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Technical Report

Analyzing the Influence of Descale Process Parameters on Electroplating by using Statistical Tools

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Abstract

Background and Objective: Surface of substrates used in electroplating needs to be free from contaminants during the electroplating process. For this study the substrate material is nickel-cobalt (Ni-Co) which is contaminated with gold and the gold is transformed to gold oxide during heat treatment. The contamination results in poor adhesion of the coating on the substrate during electroplating and thereby, causing inefficient solderability. The objective of this study was to find a correlation of the descale process parameters on the occurrence of exposed base metal of nickel-cobalt film which was contaminated with gold. **Methodology:** In this study, the statistical analysis on the effects of modified pretreatment was investigated. Apart from that, the steps of surface preparation were also modified. The actual electroplating line was used to conduct the experimental runs and research. The results were analyzed using factorial analysis of variance method and using the 3-Dimensional plot and optimized using the response optimizer. Ni-Co substrate which was contaminated with gold was used as a base material for plating, pure tin was used as a plating medium on the substrate. Descale process used different types of acid as an electrolyte medium. A significant factor or α of 0.05 was employed for this experiment. **Results:** In the modified pre-cleaning process the interactive effects of the electrolyte concentration, the immersion time and the electrolysis current played a crucial role in eliminating the rejects. **Conclusion:** Individual input variables do have a significant effect on the variation of exposed base metal. Moreover, the interactive effects of the variables also have a significant effect on eliminating the occurrences of defects during electroplating. The rejection of substrate could be eliminated completely after electroplating.

Key words: Exposed base metal, descale, concentration, current, contamination, correlation

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Data Availability: All relevant data are within the paper and its supporting information files.

INTRODUCTION

The relationship of the descale input parameters play a role in the effective descaling of the parts to be plated. In order to achieve a good adhesion between the coating material and the base material, the surface of the base material needs to be cleaned thoroughly to remove traces of grease and dirt[1]. Adhesion of the plating film over the substrate is considered one of the most important factors in the electroplating process[2,3]. A properly cleaned surface prior to electroplating is crucial for the good adhesion of the plating film. Here the independent variables are the type of pre-cleaning chemical used and the dependent variables are the input parameters setup.

Pre-cleaning is required on the surface of the substrate intended for plating prior to the electroplating process. This is in order to obtain a plating finish that is flawless and has a good adhesion onto the base metal[4]. The pre-cleaning processes consist of cleaning the surface intended for plating through the use of alkaline cleaning which is followed by a short high pressure water jet during the pre-cleaning process, the components are washed thoroughly to remove all dirt and greases, oxide and to prepare the surface for electroplating. This pre-cleaning process requires a set of procedures for each step of the pre-cleaning.

The input parameters of the plating process does not have an impact on the adhesion of the plating film to the substrate, however, this is in comparison with the input parameters of the pre-cleaning parameters which have a direct impact[5-7]. Descale which is a part of the pre-cleaning process plays an important role in the adhesion of the plating film over the substrate, also descale which are of different composition and recipe will have different effects on heat scale which were formed over the substrate surface and also on the oxide and contaminant removal from the substrate prior to plating[8].

For electroplating in the electronic industry the common materials used are, copper and copper alloys, aluminum, silicon and glass and Ni-Co alloy. Copper and copper alloys are widely used in plating due to good electrical conductivity which is relevant in the electronics industry, however these metals are also prone to corrosion, which in turn decreases the electrical conductivity of the substrates[9]. In order to prevent the corrosion and for soldering purpose these materials are electroplated. Numerous alloys are used as substrates among which Ni-Co alloys have been widely used as an important engineering material in industry because of their high strength, good corrosion resistance, heat-conductive and electro-catalytic activity[10]. When the adhesion of the plating to the base metal is weak, there possibility of peeling especially after a heat induced process such a reflow is high. This will result in the scrapping of the product, which in turn increases the product cost[11].

In this study, the effect of the interactions between the different descale input variables during processing Ni-Co substrate contaminated with gold is investigated. From the study, it was observed that the effects form the individual variables and the effects from the interaction of the variables played a role in the reduction of the exposed base metal on the substrate surface. These effects played an important role in determining the rejection rate which ultimately will translate to cost reduction.

