Study and evaluation of process parameter on Nimonic 75 alloy by Electrochemical micromachining

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Abstract. Electrochemical micromachining (EMM) is an effective technique for creating micro features in difficult to cut materials like super alloys. Nimonic 75 a super alloy is used to fabricate the aero combustor parts as it can withstand hot gases during the gas turbine operations. In EMM process, Nimonic 75 alloy of thickness 500 µm has been machined by applying two combination of electrolytes. A solid tungsten carbide with a diameter of 500 µm has been used as the tool in the present examination. The variable process parameters considered are machining voltage, micro tool feed rate and duty ratio and their influence on performance characteristics namely circularity entry, exit and surface roughness were studied. By using multi-response technique TOPSIS, the best combination of process parameter is found. SEM was employed to observe the morphology of the machined surfaces.

Keyword: Micromachining, EMM, Superalloy, Optimization

1. Introduction

Micromachining can be prescribed as the production of micro product/micro feature in macro component in the range of 1-999 µm. Combustor liner is one such component where number of micro holes are machined in order to achieve thin film cooling. Nimonic 75 is a nickel based super alloy, used for fabricating aero combustor liner as it can withstand high temperature. LBM and EDM are the processes used for machining micro-holes in combustor liner, to ensure the liner is cool enough to maintain its structural integrity. Due to their drawbacks like heat affected zone and thermal stresses, electrochemical micromachining (EMM) can be employed, as it combines benefits like absenteeism of HAZ and tool wear [1]. Electro chemical machining (ECM) is the better alternative process for generating micro features in hard-to-machine materials and EMM is a favored process for creating cooling holes in nickel based super alloys [2][3].

EMM is effective for generating high precision operations, thus it is widely used in current research and industrial practices [4]. ECM can be defined as the opposite of electroplating, used for machining conductive and hard-to-machine materials. The metal removal is attained by anodic dissolution of material. In electrochemical machining process the tool is made as the cathode, the workpiece as anode and inter-electrode gap is maintained between them, the whole arrangement is submerged in a
electrolytic cell. While electricity is given, the anode starts to dissolve and the mirror image of the tool is obtained, electrolyte is pumped in the inter-electrode gap to remove the debris from the machining zone to achieve good surface finish on the specimen. Sandip et al [5] presented an examination of fitness of electrolyte for machining titanium alloy by EMM process, in order to examine the efficiency of electrolytes.

Dengyong wang et al [1] states that anodic dissolution depends on the workpiece material, the electrolyte, power supply and the significant process parameters in ECM are frequency, voltage, duty cycle. Jerzy et al [6] investigated the outcome of feed rate and voltage on process efficacy. Mohanty et al [7] examined the machinability of inconel 825 by using copper as a tool and studied the significance of machining parameters on response characteristics like surface roughness and MRR. Elevation in EMM performance features can be attained by establishment of short pulse period [8].

Hariharan et al [9] presented a approach in order to optimize the process parameters on drilling nimonic 75 alloy by adopting EMM process. This work states about the augmentation of multiresponse characteristics of AWJ process, accomplished by utilizing TOPSIS method [10]. Literature analysis notify that only minimal investigations were presented on the subject of process parameters and combinations of electrolytes in EMM of Nickel based super alloys. This work objects to recognize the outcome of machining parameters on nimonic 75 alloy under various combination of electrolytes.

2. Materials and Methods

2.1 Selection of work materials
A circular shaped Tungsten carbide with a diameter 500µm is used as the cathode tool. Table 1 indicate the chemical composition of nimonic 75 alloy. Nimonic 75 is a nickel based super alloy that find application in aero combustor liner due to its innate properties.

