration (X_1) and then again decreases moderately. And for constant values of X_2 , the Surface Roughness increases upto a certain value of Copper source concentration (X_2) and then again decreases moderately.

From Fig 5 it can be observed that keeping $X_2 = 0$, for constant values of reducing agent concentration (X_3), the Surface Roughness decreases moderately and then again increases moderately upto a certain value of Nickel source concentration (X_1). And for constant values of X_1 , the Surface Roughness increases moderately upto a certain value of reducing agent concentration (X_3) and then again increases.

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