MATERIALS AND METHODS

Response surface methodology: For this study, the response surface methodology or RSM method was used in design of experiment (DOE) where the input factors, which were likely to be important on the response that is, the exposed base metals were generated first. This was also used to investigate the factors which are mainly influencing the response and eliminating the factors which are not significant. Upon the completion of this stage, all of the important factors were identified and the next stage was to determine if the collected data lie relative to the ideal response. The next phase was to optimize the process. The response surface methodology analysis was carried out in year 2017 at University Tenaga Nasional.

Factorial designs: A set of advanced DOE techniques was used which gave a better understanding of the plating and pre-cleaning process and enabled the processes to be optimized. This is part of the response surface design methodology. There are two types of response surface designs which are centre composite design which can fit a full quadratic model and used when there is a design plan for sequential experimentation as the design will accommodate the information form a factorial experiment which has been planned correctly[12]. The factorial design can either be full or fractional design.

Use of Minitab: Minitab was used to create the design of experiments for the intended run for all the experiment set up. In order to study and obtain a better understanding for each of the input factors on the output variables and also to study the effects of the interactions between the input factors on the response.

Experimental procedure: In electroplating process the cleanliness of the substrate plays an important role in order to achieve a good adhesion. The experiments were conducted under three different set conditions, where the pre-cleaning parameters, the precleaning setup and the plating parameters were changed, respectively.

In the first set, the electroplating process variables were changed, following the standard pre-treatment steps. Three plating parameters: Plating acid concentration, the electrolyte concentration and the plating temperatures were considered. In the second set of experiments the descale parameters were varied namely the descale concentration, descale current and descale immersion time and in the third set the entire descale set up was changed where two different types of descale acids were used with variation in the descale concentration, descale current and descale immersion time for each of the descale acid. A design of experiment (DOE) was conducted using statistical tool to carry out the experiments.

Electrolyte concentration: The electrolyte concentration is crucial to have quality end product. This will indirectly affect the throwing power of the electroplating bath. Furthermore, the settings should be within the recommended limits. Hence, the parameter was chosen within minimum and maximum values for the pre-cleaning and plating baths[13,14].

Electrolyte current: To maintain electrolyte current, the voltage was selected within the rage of the desired voltage settings. Any increase or decrease in the voltage will increase or decrease the reaction which takes place on the substrate surface during electroplating. Too low voltage setting will disrupt the flow of the electrons in the process cell and too high of a voltage will result on excessive reaction on the substrate surface[15,16].

Electrolyte immersion time: The immersion time of the substrate in the electrolyte will affect the reaction which takes place, a longer immersion time will results in increased reaction and a shorter immersion time will result in a decreased reaction at the cathode or on the substrate[17-20].

In Table 1-3, the experimental conditions and exposed surface after electroplating are presented.

RESULTS

For the 1st set of experiments, the variation of electroplating parameters with standard pre-treatment did not improve the rejection rate of substrate to be used in integrated circuits for electronic industry. There was only effect on the appearance and thickness of the plating surface. Hence, the electroplating conditions do not play any role in reducing the defect. In Fig. 1, the poor solderability from first set of experiment is presented.

Table 1: Experimental condition and exposed surface with standard pre-cleaning but modified plating parameters

	Factors			Results
Run condition	Concentration of tin in electrolyte (g L^{-1})	Immersion time (sec)	Current of plating (amp)	Exposed base metal (%)
1	55	120	85	90
2	145	120	85	92
3	55	240	85	91
4	145	240	85	95
5	55	120	155	97
6	145	120	155	96
7	55	240	155	92
8	145	240	155	91
9	100	180	120	95

Table 2: Experimental condition and exposed surface with variation of standard pre-cleaning parameters

	Factors			Results
Run condition	Concentration of descale acid (g L^{-1})	Immersion time (sec)	Descale current (amp)	Exposed base metal (%)
1	55	55	1	35
2	55	35	1	34
3	55	35	24	39
4	55	55	24	38
5	100	45	12.5	29
6	100	45	12.5	31
7	100	45	12.5	39
8	145	55	1	31
9	145	35	1	32
10	145	35	24	27
11	145	55	24	28

