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# A STUDY ON ELECTROCHEMICAL MICROMACHINING OF SUPER DUPLEX STAINLESS STEEL FOR BIOMEDICAL FILTERS

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### ABSTRACT

In recent days, industries are looking for miniature components which can perform complex functions in the areas of electronics, biomedical and nuclear applications. Electrochemical micromachining (ECMM) is an emerging nonconventional technology for producing micro/meso scale components. The micromachining of super duplex stainless steel (SDSS) is generally very difficult, less accurate and time consuming when using many of the nonconventional machining methods. By the use of ECMM the above limitations can be overcome. In this paper, an experimental investigation is carried out to identify the optimal machining parameters for machining SDSS using ECMM. Experiments are conducted based on Taguchi L<sub>18</sub> orthogonal array to find the influencing machining parameters on Material Removal Rate (MRR). Statistical analysis of variance is performed to determine the percentage contribution of individual process parameters on MRR. Among the various factors investigated, duty cycle is found to be the most significant factor and contributes about 42% to the MRR. As the duty cycle increases, the pulse on time also increases which contributes to more MRR. The parameters such as current 0.6 amps, voltage 9V, frequency 30 Hz, electrolyte concentration 0.5 mol/lit and duty cycle 66.66% produce maximum MRR in the micromachining of SDSS. This research helps to understand the selection of the machining parameters for ECMM of SDSS.

Keywords: electrochemical micromachining, super duplex stainless steel, material removal rate, Taguchi L<sub>18</sub> orthogonal array, machining parameters.

### 1. INTRODUCTION

Now-a-days, micro manufacturing techniques find wider application in various industries like electronic, semi-conductors, medicine, aerospace, naval and ultraprecision machines. The shaping of parts with dimensions in the range of 5 to 500 µm and production of parts with high surface finish have a lot of applications in industries [1]. Machining materials on micro and sub-micro scale is considered a key technology for miniaturizing mechanical parts and complete machines. Micro-machining refers to small amount of material removal that ranges from 1-999µm [2]. Machining of hard and difficult machine alloys with conventional methods results in subsurface damage to the work piece and the machining tools. Many of the nonconventional machining processes can machine these alloys without the above limitation. All the nonconventional techniques remove material by utilizing chemical, thermal and electrical energy either independently or in a combination[3]. However, non conventional techniques also suffer from the limitations such as restricted materials choice, inability to produce complex profiles and huge investment cost [4].

Electrochemical machining (ECM) is one of the nonconventional machining processes. It finds wider application because it produces good quality surfaces without affecting the metallurgical properties of the work material. This process does not change surface integrity of the material and does not induce any deformation because no heat is generated while machining. In an ECM process, the work piece is connected to anode and the tool is connected to cathode and both are placed inside an electrolyte bath with a small gap between them known as Inter Electrode Gap (IEG). On the application of adequate electrical energy, positive metal ions leave the work piece. The positive ions react with the negative ions in the electrolyte forming hydroxides. The electrolyte is constantly flushed to remove the hydroxides, which otherwise, would grow to create a short circuit between the electrodes [5].

The fabrication of microstructures by ECM process is known as electrochemical micromachining (ECMM). ECMM is the key technology for the, electronics, optics, biomedical, automotive, avionics and ultra precision machinery industries [6]. The ECMM appears to be very promising as a future micromachining technique, since it offers several advantages such as higher machining rate, better precision, control and capability to machine wide range of materials [2]. The ECMM process produces a stress free surface using precise process control along with higher Material Removal Rate (MRR). Hard metals can be shaped with ECM and ECMM process using electrolysis principle. In this process, the MRR is not affected by the hardness of the work piece. The ECMM is still in its initial stages of development and a lot of research needs to be done to improve MRR, surface quality and accuracy by optimizing the various process parameters [7].

The ECMM micro hole making process satisfies the quality requirements with respect to their geometrical characteristics like over-cut, taper, aspect ratio or metallurgical characteristics like heat affected zone and micro-cracking [8]. This process produces no tool wear, having shorter machining time and cost effective [9]. The good surface finish obtained from this process makes it



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more attractive for drilling micro holes for the components exposed to high temperature [7].

