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Experimental Investigation of Photochemical Machining on Inconel 600 using Ferric Chloride

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***Abstract*-This experimental investigation analyses the influence of etching conditions on maximum etch factor during photo chemical machining process of Inconel 600 with etchant Ferric chloride. Analysis of variance (ANOVA) is employed to investigate the influence of time, temperature and concentration on maximum etch factor of the material. In this paper experiments were conducted to varying the parameters of photo chemical machining process like temperature from 55^{0}C to 65^{0}C, Time from 30 min to 70 min and concentration 300 gm/lit to 700 gm/lit are studied in details according to Face centered composite design of experiments. At last results are analyzed using Response surface methodology, ANOVA and parametric optimization is done for maximum etch factor of material. From the experimentation, it is concluded that for photo chemical machining of Inconel 600 optimum machining condition for maximum etch factor with Time (30 min), Temperature (65^{0}C), and Concentration (700 gm/lit).**

Keywords - Photochemical machining (PCM), Response surface methodology (RSM), Face centered composite design (FCCD), Inconel 600, etch factor.

I. INTRODUCTION

Photochemical machining (PCM) is one of the least well known non-conventional machining processes. Photochemical machining basically removes material by chemical action. Application of the process often produces a flat metal blank. The features are created by exposing the substrate of interest through a photographic mask and chemically etching away areas that leave the features of interest. The manufacturing process creates features by dissolving away metal rather than cutting or burning it away. So the stresses and defects that normally arise from metal cutting or burning are absent in the final part. That means there are no burrs, no residual stresses, no changes in magnetic properties, and no deformations. There are no changes in hardness, grain structure, or ductility during the process. The main limitation of PCM is to be found in the characteristics of isotropic etching whereby the etchant will attack not only downwards in to the metal but also sideways beneath the resist stencil layer it is also known as undercut .The ratio of depth to the undercut is termed the "etch factor". In this, paper the use of Response surface methodology to optimize the etch factor during the PCM process of Inconel 600 material. Inconel 600 has excellent mechanical properties and having desirable combination of high strength and good workability. The chemical composition of Inconel 600 is shown in Table I. The high nickel content in Inconel 600 alloy gives the resistance to corrosion by many organic and inorganic compounds. Chromium confers resistance to sulfur compounds & oxidizing conditions at high temperatures or in corrosive solutions. The adaptability of Inconel 600 has led to it's utilize in a variety of applications involving temperatures from cryogenic to above 1000°C. The alloy is used extensively in the chemical industry for its strength and corrosion resistance. The alloy's strength and oxidation resistance at high temperatures make it useful for many applications in the heat-treating industry. In the aeronautical field, Inconel 600 is used for a variety of engine and airframe components which must withstand high temperatures. Table I shows Chemical composition of Inconel 600.

In the literature, David et al. [2] has studied Characterization of aqueous ferric chloride etchants used in industrial photochemical machining process. Fecl$_3$ most commonly used as etchants. But there is wide variety in grades of Fecl$_3$. Defining standards for industrial purpose etchants and methods to analyze and monitor them. Rajkumar et al. [3] have explained the Cost of photochemical machining in which they gave the cost model for PCM. Saraf et. Al. [4] has studied optimization of photochemical machining of OFHC copper by using ANOVA. Saraf and Sadaiah et. Al. [5] have investigated optimization of photochemical machining of SS304. Cakir O, et. Al. [6] found that ferric chloride (FeCl3) was a suitable etchant for aluminum etching. From the literature it is found that no statistical study has been reported to analyze the interaction effects of input parameters on etching process of Inconel 600. The process parameters affecting etch factor are grouped into three broad areas, etchant concentration, etching time and etchant temperature. To improve the product quality proper selection of PCM process parameter is very important. In this paper we used Response surface methodology (RSM) to optimize the process parameters of PCM on Inconel 600 with consideration of output parameter such as etch factor is reported. RSM is frequently employed to obtain the optimum parameter setting following analysis of variance (ANOVA) for identifying significant factors.