Table 3: Experimental condition and exposed surface modified descale set up

	Descaling with sulfuric acid			Descaling with sodium persulfate+hydrogen sulfate			Results
Run condition	Concentration (%)	Immersion time (sec)	Descale current (amp)	Concentration (g L^{-1})	Immersion time (sec)	Descale current (amp)	Exposed base metal (%)
1	12.5	35	1	55	35	1	0.86
2	25.0	35	1	55	35	24	0.00
3	12.5	55	1	55	35	24	0.37
4	25.0	55	1	55	35	1	0.00
5	12.5	35	24	55	35	24	0.00
6	25.0	35	24	55	35	1	0.00
7	12.5	55	24	55	35	1	0.00
8	25.0	55	24	55	35	24	0.00
9	12.5	35	1	145	35	24	0.79
10	25.0	35	1	145	35	1	0.00
11	12.5	55	1	145	35	1	0.19
12	25.0	55	1	145	35	24	0.00
13	12.5	35	24	145	35	1	0.00
14	25.0	35	24	145	35	24	0.00
15	12.5	55	24	145	35	24	0.00
16	25.0	55	24	145	35	1	0.00
17	12.5	35	1	55	55	24	0.54
18	25.0	35	1	55	55	1	0.00
19	12.5	55	1	55	55	1	0.19
20	25.0	55	1	55	55	24	0.00
21	12.5	35	24	55	55	1	0.00
22	25.0	35	24	55	55	24	0.00
23	12.5	55	24	55	55	24	0.00
24	25.0	55	24	55	55	1	0.00
25	12.5	35	1	145	55	1	0.62
26	25.0	35	1	145	55	24	0.00
27	12.5	55	1	145	55	24	0.39
28	25.0	55	1	145	55	1	0.00
29	12.5	35	24	145	55	24	0.00
30	25.0	35	24	145	55	1	0.00
31	12.5	55	24	145	55	1	0.00
32	25.0	55	24	145	55	24	0.00
33	18.75	45	12.5	18.75	45	12.5	0.14
34	18.75	45	12.5	18.75	45	12.5	0.22
35	18.75	45	12.5	18.75	45	12.5	0.08

Fig. 1: Solder test after electroplating with standard surface cleaning[10]

Fig. 2: Solder test after electroplating with persulfate as descale[10]

In the 2nd set of experiments, the variation of the descale parameters did not have a significant impact on the occurrence of exposed base metal although there was a slight improvement in the adhesion. The set conditions also resulted inefficient solderability as presented in Fig. 2.

Fig. 3: Plating with good adhesion onto substrate under modified pre-cleaning[10]

In the 3rd set of experiments, the improper adhesion of the plating film was able to be resolved through modifying the pre-treatment method, thereby, making efficient solderability without failure as depicted in Fig. 3. The rejection rate of substrate was totally eliminated through the modification of the pre-plating. The foreign metallic elements which are present on the substrate caused improper adhesion and poor adhesion.

Statistical method: For the third set of experiment which produced promising results, a response surface methodology (RSM) method was carried out using factorial ANOVA. A significant factor or α of 0.05 was employed for this experiment. Here, the RSM method used statistical and mathematical techniques with the purpose of developing and optimizing the process, designing, developing and formulation of new improved method and the improvement of existing design. The quality and performance in terms of exposed base metal due to blistering were measured in a continuous scale which is known as the response. The input variables are known as independent variables[21,22].

For this experiment, a fractional factorial design was created using Minitab. The high and low settings from all of the input parameters which can be adjusted or controlled were selected for these designs. Upon the completion of the DOE design, the matrix was generated in a random order. The runs for each experiment were carried out in blocks with the purpose of detecting variables which can cause variation in the results. This variation is also known as the nuisances variables. The experiment was carried out using orthogonal blocking[23].

Minitab was used to create an RSM blocking design which was used to optimize the runs. The results of the block design with the exposed base metal are presented in Table 4. Additional input variables were generated. These were center points. The exposed base metals were obtained through the experiments carried out as per the settings in the Table 4.

The main and interacting effects of the input variables are shown in Table 5.