The Super Duplex Stainless Steel (SDSS) is a high strength non corrosive metal and is difficult to machine using conventional processes due to the dual phase microstructure. This alloy can be easily machined by ECMM because in this process material is removed by anodic dissolution and not by metal shearing. This study aims at selection of optimum ECMM process parameters for machining of SDSS. The most important parameters implicated in ECMM on MRR such as electrolyte concentration, machining voltage, machining current, duty cycle and frequency are chosen as a process parameters in this study. The MRR during the machining of micro hole on a thin work piece (0.216 mm) is used for the experimentation. Taguchi technique is used to identify the optimum combination levels of the parameters to maximize the MRR. Taguchi  $L_{18}$  orthogonal array is used for designing the experiential procedure. The ANOVA is used to identify the contribution of individual process parameters.

### 2. PROCESS PARAMETERS

In this section the process parameters and the material under study are discussed.

### 2.1 Process parameters

During ECMM, metal from the anode is removed atom by atom known as anodic dissolution. The ionized atoms of work piece material are then positively charged and are attracted away from the work piece by an electric field. The shape of the work piece due to anodic dissolution will be that of mirror image of the cathodic tool. In ECMM the material removal is governed by Faradays laws of electrolysis [8]. The factors that affect the rate of machining are the type of electrolyte, flow rate of electrolyte, temperature of electrolyte, and its pH value [10]. The geometry, condition, and accuracy of the machined surfaces are depending on the electrolyte salt type and concentration, machining gap, pulse power supply setting, flow velocity and flow profile [11].

### 2.1.1 Electrolyte

The electrolyte in the ECMM assists the dissolution of work piece material, carries electricity between the electrodes, carries away the removed material and heat generated, and maintains constant temperature in the machining area. The most commonly used electrolytes are Sodium Chloride and Sodium Nitrate. By using an electrolyte with a lower concentration, inter electrode gap could be reduced resulting in improved accuracy [12]. The amount of material removed at the work piece depends on the properties of the electrolyte and its flow rate [4]. The electrolyte conductivity in the gap between the cathode and the anode is dependent on starting electrode distance, concentration of salt in the solution, local hydroxide concentration in electrolyte, bulk and local temperature, electrolyte flow rate, and the velocity of electrolyte [13]. The high flow rate of electrolyte causes the tool erosion.

#### 2.1.2 Pulsed current

The amount of material removed is proportional to the quantity of current passed. Instead of continuous current, the use of pulsed current in ECMM facilitates prompt removal of hydroxides formed between electrodes and maintenance of IEG for better MRR and surface finish. The use of pulsed current gives good control over the etching process [14]. It is observed that the MRR is increased for increased pulse ON time indicating that the MRR is higher at lower frequencies [5].

### 2.1.3 Voltage

As machining voltage is increased, the machining rate is increased. The machining rate reaches its maximum value at a particular voltage and decreased because electrode surface is gradually covered by bubbles generated at increased voltage [1]. It is observed that a power supply which maintains a constant voltage and current throughout the machining process is the most effective for electrochemical machining [15].

### 2.1.4 Duty cycle and frequency

In a pulsed current, duty cycle is the ratio between power ON time and the total ON and OFF time [3].

i.e., Duty cycle = 
$$T_{on}$$
 (milli sec.)/ $T_{total}$  (milli sec.) (1)

The number of duty cycles used per unit time is called frequency (frequency  $=1/T_{total}$ ). The duty cycle and frequency of the pulsed power supply affect the MRR [11]. During the experiments, pulse rectifier is switched on and the desired value of machining voltage, current, duty cycle and frequency are set before commencing the machining.

### 2.2 Material

The SDSS may be defined as a group of steels having a two phase ferrite-austenite microstructure after heat treatment and water quenching. The high chromium and molybdenum content of super duplex makes it extremely resistant to uniform corrosion by organic acids like formic and acetic acids. SDSS also provides excellent resistance to inorganic acids, especially those containing chlorides. These alloys have been widely used as structural materials for chemical plants, especially those engaged in phosphoric acid production, in the hydrometallurgical industries and as materials for offshore applications. The SDSS is more difficult to machine than the 300-series austenitic stainless steels of similar corrosion resistance. SDSS finds wider applications in miniature components like bio filters used in medical and navel applications.

# **3. EXPERIMENTAL PROCEDURE**

A suitable micro-tool vibration system has been developed, which consists of tool holding unit, work holding platform etc. The developed system is used successfully to control MRR to meet the machining requirements.