Table 1. Nimonic 75 alloy – chemical composition

| Component | Ni % | Cr % | Cu % | Fe % | C % | Mn % | Si % | Ti % |
|-----------|------|------|------|------|-----|------|------|------|
| Nominal Weight (%) | Remaining Max 21 Max 0.5 Max 5 Max 0.15 Max 1.0 Max 1.0 Max 0.6 |
| Actual Weight (%) | 75.24 18.63 0.328 4.326 0.119 0.421 0.483 0.453 |

2.2 Selection of electrolyte
The first electrolyte (E1) selected for experimentation is a combination of C₂H₆O₂ (2.2ml), NaBr (2.2g), HF acid (3.2ml) and consolidation of NaNO₃ (5g), NaCl (2.5g) is considered as the second electrolyte (E2) based on trial experiments.

2.3 Experimental arrangement
The ECM arrangement consist of following components as indicated in figure 1. The metal is held in a fixture and placed inside the machining chamber and electrolyte tank aids in recycling the electrolyte by utilizing a filter. When power is switched on, the prescribed feed rate to approach the metal is attained by adopting a stepper motor.
3. Experimental Design

To evaluate the impact of process parameters on the response characteristics like circularity entry, exit and surface roughness, a set of experiments were conducted. The experimental plan has three process parameters namely machining voltage (A), micro tool feed rate (B) and duty ratio (C) as shown in table 2.

| Parameters | Levels         |
|------------|----------------|
| A          | 18, 20, 22     |
| B          | 0.5, 0.6, 0.7  |
| C          | 33, 50, 66     |

3.1 Performance characteristics

Circularity: It can be described as the diametric deviation of the machined hole and denoted by equation 1. Circularity entry ($C_{\text{entry}}$) and circularity exit ($C_{\text{exit}}$) of the drilled hole is assessed by means of Video measuring system.

\[ \text{Circularity} = D_{\text{max}} - D_{\text{min}} \] (1)

Surface roughness: The irregularity of the machined area is measured by the most common parameter $R_a$, which represents the arithmetic average of the profile. Surface roughness (SR) is assessed by using Talysurf CCI Lite, non contact 3D surface roughness tester.

3.2 Experimental observations

Circularity entry, circularity exit and surface roughness (SR) is calculated for each experiment and displayed in table 3.
## Table 3. Experimental observation with response

| S. No | Voltage (V) | Tool feed rate (µm/s) | Duty cycle (%) | E1 Circularity entry (mm) | E1 Circularity exit (mm) | E1 SR (µm) | E2 Circularity entry (mm) | E2 Circularity exit (mm) | E2 SR (µm) |
|-------|-------------|-----------------------|----------------|---------------------------|-------------------------|-----------|---------------------------|-------------------------|-----------|
| 1     | 18          | 0.5                   | 33             | 0.003                     | 0.005                   | 0.458     | 0.002                     | 0.004                   | 0.345     |
| 2     | 18          | 0.6                   | 50             | 0.016                     | 0.016                   | 0.601     | 0.001                     | 0.001                   | 0.389     |
| 3     | 18          | 0.7                   | 66             | 0.033                     | 0.035                   | 0.73      | 0.014                     | 0.01        | 0.412     |
| 4     | 20          | 0.5                   | 50             | 0.039                     | 0.038                   | 0.504     | 0.026                     | 0.008                   | 0.647     |
| 5     | 20          | 0.6                   | 66             | 0.046                     | 0.041                   | 0.787     | 0.017                     | 0.041                   | 0.717     |
| 6     | 20          | 0.7                   | 33             | 0.014                     | 0.029                   | 0.639     | 0.021                     | 0.021                   | 0.688     |
| 7     | 22          | 0.5                   | 66             | 0.114                     | 0.12                    | 2.281     | 0.021                     | 0.015                   | 0.847     |
| 8     | 22          | 0.6                   | 33             | 0.108                     | 0.103                   | 1.171     | 0.024                     | 0.018                   | 0.97      |
| 9     | 22          | 0.7                   | 50             | 0.085                     | 0.074                   | 1.12      | 0.067                     | 0.071                   | 0.854     |