From the main and interacting effects which were derived, it was observed that the individual (main) and interactive effects of some of the variables played a role in the reduction of occurrence of exposed base metal. The individual (main) effects are the concentration of the sulfuric based descaler, immersion time of the substrate in the sulfuric descaler and the electrolyte current of the sulfuric descaler. The interaction effect of the concentration of the sulfuric based descaler combined with the current flow in the sulfuric based descaler and the concentration of the sulfuric descaler and the immersion time of the substrate in the sulfuric descaler has a significant effect on the occurrence of exposed base metal. For this experiment a significant effect of 0.05 was chosen where the value 0.05 represents 5% from the 95% confidence level.

Linear regression equation: Multiple regression is used to predict a single variable from one or more independent variables. Here the regression equation is an equation that describes the relationship between one or more predictor variables and the response variable[24]. The developed mathematical model on the exposed base metal is displayed below:

$$\begin{gathered}
\text{Maximum exposed base metal} = 0.1310-0.1243\ A-0.0536\ B- \\
0.1243\ C-0.0222\ D-0.0214\ E+0.0007\ F+0.0536\ A*B+ \\
0.1243\ A*C-0.0012\ A*D+0.0214\ A*E-0.0007\ A*F+ \\
0.0535\ B*C+0.0004\ B*D+0.0159\ B*E+0.0165\ B*F- \\
0.0012\ C*D+0.0147\ C*E-0.0071\ C*F+0.0231\ D*E+ \\
0.0223\ D*F+0.0003\ E*F-0.0535\ A*B*C-0.0004\ A*B*D- \\
0.0159\ A*B*E-0.0165\ A*B*F+0.0012\ A*C*D- \\
0.0148\ A*C*E+0.0071\ A*C*F-0.0231\ A*D*E- \\
0.0223\ A*D*F-0.0002\ A*E*F+0.0167\ A*B*C*E*F
\end{gathered}$$

where, factors A, B, C, D, E and F are defined in Table 5.

The developed equation showed that the most significant factors are the concentration, current and the immersion time of the sulfuric descale and the interaction of the three variables.

Dimensional surface plot for exposed base metal: In order to obtain a better description on how the exposed base metal varies with the settings of the concentrations of sulfuric descale, the immersion time of sulfuric descale and the current

Table 4: RSM block design and exposed base metal

Std order	Run order	Center Pt	Blocks	Concentration sulfuric based descaler	Immersion time sulfuric descale	Current sulfuric descale	Concentration persulfate descale	Immersion time persulfate descale	Current persulfate descale	Exposed base metal (%)
19	1	1	1	12.50	55	1	55	55	1	0.19
4	2	1	1	25.00	55	1	55	35	1	0
16	3	1	1	25.00	55	24	145	35	1	0
28	4	1	1	25.00	55	1	145	55	1	0
22	5	1	1	25.00	35	24	55	55	24	0
7	6	1	1	12.50	55	24	55	35	1	0
18	7	1	1	25.00	35	1	55	55	1	0
6	8	1	1	25.00	35	24	55	35	1	0
2	9	1	1	25.00	35	1	55	35	24	0
31	10	1	1	12.50	55	24	145	55	1	0
3	11	1	1	12.50	55	1	55	35	24	0.37
33	12	0	1	18.75	45	12.5	18.75	45	12.5	0.13
13	13	1	1	12.50	35	24	145	35	1	0
9	14	1	1	12.50	35	1	145	35	24	0.78
24	15	1	1	25.00	55	24	55	55	1	0
30	16	1	1	25.00	35	24	145	55	1	0
5	17	1	1	12.50	35	24	55	35	24	0
32	18	1	1	25.00	55	24	145	55	24	0
20	19	1	1	25.00	55	1	55	55	24	0
21	20	1	1	12.50	35	24	55	55	1	0
17	21	1	1	12.50	35	1	55	55	24	0.53
25	22	1	1	12.50	35	1	145	55	1	0.62
27	23	1	1	12.50	55	1	145	55	24	0.38
35	24	0	1	18.75	45	12.5	18.75	45	12.5	0.22
10	25	1	1	25.00	35	1	145	35	1	0
29	26	1	1	12.50	35	24	145	55	24	0
23	27	1	1	12.50	55	24	55	55	24	0
26	28	1	1	25.00	35	1	145	55	24	0
12	29	1	1	25.00	55	1	145	35	24	0
15	30	1	1	12.50	55	24	145	35	24	0
1	31	1	1	12.50	35	1	55	35	1	0.85
8	32	1	1	25.00	55	24	55	35	24	0
34	33	0	1	18.75	45	12.5	18.75	45	12.5	0.08
14	34	1	1	25.00	35	24	145	35	24	0
11	35	1	1	12.50	55	1	145	35	1	0.19

