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RESPONSE SURFACE OPTIMIZATION FOR SS316L MATERIAL IN PHOTOCHEMICAL MACHINING

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Abstract - *Photo chemical machining is an engineering production technique for the manufacture of burr free and stress free flat metal components by selective chemical etching through a photographically produced mask.*

From the survey of above research papers, it is found that many people have worked for the optimization of process parameters of photochemical machining. They have considered the process parameters like machining time, temperature and concentration of etchant. The responses considered were either material removal rate or surface roughness or undercut. They have found the optimum values for either one or two responses.

But in my project work I have found common optimum values for material removal rate, surface roughness and undercut. The process parameters like machining time, temperature and concentration of etchant are considered.

The material selected for the experimentation is stainless steel (SS316L) and etchant selected is FECL3 (Ferric chloride). Response surface methodology is used for the optimization of process parameters.

Key Words: **Chemical etching1, machining time2, temperature3, concentration of etchant4, surface roughness5, material removal rate6, FECL3 (Ferric chloride)7,**

1. INTRODUCTION

Photo chemical machining is an engineering production technique for the manufacture of burr free and stress free flat metal components by selective chemical etching through a photographically produced mask.

The development of knowledge of acid attack upon metals is not new its origins lie in antiquity. Legend tells that the ancient Greeks had discovered a fluid, which is referred to as liquid fire that attacked both inorganic and organic materials. However as this was the Bronze Age it is unlikely that they possessed the technology to manufacture such an acidic chemical. The ancient Egyptians etched copper jewelry with citric acid as long ago as 2500BC. The Hohokam people, of what is now Arizona, etched snail shell jewelers with fermented cactus juice around 1000BC. The earliest reference to this process describes an etchant made from common salt, vinegar and charcoal acting through a hand scribed mask of linseed oil paint. Decorative patterns were also etched into swords by means of scribed wax resist. These techniques were adapted and improved by etchers operating in close co-operation with armourers until, by the seventeenth century; armour had become wholly ceremonial and great works of etched art.

The main advantage is the lack of burrs. During the mid seventeenth century etching was used for the indelible calibration of measuring instruments and scales such as artillery gunners conversion table etched around 1650. Two developments within the space of forty years in photography laid the foundations for the photo resists we use today. In 1782 John Senebier of Geneva investigated the property of certain resins to become insoluble in turpentine after exposure to sunlight. Inspired by this, Joseph Nicephore Niepce resurrected an ancient Egyptian embalming technique that involved the use of what is now known as Syrian asphalt. This hardens after exposure to several hours of sunlight, into an acid resistant film. However, it took constant experimentation until this development was a success in 1822. The result was a resist that could be photo-polymerized in the exposed areas whereas the unexposed areas could be developed off in a solution of oil of lavender in turpentine. The age of photo etching had arrived. By 1925 the huge daily newspaper industry made large-scale use of printing plates etched in nitric acid solution. By 1927 the use of chemical milling through a rubberized paint mask, which was hand cut around a template, was being used as an engineering production tool.

1.1 Photochemical Machining Process

Photochemical machining (PCM) is non-conventional machining processes. It employs chemical etching through a photo resist stencil as the method of material removal over selected areas.

First, the material is cleaned to remove the oil, grease, dust, rust or any substance from the surface of material that would provide good adhesion of the photo resist. The most widely used cleaning method is chemical method due to less damages occurred comparing to mechanical cleaning method. Coating with photo resist (dry or wet) is the next stage of PCM. Then the expose of the prepared photo tool is carried out with UV light. Developing stage is used to remove unexposed areas of the photo resist that is carried out by various chemical liquids. Then the chemical etching operation is carried out in spray etchant machine. The selected etchant for work piece material is heated up to 50-55 °C depending on the spray machine allowance and etchant is sprayed from nozzles onto the work piece surface. Removal of photo resist film from etched work piece surface is the last stage of the PCM.

1.2 Metal selection

The metal is cold rolled, high precision, especially in relation to the tolerancing of the thickness of gauge. It also has a superior surface finish to standard commercial grade material. Although there are slight variations between metal types, the general rule for thickness tolerance is ± 8% material thickness. It is very rare that precision strip deviates to this tolerance band and the normal deviation in the 'as rolled condition' is within ± 4% of metal thickness. Surface finish varies according to metal type and condition. The raw material for processing is received in three forms; flat sheets, coil and specific size cut blanks. Sheet material, which is usually supplied in 600mm x1200mm or 2000mm x 1000 mm, is confined to thicker gauge copper and brass (including and above 0.4mm), aluminium and annealed stainless steel (0.6mm upwards).