TABLE I. CHEMICAL COMPOSITION OF INCONEL 600

Ni	Cr	Mn	C	Cu	Si	S	Fe
72	14-17	1	0.15	0.50	0.50	0.015	6-10

II. EXPERIMENTAL DESIGN

A. Response surface methodology

Response surface methodology (RSM) is a collection of mathematical and statistical techniques useful for analyzing problems in which several independent variables influence a dependent variable or response, and the goal is to optimize this response. In many experimental conditions, it is possible to represent independent factors in quantitative form as given in (1). Then these factors can be thought of as having a functional relationship with response as follows:

$$Y=\phi(X_1,X_2,X_3,X_4,X_5,\ldots\ldots\ldots..,X_n)\pm\varepsilon \qquad (1)$$

This represents the relation between response Y and X1, X2,…, X_n of n quantitative factors. The function Φ is called response surface or response function. The residual 'ε 'measure the experimental errors. For a given set of input variables, a characteristic surface is responded. Mathematical form of Φ is not known then it can be approximated satisfactorily within the experimental region by a polynomial. Upper the degree of polynomial superior is the correlation but at the same time costs of experimentation become higher.

For the present work, RSM has been applied for developing the mathematical models in the form of multiple regression equations for the etch factor during the PCM process of Inconel 600 material. By using the response surface methodology, the dependent variable is viewed as a surface to which a mathematical model is fitted. For the development of regression equations related to etch factor during the PCM process of Inconel 600 material, the second order response surface has been assumed as:

$$y = c_0 + \sum_{i=1}^{N} c_i x_i + \sum_{i=1}^{N} c_{ii} x_i^2 + \sum_{i,j=1;\, j<i}^{N} c_{ij} x_i x_j$$

This assumed surface Y contains squared, linear and cross product terms of input variables xi's. In order to determine the regression coefficients, various experimental design techniques are available.

B. Response surface design

The present article gives the application of the response surface methodology. The scheme of carrying out experiments was selected and the experiments were conducted to investigate the effect of process parameters on the etch factor during the PCM process of Inconel 600 material. The experimental results will be discussed subsequently in the following sections. The chosen input variables were varied up to three levels and face-centered central composite design was adopted to design the experiments as shown in Fig. 1. Response Surface Methodology was used to develop second order regression equation relating response characteristics and process variables.

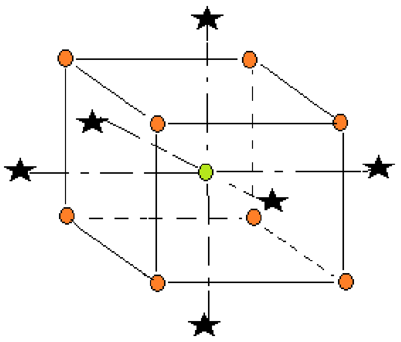


Fig. 1. Face centered central composite design for k=3

The present investigation studied the results of the effects of Concentration, Time and Temperature on the undercut during the PCM process of Inconel 600 material. Input parameters and their levels are shown in Table II. Table III shows experimental design matrix with coded and un-coded values of Face centered composite design

TABLE II. INPUT PARAMETERS AND THEIR LEVELS

Input Parameter	**Level 1**	**Level 2**	**Level 3**
Concentration (gm/lit)	300	500	700
Time (min)	30	50	70
Temperature (^{0}C)	55	60	65

TABLE III. EXPERIMENTAL LAYOUT PLAN FOR FACE CENTERED COMPOSITE DESIGN OF EXPERIMENTS

Sr. No.	**Coded Values**			**Un-coded Values**		
	A	***B***	***C***	***Conc. (gm/lit)***	***Time (min)***	***Temp (^{0}C)***
1	-1	-1	-1	300	30	55
2	1	-1	-1	700	30	55
3	-1	1	-1	300	70	55
4	1	1	-1	700	70	55
5	-1	-1	1	300	30	65
6	1	-1	1	700	30	65
7	-1	1	1	300	70	65
8	1	1	1	700	70	65
9	-1	0	0	300	50	60
10	1	0	0	700	50	60
11	0	-1	0	500	30	60
12	0	1	0	500	70	60
13	0	0	-1	500	50	55
14	0	0	1	500	50	65
15	0	0	0	500	50	60
16	0	0	0	500	50	60
17	0	0	0	500	50	60
18	0	0	0	500	50	60
19	0	0	0	500	50	60
20	0	0	0	500	50	60