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# 3.1 Set-up of ECMM

The machining set-up of ECMM consists of various sub components as shown in Figure-1. The sub systems are: work holding platform; tool feeding device; control system; electrolyte flow system and power supply system.



Figure-1. Set up of ECMM.

Work holding platform consists of two rectangular platforms to hold the work, which are made of acrylic because of its non-corrosive property. The work is placed in-between two detachable plates which are fastened together by means of screws. The work holding platform is immersed inside the electrolyte tank during machining. The machining chamber filled with electrolyte, rests on the base and the electrolyte re-circulation is carried out in the chamber itself. The chamber is clamped to the base. The feeding device actuated with stepper motor moves the tool by receiving the signals from the control unit.

The circumference of the tool is insulated by coating a thin film of wax and paint, to avoid stray current effect [6]. In the machining process flow of current is only at the tip, hence the tip of the tool is not insulated or coated. The control system takes care of the entire machining process. It helps in maintaining the electrode gap at a desirable value for obtaining the required shape. Ammeter is used to check the gap maintained between the tool and the work. When the tool electrode gets in contact with the work, the stepper motor moves it upward gradually until the contact is broken. The required feed is controlled using number of passes. The tool moves four microns for a single step rotation of the stepper motor. The tool can be moved according to the amount of inter electrode gap required between the tool and the work piece [16].

A pumping system of electrolyte directs the electrolyte to the working zone with a medium velocity and drives out the material removed. The electrolyte passes through the two nozzles with desired pressure in the machining chamber. The material removed from the work is dissolved into the electrolyte. The electrolyte is filtered before re-circulated to the chamber by the pump. During the machining operation hydrogen gas is evolved at the tool. The gas bubbles formed act as a short circuiting medium creating micro sparks which can erode the tool material. Hence, to avoid the micro spark generation, the electrolyte is pumped out at a moderate pressure and removes the hydrogen gas generated. The different electrolytes that can be used for SDSS machining are HCl or mixture of brine and  $H_2SO_4$ . The magnetic pump used to circulate the electrolyte is having the flow rate of 16 -18 lit/min and head of 2.5m. Non continuous pulsed DC supply is used in ECMM. The AC power supply is converted to low voltage pulsed DC power supply using a pulse rectifier. The voltage and current are set digitally according to the need for the machining. A protective fuse is provided to prevent the short circuit phenomena [17].

### 3.2 Experiments design

Taguchi technique [18] is used to design and analyze the experiments. Based on the required quality objective, various parameters such as electrolyte concentration, voltage, current, duty cycle, and frequency factors are selected [19]. The various levels of parameter factors are determined which are on tool material, work material and size hole. The factors and their levels are given in Table-1. Based on the levels used, the total degrees of freedom are calculated and suitable orthogonal array is chosen [10]. The experiments are carried out based on the orthogonal array. Experimental results are obtained and the ANOVA is carried to identify the contribution of individual parameters on MRR. Then signal-to-noise ratio (S/N ratio) is computed to get optimized parameter levels. The optimal parameter level combination is used to proceed with the conformation experiment. The S/N ratio for larger the better is important in this study as the MRR is to be maximized.

Table-1. Factors and their levels.

Factors	Α	В	С	D	Е
Level 1	0.40	8	0.6	33.33	30
Level 2	0.45	9	0.8	50	40
Level 3	0.50	10	1.0	66.66	50

A. Electrolyte Concentration (mol/lit), B. Voltage (V),

C. Current (A), D. Duty Cycle (%), E. Frequency (Hz).

# 3.3 Experimentation

In this study, the work piece specimens are made of 50 mm  $\times$  10 mm  $\times$  0.216 mm SDSS 2205 plates. The tool electrode is made up of stainless steel of diameter 380µm. The sidewalls of tool electrode are coated with bonding liquid [14]. The electrolyte used for experimentation is fresh aqueous solution of sodium nitrate having different concentrations. Electrolyte concentrations of 0.4, 0.45 and 0.5 mol/lit have been chosen for experiments.

Variable rectangular DC pulsed supply has been used for experimentation. Current of level 0.6, 0.8 and 1 amp is considered for experiments. Applied machining voltages of 8, 9 and 10 V have been selected for experiments. Duty cycle of 33.33, 50 and 66.66% is applied for experiments. Frequencies of pulsed power



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supply of 30, 40 and 50 Hz have been considered for various experiments. During the experiments, pulse rectifier was switched on and the desired value of voltage, current, duty cycle and frequency is set before machining is commenced.