1.3 Photo tooling

Modern technology now allows an image of the profile of the flat component to be transferred directly to the photographic film that is to be used as the photo tool by way of a light pen plotter.

1.4 Protective coatings (photo resist)

A photo resist made by synthetic polymer such as HTP Hitch Photopolymer AG, casein. This resist showed its general suitability for this process. It has to be developed further to increase its stability while machining. The most photo resist are having good adhesion to substrate for wet chemical processes or high thermal stability for dry chemical etching.

1.5 Etchant

Preparation of etchant for work piece material should be carried out according to the corrosive resistance of the material. High corrosion resistance materials require high level of etchant concentration.

Ferric chloride ($FeCl3$) is the most widely used etchant in the PCM application for etching all iron-based alloys as well as nickel, copper and its alloys, aluminium and its alloys, etc. cupric chloride ($CuCl2$) is generally applied for copper and copper based alloys in electronics industry. The importance of this etchant is regeneration of waste etchant and recovery of etched copper could be carried out simultaneously. Alkaline etchants are introduced copper in the fabrication of electronic components such as printed circuit board etc. These etchant are expensive and generally selected for high volume of copper etching. Corrosion resistance materials such as titanium, silicon and glass could be etched by diluted hydrofluoric acid. This etchant is very hazardous; hence major precautions must be taken for etching of these types of materials.

2. OBJECTIVE OF PRESENT WORK:-

From the survey of above research papers, it is found that many people have worked for the optimization of process parameters of photochemical machining. They have considered the process parameters like machining time, temperature and concentration of etchant. The responses considered were either material removal rate or surface roughness or undercut. They have found the optimum values for either one or two responses.

But in my project work I have found common optimum values for material removal rate, surface roughness and undercut. The process parameters like machining time, temperature and concentration of etchant are considered. The material selected for the experimentation is stainless steel (SS316L) and etchant selected is FECL3 (Ferric chloride). Response surface methodology is used for the optimization of process parameters.

I have framed the following objectives for my project work.

1. To study the effect of process parameters on responses.
2. To study the influence of process parameters on responses by analysis of variance.
3. To prepare mathematical models for the responses.
4. To find out the common optimum values of process parameters for the responses.

3. DESIGN OF EXPERIMENT:

Design of Experiments (DOE) refers to planning, designing and analyzing an experiment so that valid and objective conclusions can be drawn effectively and efficiently. In performing a designed experiment, changes are made to the input variables and the corresponding changes in the output variables are observed. The input variables are called resources and the output variables are called response. Resources may be either qualitative or quantitative. Qualitative factors are discrete in nature (such as type of material, color of sample). Each factor can take several values during the experiment. Each such value of the factor is called a level. A trial or run is a certain combination of factor levels whose effect on the output is of interest.

Before starting the experiments, several things needed to be done in order to run the experiments smoothly and accurately. Basically, there are five general steps that had been set so that the utilization of DOE tools can be hold efficiently. The five general steps are -

- Plan the experiment
- Design the experiment

- Conducting the experiment
- Analyze the data from the experiment
- Confirmation of experiment

3.1 Selection Of Process Parameters:

The process parameters like machining time, temperature and concentration of etchant are considered.

Time (min)	Temperature (0C)	Concentration (gm/litre)
20-60	40-60	600-900

Table 3.1: Range of parameters

After doing the trials on the setup the machining time selected is in the range of 10 to 40 minutes. The temperature selected is in the range of 45 to 60 degrees and the concentration of the etchant selected is 600 to 900 gm/litre.

3.1.1 Input factors with their levels:

Factor/level	Notation	-1	0	+1
Time (min)	t	20	40	60
Temperature (0C)	T	40	50	60
Concentration (gm/lit)	C	600	750	900

Table 3.2: Input factors with their levels.

3.2 Responses (Output parameters):

The responses considered for optimization are given below.

1. Material removal rate (MRR)
2. Surface roughness (Ra)
3. Undercut (Uc).

3.3 Material selection:

The material selected for the experimentation is stainless steel (SS316L). Type 316 is an austenitic chromium nickel stainless steel containing molybdenum. This addition increases general corrosion resistance, improves resistance to pitting from chloride ion solutions, and provides increased strength at elevated temperatures. Properties are similar to those of Type 304 except that this alloy is somewhat stronger at elevated temperatures.