III. EXPERIMENTAL PROCEDURE

When starting photo chemical machining thin metal sheets are cleaned and laminated with a very thin layer of photo-resist film. Using a CAD created Photo-Tool image is printed on one side of the sheet using calibrated ultra violet lights. Printed sheets are developed in chemical solutions to expose the metal surface in the printed areas. The sheets are then etched with Fecl3 solution so that the exposed metal dissolves away. The photo-resist film used in the photochemical machining process protects the material in the areas that were not washed away in the developing process. After chemical etching process the remaining photo-resist film is stripped in alkaline solution. Process parameters at each stage are controlled to obtain the desired dimensions and finish. The thickness of specimen was 0.3 mm and cut at 20mmX20mm dimension. FeCl3 chemical etchant was prepared. The amount of etchant for each experiment was 100ml. Single sided chemical etching was conducted. The measurements of Undercut and depth of etch were carried out by Tool maker's Microscope (± 0.001 mm) and digital micrometer ((± 0.001 mm) respectively. The main limitation of PCM is to be found in the characteristics of isotropic etching whereby the etchant will attack not only downwards into the metal but also sideways beneath the resist stencil layer it is also known as undercut. Fig. 2, shows etch factor for PCM process. For accurate etching we have to minimize undercut and increase etch factor. Fig. 3, shows schematic representation of photochemical machining experimental setup & Etch factor where

Etch factor = Depth of etch (D) /Undercut where
Undercut= (B-A)/2

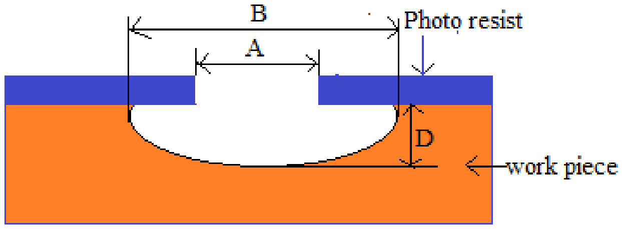
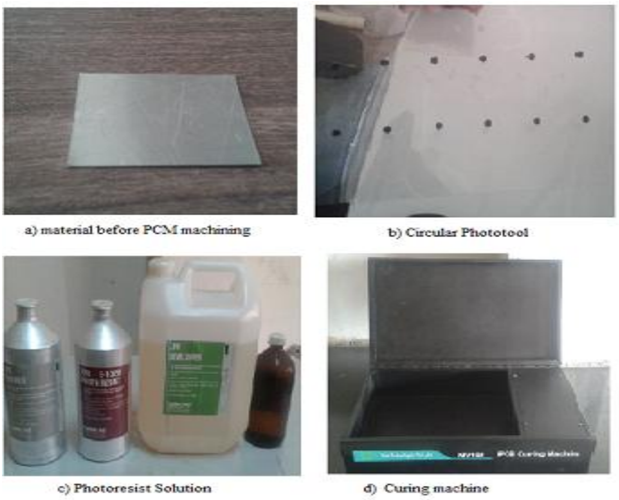
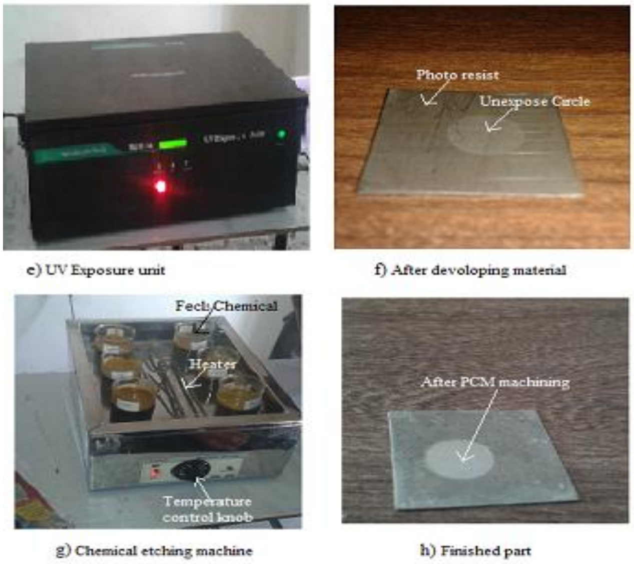


Fig. 2. Etch factor of PCM


a) material before PCM machining
b) Circular Phototool
c) Photoresist Solution
d) Curing machine



e) UV Exposure unit
f) After devoloping material
g) Chemical etching machine
h) Finished part

Experimental setup

IV. RESULT AND DISCUSSION

A. *Analysis of variance*

The summary of analysis of variance (ANOVA) is shown in Table IV. It is observed that the factor etch factor has a significant effect at 95% confidence interval as evident from ANOVA.