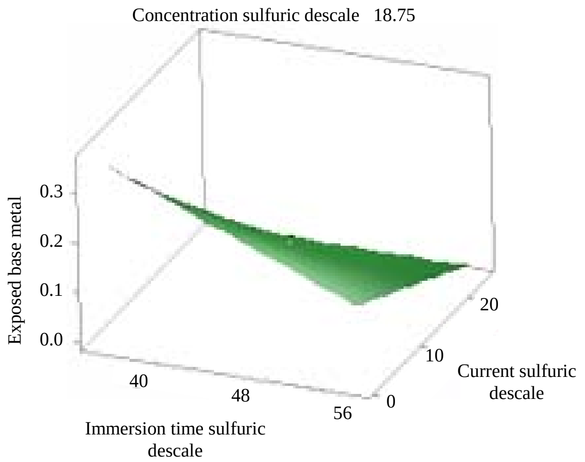

Fig. 4: Surface plot of exposed base metal vs. current flow in sulfuric descale and immersion time in sulfuric descale

of sulfuric descale, 3 Dimensional (3D) surface plot were generated in Minitab. The relationships between the variables are shown in Fig. 4-6.

The 3 dimensional plots of current sulfuric descale vs. the immersion time sulfuric descale are generated and shown in Fig. 4.

From Fig. 4, it can be seen that the lowest point of exposed base metal is at current sulfuric descale at approximately 24 amp and the immersion time of the substrate in the sulfuric acid descale is at approximately 25 sec.

Figure 5 shows the exposed base metal with respect to concentration and immersion time. The lowest point of exposed base metal is at 25% concentration of sulfuric descale and 55 sec of immersion time in sulfuric descale.

Figure 6 presents the plot of exposed base metal with respect to concentration of sulfuric descale and current flow

Table 5: Main and interacting effects of different variables on exposed base metal

Term	Term	Coefficient	p-value	Significance
Constant/label	Constant/label	0.1310	0.009	
Concentration sulfuric based descaler	A	-0.1243	0.010	Significant
Immersion time sulfuric descale	B	-0.0536	0.049	Significant
Current sulfuric descale	C	-0.1243	0.010	Significant
Concentration persulfate descale	D	-0.0222	0.542	
Immersion time persulfate descale	E	-0.0214	0.225	
Current persulfate descale	F	0.0007	0.958	
	A*B	0.0536	0.049	Significant
	A*C	0.1243	0.010	Significant
	A*D	-0.0012	0.946	
	A*E	0.0214	0.225	
	A*F	-0.0007	0.958	
	B*C	0.0535	0.043	
	B*D	0.0004	0.985	
	B*E	0.0159	0.300	
	B*F	0.0165	0.287	
	C*D	-0.0012	0.946	
	C*E	0.0147	0.327	
	C*F	-0.0071	0.598	
	D*E	0.0231	0.287	
	D*F	0.0223	0.300	
	E*F	0.0003	0.985	
	A*B*C	-0.0535	0.043	
	A*B*D	-0.0004	0.985	
	A*B*E	-0.0159	0.300	
	A*B*F	-0.0165	0.287	
	A*C*D	0.0012	0.946	
	A*C*E	-0.0148	0.327	
	A*C*F	0.0071	0.598	
	A*D*E	-0.0231	0.287	
	A*D*F	-0.0223	0.300	
	A*E*F	-0.0002	0.985	
	A*B*C*E*F	0.0167	0.566	