Corrosion resistance is improved, particularly against sulfuric, hydrochloric, acetic, formic and tartaric acids; acid sulfates and alkaline chlorides.

Type 316L is an extra-low carbon version of Type 316 that minimizes harmful carbide precipitation due to welding. Typical uses include exhaust manifolds, furnace parts, heat exchangers, jet engine parts, pharmaceutical and photographic equipment, valve and pump trim, chemical equipment, digesters, tanks, evaporators, pulp, paper and textile processing equipment, parts exposed to marine atmospheres and tubing. Type 316L is used extensively for weldments where its immunity to carbide precipitation due to welding assures optimum corrosion resistance.

3.3.1 Composition:

Content	**Percentage**
Carbon	0.03 max
Manganese	2.00 max.
Phosphorus	0.045 max
Sulphur	0.03 max.
Silicon	0.75 max
Chromium	16.00 - 18.00
Nickel	10.00 - 14.00
Molybdenum	2.00 - 3.00
Nitrogen	0.10 max
Iron	Balance

Table 3.3: Composition of material SS316L

3.3.2 Mechanical properties of material SS316L:

Typical Room Temperature Properties are given below.

UTS (Mpa)	558
0.2% YS (Mpa)	290
Elongation % in mm	50
Hardness Rockwell	B79

Table 3.4: Mechanical properties of material SS316L

3.3.3 Physical properties of material SS316L:

1. Density of the material is 7.99 g/cm3.
2. Electrical Resistivity, microhm-in (microhm-cm) 68°F (20°C) – 29.4 (74).
3. Specific Heat, BTU/lb/°F (kJ/kg•K) 32 - 212°F (0-100°C) – 0.12 (0.50)
4. Thermal Conductivity, BTU/hr/ft2/ft/°F (W/m•K) at 212°F (100°C) – 9.4 (16.2) at 32°F (500°C) – 12.4 (21.4).
5. Modulus of Elasticity, ksi (MPa) 28.0 x 103 (193 x 103) in tension 11.2 x 103 (77 x 103) in torsion

6. Mean Coefficient of Thermal Expansion, in/in/°F (µm/m•K)

32 - 1212°F (0 - 100°C) – 8.9 x 10-6(16.0)
32 - 1600°F (0 - 315°C) – 9.0 x 10-6(16.2)
32 - 1000°F (0 - 538°C) – 9.7 x 10-6(17.5)
32 - 1200°F (0 - 649°C) –10.3 x 10-6(18.5)
32 - 1500°F (0 - 871°C) –11.1 x 10-6(19.9)

7. Magnetic Permeability, H = 200 Oersteds, Annealed – 1.02 max.
8. Melting Range, °F (°C) – 2500 – 2550 (1371 - 1399)

3.4 Etchant selection:

The most commonly used etchants in photochemical machining for the stainless materials are ferric chloride etchant (FECL3) and cuprous chloride etchant (CuCl3). Ferric chloride (FeCl3) is the most commonly used etchant for photochemical machining (PCM) but there is a great variety in the grades of the commercial product.

In an ideal world, to maintain a constant rate of etching and hence control of part dimensions dependent on etch time, the etchant composition would be constant. Unfortunately, in the real world, the etchant composition changes continuously. As an n-valent metal (M) is dissolved into solution, etchant is consumed and the by-products of ferrous chloride (FeCl2) and metal chlorides (MCln) are generated, i.e.
nFeCl3 + M → nFeCl2 + MCln.
The etchant selected for the experimentation is ferric chloride etchant (FECL3).

3.5 Methodology:

Response surface methodology (RSM) is a collection of mathematical and statistical techniques that are useful for modeling and analysis of problems in which output or response influenced by several variables and the goal is to find the correlation between the response and the variables. It can be used for optimizing the response. It is an empirical modelization technique devoted to the evaluation of relations existing between a group of controlled experimental factors and the observed results of one or more selected criteria. A prior knowledge of the studied process is thus necessary to achieve a realistic model. We selected only three experimental factors capable of influencing the studied process yield: three factors machining time, temperature and concentration of etchant.

The first step of RSM is to define the limits of the experimental domain to be explored. These limits are made as wide as possible to obtain a clear response from the model.

In the next step, the planning to accomplish the experiments by means of response surface methodology (RSM) using a Central Composite Design (CCD) with three variables.