The Taguchi method is used to determine various experimental combinations of parameters to be performed using  $L_{18}$  orthogonal array. Experiments are performed by setting particular levels of parameters as per  $L_{18}$  orthogonal array. Each of the experiments is repeated for three times to eliminate the random variations. The interactions between the machining parameters are neglected. Therefore, there are ten degrees of freedom owing to the five machining parameters. An  $L_{18}$  orthogonal array with eight columns and eighteen rows is selected as ECMM involving ten degree of

freedom and the next corresponding orthogonal array is  $L_{18}$ . The experimental combinations of machining parameters are as given below in the Table-2.

The unit removal (UR) which is defined as material removed per pulse or material removal per unit time have been measured for set of experiments with various combinations of process parameters. Machining time required for making a hole is measured for each experiment. MRR is obtained by calculating the volume of material removed per unit time. The average MRR calculated from each combination of parameter levels of experiment are tabulated in Table-2. ANOVA is performed to determine dominant factor significantly and it affects the MRR (Table-3). Using "Minitab 15" software S/N response Table is obtained for larger is better performance.

Ex. No.	Α	В	С	D	Е	MRR × 10 <sup>-3</sup>	S/N ratio
1	0.4	8	0.6	33.33	30	0. 9092	-60.83
2	0.4	9	0.8	50.00	40	2.0666	-53.69
3	0.4	10	1.0	66.66	50	2.6119	-51.66
4	0.45	8	0.6	50.00	40	2.5774	-51.78
5	0.45	9	0.8	66.66	50	4.5603	-46.82
6	0.45	10	1.0	33.33	30	2.5698	-51.80
7	0.5	8	0.8	33.33	50	0.8991	-60.92
8	0.5	9	1.0	50.00	30	5.7275	-44.84
9	0.5	10	0.6	66.66	40	7.5404	-42.45
10	0.4	8	1.0	66.66	40	2.0446	-53.79
11	0.4	9	0.6	33.33	50	1.8904	-54.47
12	0.4	10	0.8	50.00	30	2.8575	-50.88
13	0.45	8	0.8	66.66	30	4.3241	-47.28
14	0.45	9	1.0	33.33	40	1.7994	-54.90
15	0.45	10	0.6	50.00	50	4.6340	-46.68
16	0.5	8	1.0	50.00	50	9.2938	-40.63
17	0.5	9	0.6	66.66	30	17.0660	-35.36
18	0.5	10	0.8	33.33	40	2.5572	-51.84

Table-2. Experimental combination of machining parameters.

A. Electrolyte concentration (mol/lit.); B. Machining voltage (volts);

C. Machining current, (amp);

D. Duty cycle (%); E. Frequency (Hz)

The predicted or estimated S/N ratio  $\eta$  [18] can be calculated as,

$$\eta = \eta_m + i = 1 \qquad (2)$$

Where,

 $\eta_m$  = total mean of S/N ratio,

 $q = \text{no. of significant parameters, and } \eta_i = \text{mean of S/N}$  ratio at optimum level.

The mean of the S/N ratio for each level of machining parameters is summarized in S/N response also in Table-2. It indicates the optimized level of parameters for each parameter to obtain higher MRR.

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### 4. RESULTS AND DISCUSSIONS

From the results obtained, it is observed that as greater is the S/N ratio, higher is the MRR. From Table-3 column A is seen, the effect of electrolyte concentration on MRR. At higher concentration large number of positive ions dissolved in machining process results in higher S/N ratio and hence higher MRR. Also MRR increases with higher voltage up to 9V then decreases which is due to other factors in a particular experiment. As duty cycle increases, MRR increases. Duty cycle is more; pulse on time is more hence that more time has been allowed to machine the work piece. During pulse on time only the metal removal takes place. Frequency increases, MRR decreases as pulse on time decreases. The mean of S/N ratio for each level of all the machining parameters are summarized in Table-3 and S/N response graph in Figure-2.