### 4. Evaluation and Discussion

#### 4.1 Effect of voltage on response parameters under different electrolytes

From figure 2 it is noticed that circularity entry and exit are proportional to the machining voltage, as directed by faraday’s law of electrolysis. E2 exhibit lower circularity entry, exit and surface roughness values, at the chosen levels of voltage when compared to E1 combination. The presence of sodium chloride in E2 combination has ensured required amount of metal removal, as it contains aggressive anions, whereas the presence of sodium nitrate have resulted in lower surface roughness, due to the presence of oxidizing anions resulting in better surface finish. Mixed electrolytes provide combined advantages effect, thus the perfect blend of non-passive and passive electrolyte resulted in lower circularity entry and exit, which shows that the metal is eliminated in desired region only.

Figure 2 also depicts that stray current rises with voltage, hence incrementing the circularity entry and exit values. Circularity error (entry and exit) is more predominant in E1 electrolytic combination, at the chosen voltages, since E1 combination contains hydrofluoric acid which is a strong etchant, due to the etching factor, the atoms has been removed rapidly, as a result the metal is removed from undesired region and this yield poor geometrical accuracy and surface finish.

Based on the outcomes, it is instituted that circularity entry error was scaled down in the range of 67 to 63.7 %, circularity exit from 73% to 65% and surface roughness increased from 35% to 47.6% by using E2 electrolyte.

![Figure 2. Effect of voltage on response parameters](image-url)
4.2 Effect of tool feed rate on response parameters under different electrolytes

At lesser tool feed rate (TFR), tool rest at a specific region of workpiece for elongated duration resulting in desired circular feature. During high tool feed rate, the span of machining declines and therefore the desired circular feature is not obtained. By adopting low feed rate, high dimensional accuracy and finish can be obtained in the machined hole.

By using E2 combination, the hydrogen bubble formation was reduced at the tip of the microtool, because of the combination of nonpassive and passive electrolyte, which result in selective electrochemical dissolution. In relating the E1 electrolytic combination with E2, E2 yields lower values of circularity entry, circularity exit and surface roughness, at the chosen levels of tool feed rate.

From figure 3 it is found that at tool feed rate of 0.7 µm/sec, sufficient exposure to metal under E1 combination is not attained and hence the values of circularity entry and exit is lessened. Circularity entry error was decremented in the range of 69 to 22.7%, circularity exit from 83% to 26% and surface roughness increased from 26% to 37% by using E2 electrolyte.

![Figure 3. Effect of tool feed rate on response parameters](image)

4.3 Effect of duty ratio on response parameters under different electrolytes

Figure 4 shows the significance of duty ratio on response parameters of the machined hole. At higher values of duty ratio, pulse on time is more which increase the electrochemical dissolution not only in linear path but also in sideward path which result in added metal removal from the sideward direction of the material triggering circularity entry and exit error as shown in Figure 4.

When pulse off time is more, metal is dissolved in a linear pattern because increased pulse off period ensure to remove heat and sludges from the machining slot and diminish the metal removal from the sideward direction ensuring low circularity(entry and exit). E2 combination is more advantageous over E1 due to the existence of sodium nitrate which has less throwing power ensuing in controlled metal removal whereas sodium bromide in E1 combination has high throwing power.

At 66% of duty ratio it is observed that circularity entry and exit values were scaled down for E2 combination as elevated values of duty ratio result in less metal removal. At higher duty ratio, pulse off period is less than pulse on period resulting in untimely removal of sludges from the machining zone thus affecting the metal removal which influence the values of circularity entry and exit.