The 2FI Model having F-value of 4.83 implies the model is significant. There is only a 0.84% chance that an F-value this large could occur due to noise. Values of "Prob> F" less than 0.05 specify model terms are significant. In this case C, AB, AC are significant model terms. Values larger than 0.1 specify the model terms are not significant.

TABLE IV. ANALYSIS OF VARIANCE TABLE FOR ETCH FACTOR

Source	**ss**	**df**	**MS**	**F-value**	**p-value Prob.>F**	
Model	65.55	6	10.92	4.83	0.0084	significant
A-Conc.	8.20	1	8.20	3.62	0.0793	
B-Time	8.09	1	8.09	3.58	0.0811	
C-Temp.	17.38	1	17.38	7.68	0.0159	
AB	11.82	1	11.82	5.22	0.0397	
AC	11.38	1	11.38	5.03	0.0429	
BC	8.68	1	8.68	3.84	0.0720	
Residual	29.41	13	2.26			
Lack of Fit	29.41	8	3.68			
Pure Error	0.000	5	0.000			
Cor Total	94.96	19				

Three-dimensional plots were drawn by using the response surface methodology to investigate the effects of the time, concentration and temperature factors on the etch factor during the PCM process of Inconel 600 material. Fig. 4, indicates etch factor along with temperature and concentration and it is clear that with increasing etchant temperature and concentration etch factor increases. Fig. 5, indicates etch factor increases as etchant temperature increases but decreases with etching time. Fig. 6, indicates etch factor increases as concentration increases but decreases with etching time.

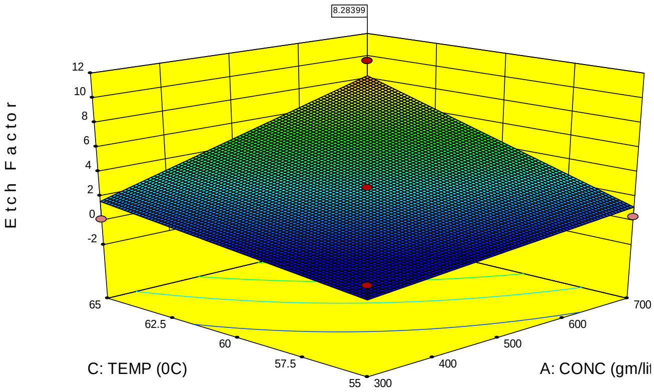


Fig. 4. The combined effect of temperature and concentration on etch factor

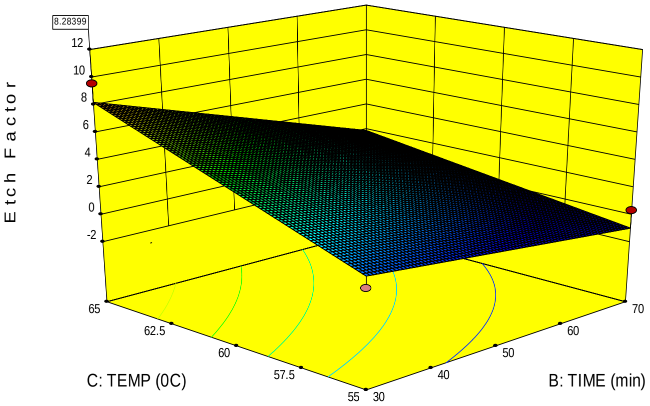


Fig. 5. The combined effect of time & Temperature on etch factor

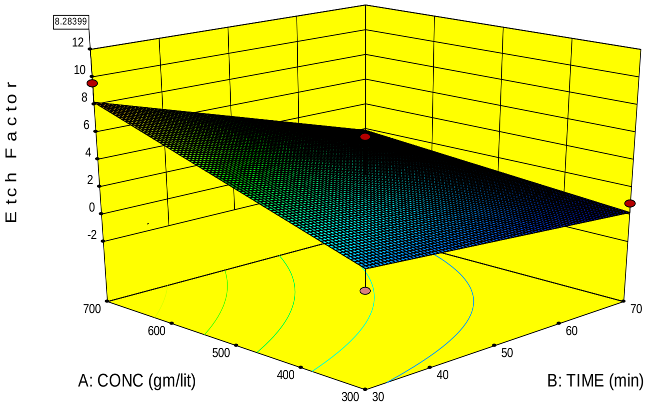


Fig. 6. The combined effect of time and concentration on Etch factor

B. *Main effects plots*

Main effects plots are drawn showing the effect of various input parameters on etch factor. From the above mentioned main effects plot for etch factor shown in Fig. 7, it is clear that etchant concentration and Temperature are the most affecting parameter for etch factor. While etching time is less significant for MRR.