The central composite design used since it gives a comparatively accurate prediction of all response variable averages related to quantities measured during experimentation .CCD offers the advantage that certain level adjustments are allowed and can be used in two-step chronological response surface methods. In these methods, there is a possibility that the experiments will stop with fairly few runs and decide that the prediction model is satisfactory

The mathematical model is then developed that illustrate the relationship between the process variable and response. The behavior of the system is explained by the following empirical second-order polynomial model.

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_{i=1}^{k-1} \sum_{j=2}^{k} \beta_{ij} X_i X_j$$

3.6 Experimental Setup :

The experimental Setup consists of the following:

1. Heater- used for increasing the temperature of etchant.
2. Ultra violet exposure unit- used for developing the image for makant.
3. Photo resist and thinner.
4. Weighing balance.
5. Work piece holder.
6. Surface tester.

4. EXPERIMENTAL RESULTS:

The density of the material at room temperature is 7.99 g/cm3 (0.00799 g/mm3).

Run order	Time (min)	Temperature (^{0}C)	Concentration (gm/lit)	Initial weight (W_1) gm	Final weight (W_2) gm	MRR= $\frac{(W1-W2)}{\rho X t}$ (mm^3/min)	Ra µm	Undercut(mm)
1	20	40	600	5.52	5.50	0.120	0.64	0.158
2	40	50	750	6.29	6.20	0.280	0.82	0.186
3	40	50	750	6.32	6.23	0.275	0.79	0.180
4	60	50	750	6.12	5.98	0.290	1.00	0.200
5	20	50	750	5.73	5.70	0.163	0.59	0.134
6	20	40	900	5.94	5.90	0.248	0.77	0.117
7	20	60	900	5.84	5.78	0.351	0.74	0.157
8	60	40	900	6.04	5.85	0.388	1.00	0.168
9	60	60	900	5.84	5.65	0.396	0.84	0.256
10	40	60	750	5.76	5.65	0.350	0.74	0.235
11	40	50	600	5.82	5.75	0.230	0.80	0.224
12	40	50	750	6.05	5.96	0.280	0.86	0.188
13	40	40	750	5.67	5.60	0.214	1.01	0.116
14	20	60	600	5.90	5.88	0.139	0.54	0.163
15	60	40	600	5.86	5.74	0.242	0.85	0.196
16	40	50	900	6.00	5.90	0.328	0.77	0.155
17	40	50	750	5.93	5.84	0.284	0.87	0.183
18	40	50	750	5.85	5.76	0.285	0.83	0.200
19	40	50	750	5.94	5.85	0.280	0.80	0.190
20	60	60	600	5.94	5.80	0.292	0.65	0.230

5. CONCLUSIONS:

In the present study, the common optimum values of process parameters for MRR, surface roughness (Ra) and undercut for material stainless steel (SS316L) are obtained using ferric chloride etchant (FECL3) in photochemical machining (PCM).

The experiments are conducted under various parameters setting of Machining Time (min), Temperature of etchant in (0C) and Concentration (gm/lit).

Design of experiment by Response surface methodology is used for experimentation. Response surface method is multi response optimization method. Therefore we can get common optimum values for three responses.

MINITAB software is used for DOE and analysis of the experimental result and the response was validated experimentally.

The following conclusions can be drawn from the analysis:

1. Selected parameters i.e. Etching time, Temperature and Concentration show their influence in machining.
2. The common optimum values of process parameters for MRR, surface roughness (Ra) and undercut for material stainless steel (SS316L) are:

Etching time = 20 min.
Temperature = 600C
Concentration = 900 gm/lit.

3. While machining the material stainless steel (SS316L) using ferric chloride etchant (FECL3) in photochemical machining (PCM), the industrialist can directly use these

optimum values so that the material removal rate will be maximum and surface roughness and undercut will be minimum.

4. The material removal in PCM process is rather low, where the total volume of a cavity has to be removed. If the PCM is operated at the optimum setting of parameters then this drawback can be minimized.

5.1 FUTURE SCOPE:

1) In optimizing the problem in this project, three objective functions with three controllable machining parameters are considered. However, metal removal rate is complex and many other parameters influence the end results.
2) Parameters like work piece material, type of etchant, type of maskant used; etc can influence the metal removal rate and surface roughness and undercut.
3) These parameters can also be considered in future work. The mathematical tools available for solving the optimization problems are many. It may be worthwhile to apply the various tools and assess their utility.

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