From the results, it is observed that circularity entry error was increased in the range of 63 to 73% and circularity exit from 68% to 66% and surface roughness increased from 16% to 47% by using E2 electrolyte.
4.4 Multiresponse optimization

In this study, multiresponse optimization technique namely, TOPSIS was used to examine the performance features of electro chemical micromachining process. The TOPSIS is defined as Technique for order preference by similarity to ideal solution and is reported by Olson [11]. This approach differs from other optimization methods since it uses positive and negative ideal solutions of each response. Among various alternatives, TOPSIS identifies the best solution which is very closer to the ideal solution. The following steps were considered in the multiresponse technique TOPSIS for the optimization of process parameters in the electro chemical micromachining of nimonic 75.

Step 1: Normalized value

In this step, all the observations (responses) were converted to normalized value (dimensionless) for an equal consideration of responses by using equation 2.

\[
\text{Normalized values, } r_{ij} = \frac{x_{ij}}{\sqrt{\sum_{i=1}^{n} x_{ij}^2}}
\]

where, \( i \) = Experimental runs, \( j \) = Output responses

Step 2: Weighted Normalized Matrix

Processing of this method requires input data for allocating the weights to the performance measurements. The standard weighing procedure SIMOS technique was used for this purpose. The description has been provided by Yuvaraj and Pradeep Kumar [10]. In the SIMOS technique, responses were arranged from the least to the most important. Details are tabulated in table 4.

**Table 4. Weighted Normalized Matrix**

| Variable                      | No of variables | Place of position | Non-normalized weight | Sum % |
|-------------------------------|-----------------|-------------------|------------------------|-------|
| Surface roughness             | 1               | 1                 | (1/6)*100 = 16.66 ~ 17 | 17    |
| Circularity entry and exit    | 2               | 2, 3              | (5/6)*100 = 83.33 ~ 83 | 83    |
| Total                         | 3               | 6                 |                        | 100   |

The weighted normalized values were obtained using equation 3.

\[
\text{Weighted Normalized Matrix, } y_{ij} = r_{ij} \times w_j
\]
Step 3: Determination of Greatest and Poorest Performance. In this step, the greatest and the poorest performance of each response were identified through its desirability criteria.

\[ S^+_j = \text{greatest performance}; \quad S^-_j = \text{poorest performance} \]

Step 4: Measurement of Positive and Negative ideal distance. The positive \( D^+_i \) and the negative ideal distance \( D^-_i \) for all the experimental runs were obtained using equations 4 and 5. These distances were measured from the greatest and the poorest performance of responses which are tabulated in table 5.

\[
D^+_i = \sqrt{\sum_{j=1}^{n}(Y_{ij} - S^+_j)^2} \quad i = 1 \text{ to } m \quad (4)
\]

\[
D^-_i = \sqrt{\sum_{j=1}^{n}(Y_{ij} - S^-_j)^2} \quad i = 1 \text{ to } m \quad (5)
\]

Step 5: Closeness coefficient values

The closeness coefficient values \( C_i \) were obtained using equation 6. \( C_i \) values and their ranks are given in Table 5.

\[
C_i = \frac{D^-_i}{D^+_i + D^-_i} \quad (6)
\]

| S. No. | \( D^+_i \) | \( D^-_i \) | \( C_i \) | \( D^+_j \) | \( D^-_j \) | \( C_j \) | Rank |
|--------|-------|-------|-------|-------|-------|-------|------|
| 1      | 0.000 | 0.361 | 0.970 | 0.009 | 0.352 | 0.990 | E1 1 |
| 2      | 0.025 | 0.323 | 0.927 | 0.010 | 0.364 | 0.973 | E2 2 |
| 3      | 0.094 | 0.268 | 0.741 | 0.073 | 0.292 | 0.799 | E1 4 |
| 4      | 0.106 | 0.260 | 0.709 | 0.129 | 0.263 | 0.671 | E2 5 |
| 5      | 0.123 | 0.227 | 0.648 | 0.201 | 0.176 | 0.467 | E1 6 |
| 6      | 0.059 | 0.233 | 0.799 | 0.136 | 0.156 | 0.535 | E2 7 |
| 7      | 0.361 | 0.001 | 0.002 | 0.123 | 0.245 | 0.665 | E1 9 |
| 8      | 0.314 | 0.071 | 0.184 | 0.146 | 0.223 | 0.604 | E2 8 |
| 9      | 0.181 | 0.134 | 0.425 | 0.328 | 0.122 | 0.271 | E1 7 |