Table-	3. Mean	effective	response	(Larger	the E	3etter).
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Level	А	В	С	D	Ε
1	-54.22	-52.54	-48.59	-55.79	-48.50
2	-49.88	-48.35	-51.91	-48.08	-51.41
3	-46.01	-49.22	-49.60	-46.23	-50.20
Delta	8.21	4.19	3.31	9.57	2.91
Rank	2	3	4	1	5



Figure-2. S/N response graph A. Electrolyte concentration (mol/lit.); B. Machining voltage (volts); C. Machining current, (amp); D. Duty cycle (%); E. Frequency (Hz)

From Table-3, it is observed that, higher the value of delta i.e., differences between higher and lower value of response that parameter affecting the MRR more. Hence duty cycle is affecting MRR more compare to the other factors. The optimum combination of parameters is  $A_3B_2C_1D_3E_1$  for obtaining higher MRR or S/N ratio. From the results obtained it is observed that as greater is the S/N ratio, higher is the MRR.

ANOVA is performed using "Minitab 15" software to determine percentage contribution of machining parameters on improving the MRR. From the results of ANOVA, it is clear that duty cycle is the most effective and significant parameter effective MRR. The frequency influences. Parameter on p-value is less than  $\alpha$  level having standard value 0.05, then corresponding factor is significant. Hence a duty cycle and electrolyte are significant factors. While machining voltage, current and frequency are not the significant factors. Percentage contribution indicates the relative power of factor to reduce variation.

The percentage contribution of machining parameters on the MRR is shown in ANOVA table (Table-4) reveals that duty cycle and frequency have maximum and minimum influence on MRR.

Table-4. Results of ANOVA.

Symbol	Factors	SS	DOF	MS	F	Р	Contribution (%)
А	Electrolyte concentrate-ion	202.5	2	101.2	6.70	0.024	27.51
В	Machining voltage	58.70	2	29.4	1.94	0.213	7.97
С	Machining current	34.62	2	17.31	1.15	0.371	4.71
D	Duty cycle	308.8	2	154.4	10.2	0.008	41.96
Е	Frequency	25.66	2	12.8	0.85	0.467	3.49
Е	Error	105.7	7	15.1			14.36
Т	Total	736.0	17				100.0

A. Electrolyte concentration (mol/lit.); B. Machining voltage (volts);

C. Machining current, (amp); D. Duty cycle (%); E. Frequency (Hz)



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Figure-3.Optical Microscope image of micro hole machined in 9<sup>th</sup> experiment.

Figure-3 shows micro hole obtained from 9<sup>th</sup> experiment. In this experiment, electrolyte concentration and duty cycle are at their high levels and other factors are at their medium levels.

### 4.1 Conformation test

The optimum level of process parameters has been determined by using S/N ratio values. The purpose of conformation test is to validate the conclusions drawn during analysis phase. As optimum parameters obtained from ANOVA is  $A_3B_2C_1D_3E_1$  i.e.,  $17^{th}$  experiment, the same experiment (previous Duty cycle) is repeated and compared. The conformation test results closely match with the  $17^{th}$  experimental results (Table-5). It is observed that the improvement in S/N ratio from the initial combinations to the optimal combination is 41.87%.

 Table-5. Results of initial and conformation tests machining performance.

Optimum level	Initial combination	Prediction	Experimental	
	$A_1B_1C_1D_1E_1$	$A_3B_2C_1D_3E_1\\$	$A_3B_2C_1D_3E_1\\$	
MRR mm <sup>3</sup> /min	0.00090921	0.017660	0.017066	
S/N ratio	-60.8266	-37.5357	-35.3573	

# **5. CONCLUSIONS**

In this work, the effect of electrolyte concentration, machining voltage, machining current, duty cycle and frequency of the ECMM process on MRR during machining of SDSS is studied. The process parameters such as micro-tool feed rate, electrolyte flow and pulse period and pulse on-off ratio have been successfully maintained at the desired values during all the experiments. Experimental study is successfully conducted on the developed ECMM system. Taguchi  $L_{18}$  orthogonal array is used for the experimental results. From the ANOVA results, it is found that the duty cycle is the most significant on MRR. The MRR increases with increase in the duty cycle and the frequency least affects the MRR. In

the present study, the contributions of electrolyte concentration, voltage and current follow the duty cycle. It is observed from this research work, the Taguchi method is a systematic and efficient approach for the selection of ECMM parameters in SDSS machining to obtain maximum MRR.

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