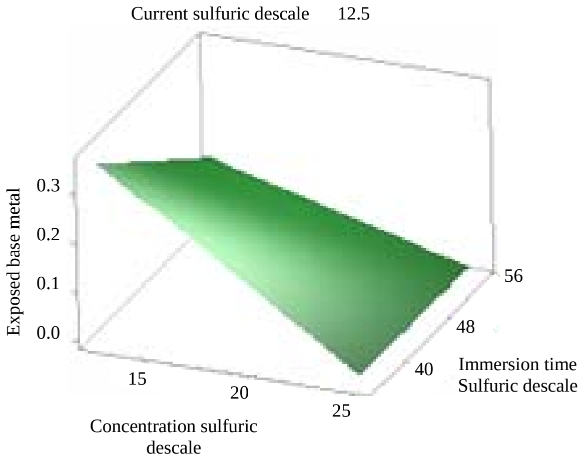

Fig. 5: Surface plot of exposed base metal vs. concentration of sulfuric descale, immersion time in sulfuric descale

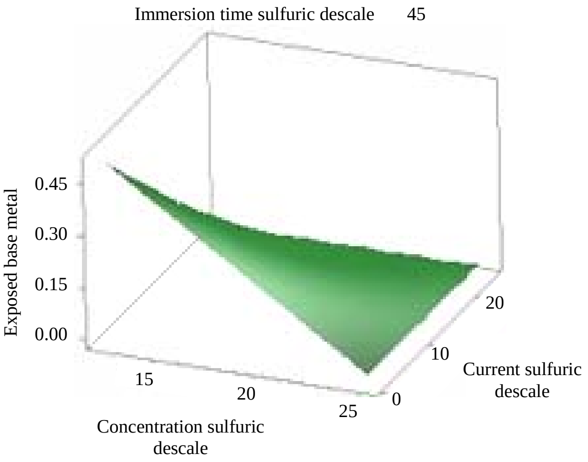

Fig. 6: Surface plot of exposed base metal vs. concentration of sulfuric descale, current flow in sulfuric descale

in sulfuric descale. From the graph it is observed that the lowest point of exposed base metal is when the concentration of the electrolyte is 25% and the current flow in sulfuric descale is 24 Amp.

Response optimizer: To have a fixed set of control limits for the descale process, response optimizer was used. By using response optimizer it was observed that the optimized values of variables are immersion time 23.73 sec, concentration