Table 5. \( C_i \) values and their ranks
Figure 5 shows the closeness coefficient values of electrochemical machining process, in which high peak value of 0.99 has been recorded for E2 which is closer to ideal solution at the range of 18V, tool feed rate of 0.5 µm/s and at duty ratio 33%. Table 5 reveals the closeness coefficient value for each experiment. From the outcomes, it is noticed that E2 has higher $c_i$ value over E1. The indicated result had happened due to the impact of E2 on the response characteristics of EMM process. This further confirms that E2 combination can be used for EMM of nimonic 75 alloy over E1, due to the presence of aggressive and oxidizing anions. Enhancement in $C_i$ for E1 and E2 are 78% and 70% respectively, as shown in table 6.

Table 6. Comparison of initial and optimal setting results

| Conditions         | Setting level | Response parameters | $C_i$ value |
|--------------------|---------------|---------------------|-------------|
|                    |               | E1                  | E2          | E1 | E2 |
|                    | E1            | C_e     (mm) | C_x     (mm) | SR  (µm) | C_e | C_x | SR  (µm) | E1 | E2 |
| Initial setting    | A3B2C1        | 0.108   | 0.103   | 1.171 | 0.067  | 0.071 | 0.854 | 0.18 | 0.27 |
| parameters         | A3B3C2        |          |          |       |        |       |       |      |     |
| Optimal            | A1B1C1        | 0.003   | 0.005   | 0.458 | 0.002  | 0.004 | 0.345 | 0.97 | 0.99 |

Analysis of variance (ANOVA) is considered as a advantageous method for statistical inference, which was performed by means of MINITAB considering a significant level of 5%. By comparing the various input factors taken in EMM, voltage is the most influencing factor in E1 and E2 combination. As the levels of voltage chosen, influence not only the metal removal but also the error associated in machining features. Table 7 and 8 reveals the voltage contribution of 87% and 47.7% in the presence of E1 and E2 combination.
Table 7. ANOVA Result - E1

| Source | Degrees of freedom | Sum of squares | Mean square | F-Value | P-Value | % Contribution |
|--------|--------------------|----------------|-------------|---------|---------|----------------|
| A      | 2                  | 0.794          | 0.397       | 30.56   | 0.032   | 87.00%         |
| B      | 2                  | 0.0108         | 0.0054      | 0.42    | 0.705   | 1.2%           |
| C      | 2                  | 0.0818         | 0.0409      | 3.15    | 0.241   | 8.96%          |
| Error  | 2                  | 0.0259         | 0.0129      |         |         | 2.85%          |
| Sum    | 8                  | 0.9126         |             |         |         | 100%           |

Table 8. ANOVA Result - E2

| Source | Degrees of freedom | Sum of squares | Mean square | F-Value | P-Value | % Contribution |
|--------|--------------------|----------------|-------------|---------|---------|----------------|
| A      | 2                  | 0.30946        | 0.15473     | 2.29    | 0.304   | 47.74%         |
| B      | 2                  | 0.14128        | 0.07064     | 1.05    | 0.488   | 21.8%          |
| C      | 2                  | 0.06262        | 0.03131     | 0.46    | 0.683   | 9.66%          |
| Error  | 2                  | 0.1349         | 0.06745     |         |         | 20.81%         |
| Sum    | 8                  | 0.64826        |             |         |         | 100%           |