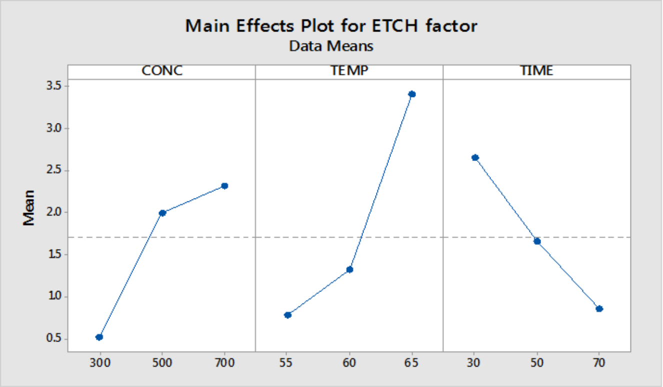


Fig. 7. Main effect plots

C. *Development of Regression Model Equation*

Face centered composite design was used to develop correlation between the etch factor during the PCM process of Inconel 600 material to concentration, temperature and time. Experimental error was determined by using 20 experiments at the center point. Associate to the sequential model sum of squares, the models were selected based on the F-value. The independent input variables of the model were significant so that the models were not aliased and the 2FI model was taken as proposed by the software Design Expert (DE9). Based on 2FI model, experiments were planned to obtain 20 trials plus a star configuration (0,±1) and their duplicates at the center point. Table 4 shows design of this experiment jointly with the experimental results. The maximum etch factor was found to be 8.28. Regression analysis was performed to fit the response function of etch factor. The mathematical model expressed by (2), where the variables fill their coded values, represents the etch factor (Y) as a function of concentration (A), time (B) and Temperature (C).

$$\text{Etch factor} = 1.71+0.91A-0.90B+1.32C-1.22AB+1.19AC-1.04BC \qquad (2)$$

D. Optimization using desirability approach

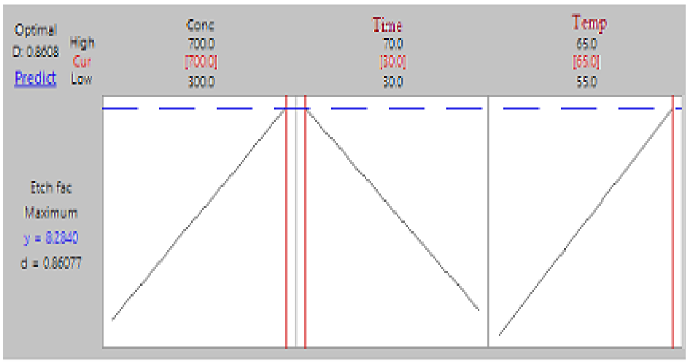


Fig. 8. Optimization graph

Fig. 8, shows optimization plot for etch factor. The ultimate objective of our work was to maximize the etch factor. Desirability approach was been used for finding out the optimum values of the variables in order to get the maximum value of etch factor. From the graph it is clear that highest value 8.2840 is obtained for the following combination of the variables:
Etchant concentration = 700 gm/lit
Etching time = 30 min
Etchant temperature = 65^{0}C
The above results were obtained with the composite Desirability of 0.86077.

V. CONCLUSION

The response surface methodology based on three variables, face centered composite design was used to determine the effect of time (ranging 30-70 min), concentrations of etchant (ranging 300– 700 gm/lit) and temperature (55-65 ^{0}C) on the etch factor during the PCM process of Inconel 600 material. The regression analysis, statistical significance and response surface were applied using Design Expert Software for forecasting the responses in all experimental areas. Models were developed to correlate variables to the responses. Through analysis of the response surfaces derived from the models, role of etchant concentration and etchant temperature were found to have the most significant effect on etch factor. Process optimization was carried out and the experimental values acquired for the etch factor during the PCM process of Inconel 600 materials are found to agree satisfactorily with the values predicted by the models. Since experimentally obtained and model predicted values are residual which shows the effectiveness of model, based on the designed experiment. The optimal predicted etch factor 8.2840 of Inconel 600 was obtained as Ferric chloride concentration, time and temperature of etching and these were found to be 700gm/lit, 30 min and 65 ^{0}C respectively.

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