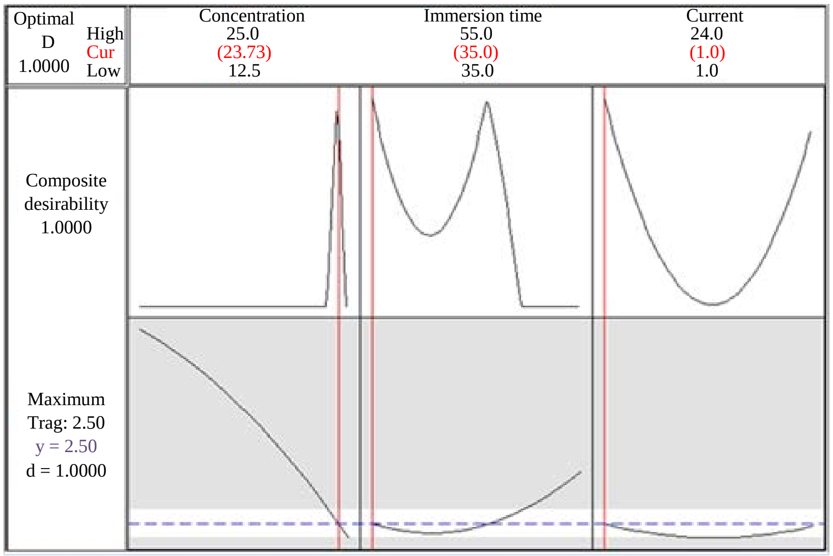

Fig. 7: Response optimizer initial plot

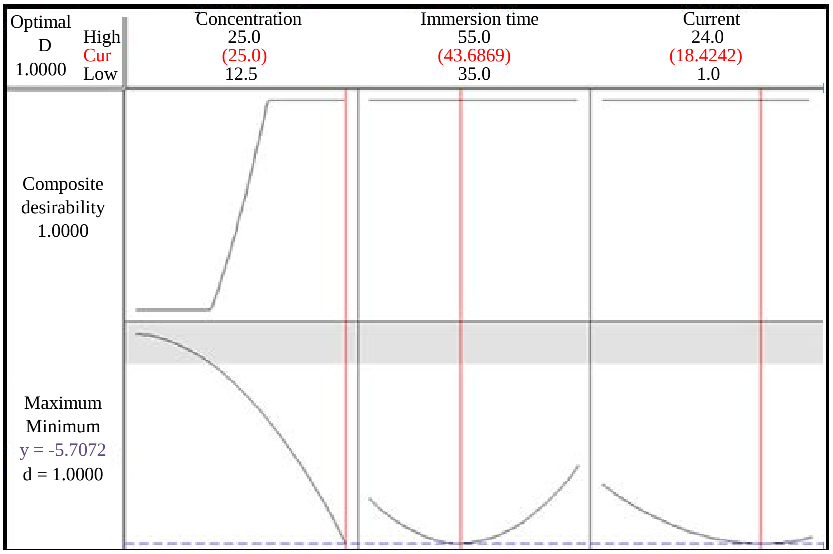

Fig. 8: Response optimizer modified plot

23.73% and current of 1.0 amp. Under this condition, the predicted exposed base metal was 2.5% of the substrate surface. The plots of optimizer are presented in Fig. 7-8.

To understand how each factor affects the response and if it is possible to be reduced further the occurrence of exposed base metal, the response optimizer was then adjusted by moving the vertical bars to. The optimized values for the variables are concentration 25.0%, immersion time 43.6869 sec and current 18.42 Amp. Under this condition, the exposed base metal was 0% substrate surface.

DISCUSSION

The combination of the concentration of the sulfuric descaler and the current flow in the descaler will increase the attack rate of the oxide on the substrate at the cathode with an increase in the reaction taking place. As shown by Senthilkumar *et al.*[25], an increase in the electrolyte concentration will have an impact on the output parameters of the process.

The next combination of the concentration of the sulfuric descaler and the immersion time will enable a longer rate of oxide removal. When there is a longer rate of oxide removal combined with a stronger etching rate to take place and the increase immersion time and the current flow will enable the reaction at the cathode to be increased for a longer period of time which will remove the oxide on the substrate[26,27]. The correlation between the immersion time and the electrochemical reaction was also confirmed by Srinivasan and Mayo[28].

It was also observed by Singh *et al.*[29] that an increase in the voltage will raise the current and increased current will enhance the surface finish. The same result was also obtained by Suresh and Mahadevan[30] and where the correlation between the material removal rate and the increase in voltage was depicted.

It is clear from the plot that the combination of concentration of the sulfuric acid descaler, the immersion time and the current used in the descale process play a very important role in the occurrence exposed base metal. As explained previously when the oxide contamination is present on the surface of the substrate, it will reduce the adhesion of the plating film onto the substrate[17,31]. As investigated by Bose and Mitra[32], the input voltage/current parameter and the electrolyte concentration will have an effect of the reaction which takes place at the cathode, where an increase in the voltage increases the reaction and also an increase in concentration increases the reaction.

To compensate for the oxidation, the etching of the substrate surface with tough descale process needs to be done without compromising the quality of the parts. In order to achieve this two-step descale was introduced and the current, immersion time and the concentration of the sulfuric based descale were adjusted.

CONCLUSION

The analysis of the end results obtained from all the experiments conducted showed that the exposed base metal varied when the set up and the input variables of the pre-cleaning process namely when the descale process was changed. It was also observed that the exposed base metal due to blistering of Ni-Co substrates contaminated with gold was eliminated completely and the process has been optimized successfully through the RSM method. The significant main and interacting effects of the input variables were identified by two level factorial designs. The response surface methodology provided an in-depth understanding of how the input variables and the output variables were correlated to each another. Hence, the overall performance of the electroplating system was enhanced by changing the process flow in the pre-cleaning step and through adjusting the input variables. The optimized condition could produce defect free electroplated substrate. The rejects were not generated even at the lower level of electroplating parameters. This transformed indirectly into savings in the form of material usage and power consumption during electroplating. Hence, it can be said that the reliability of the electronic devices was enhanced and the process became cost effective.

SIGNIFICANCE STATEMENTS

- Integrated circuit chip are built around different alloy materials for the first time which are known as substrates
- The individual effects and combination of pre-treatment variables methods for nickel-cobalt substrate prior to electroplating are studied
- Effects of different input parameter settings on the occurrence of peeling plating film were studied
- Optimization of the process was carried to find the optimum setting which provided zero defects of exposed base metal which helps researchers in future

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