5. Hole Morphology

Figure 6 and 7 depicts the scanning electron microscopic images of machined profile at the range of 18 volt, 0.5 µm/sec tool feed rate and duty ratio at 33% for E1 and E2, conforming to the maximum closeness coefficient. Comparing the SEM images of optimal parametric setting of E1 and E2 conditions, the micro-hole machined by E2 combination possess reduced circularity and surface roughness. Improved accuracy and better surface finish were achieved in E2 combination when compared to E1 combination. As even metal dissolution have occurred in the machined region, due to the effect of combination of electrolytes used. Whereas in E1 combination uneven material removal and surface irregularities have occurred due to the absence of oxidizing anions which in turn expose the metal surface resulting in increased metal removal and falling of material owing to the presence of etchant in E1.
Figure 6. SEM image of drilled hole at 18 volt, 0.5 µm/sec tool feed rate and duty cycle at 33% for E1

Figure 7. SEM image of drilled hole at 18 volt, 0.5 µm/sec tool feed rate and duty cycle at 33% for E2

6. Conclusion
EMM was instigated on nimonic 75 alloy to examine the effect of process parameters on different features like circularity entry, circularity exit and surface roughness. Utilizing multi-response technique TOPSIS optimization was performed. From this investigation, the succeeding results have been consolidated.

- Experimental outcome expose that E2 produce lesser values of surface roughness, circularity entry and circularity exit when compared to E1.
- For E1 and E2 combination, lesser range of micro tool feed rate, duty ratio and machining voltage produce preferred surface roughness and lesser circularity entry and circularity exit values.
- Based on ANOVA, voltage is the utmost influencing parameter in E1 and E2 investigation.
- The TOPSIS outcomes showed that the overall enhancement of $c_i$ is 78% and 70% in E1 and E2.

REFERENCES

[1] Wang D, Zhu Z, Wang N, Zhu D and Wang H 2015 Investigation of the electrochemical dissolution behavior of Inconel 718 and 304 stainless steel at low current density in NaNO3 solution Electrochimica Acta 156 301-307
[2] Sharma S, Jain V K and Shekhar R 2002 Electrochemical drilling of inconel superalloy with acidified sodium chloride electrolyte *The International Journal of Advanced Manufacturing Technology* 19 492-500

[3] Rajurkar K P, Sundaram M M and Malshe A P 2013 Review of electrochemical and electrodischarge machining *Procedia Cirp* 6 13-26

[4] Bhattacharyya B, Munda J and Malapati M Advancement in electrochemical micro-machining *International Journal of Machine Tools and Manufacture* 44 1577-1589

[5] Anasane S S and Bhattacharyya B 2016 Experimental investigation on suitability of electrolytes for electrochemical micromachining of titanium *The International Journal of Advanced Manufacturing Technology* 86 2147-2160

[6] Mohanty A, Talla G, Dewangan S and Gangopadhyay S 2014 Experimental study of material Removal rate, surface roughness & Microstructure in electrochemical Machining of inconel 825 *In 5th International & 26th All India manufacturing technology, Design and Research Conference AIMTDR* 174(1-6)

[7] Kozak J, Rajurkar K P and Makkar Y 2004 Selected problems of micro-electrochemical machining *Journal of Materials Processing Technology* 149 426-431

[8] Reddy M M K 2013 Influence of pulse period and duty ratio on electrochemical micro machining (emm) characteristics *International Journal of Mechanical Engineering and Applications* 1 78-86

[9] Pillai H P, Sampath S C, Elumalai R, Harirhan S and Natarajan Y 2017 Influence of Process Parameters on Electrochemical Micromachining of Nimonic 75 Alloy *International Mechanical Engineering Congress and Exposition, American Society of Mechanical Engineers Digital Collection* 2 V002T02A007

[10] Yuvaraj N and Pradeep Kumar M 2015 Multiresponse optimization of abrasive water jet cutting process parameters using TOPSIS approach *Materials and Manufacturing Processes* 30 882-889

[11] Olson D L 2004 Comparison of weights in TOPSIS models *Mathematical and Computer Modelling* 40 721-727