Review Microstructure of Nickel-Based Single-Crystal CMSX-4 Superalloy after Electrochemical Machining

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Research Article

Keywords: Electrochemical machining, CMSX-4, Microstructure, SEM, EDS

DOI: https://doi.org/10.21203/rs.3.rs-186169/v1

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Abstract

A special position has been created for using the nickel-based single-crystal CMSX-4 superalloy at high temperatures due to the improved mechanical properties of this material and the absence of grain boundary in the crystal lattice. Also, electrochemical machining can be an effective method for machining this superalloy due to its unique performance in metal machining, like creating stress-free surfaces, high-level surface smoothness, and machining of complex geometries. This single crystal superalloy's microstructure consists of three phases: Gamma, Gamma prime, and a bit of carbide. Gamma prime is distributed cubically and homogeneously in the Gamma field without any boundaries and as a single crystal. It is essential not to change the microstructure after the production process or machining. In the present research, electrochemical machining was performed on CMSX-4 single crystal superalloy. The workpiece's microstructure was then investigated before and after electrochemical machining using scanning electron microscopy and EDS analysis from two sides. No changes were seen in CMSX-4 infrastructure after electrochemical machining EDS analysis and Images.

1. Introduction

Electrochemical machining (ECM) is one of the most economical and efficient methods in non-traditional machining, in which machining occurs using electrolysis or electrochemical dissolution. In this process, the workpiece is connected to the positive pole and the tool to the negative pole of a direct current power supply. The tool moves towards the workpiece, and a very small space is created between the anode and cathode, so-called the machining gap. An electric current electrolyte conductor is flowed into the machining gap space to complete this electrical circuit and move the electrons. Hence, the electrons moved, the ions separated from the workpiece's surface, and hydrogen gas is created around the tool. This machining is an anodic dissolution process that follows the Faraday relationship [1-3]. In this process, there is no contact between the cathode and the anode, so no stress occurs on the surface of the part, and the problems caused by the heat-affected area, which is a disadvantage of the workpiece in similar methods such as electrical discharge machining, does not exist in this method. In the machining, the workpiece's hardness and toughness have no effect, and the tool does not wear. Therefore, ECM has many applications in various fields such as aerospace, medical, automotive, electronics, energy, micro components, etc [4-6].

CMSX-4 superalloy is the second generation of nickel-based superalloy, which is used to manufacture various air and ground gas turbine blades. Polycrystalline structures lack significant high-temperature mechanical properties due to grain boundaries since grain boundaries are generally considered the alloy's weaknesses at high operating temperatures. This single crystal superalloy is an extended version of CMSX-2 with a range of desirable properties such as high creep strength, mechanical and thermal fatigue, good fuzzy stability, high resistance to corrosion, and hot oxidation in the gas turbine blade applications [7-10].

Based on the previous review, little activity has been performed in this special nickel-based superalloy’s ECM [11, 12]. In the following, some previous research in the field of ECM of superalloys (especially nickel base) is reviewed.

Klocke et al. reported a high machining rate and proper surface quality in ECM of nickel and titanium base superalloys widely used in jet engine blades and discs [13]. The focus was on tool design and determination of electrochemical dissolution based on tool feedrate and electric current density in this research. The investigated nickel-base superalloys are Inconel 718, Inconel 718 DA, Waspaloy, René 88, IN 100, which no activity has been performed on the single-crystal superalloy. One of the obtained results was to create better ECM capability for microstructures with arranged granulation. Also, good ECM behavior and effective machining rate were reported.

Based on this research, ECM is an effective method in selective machining to separate stainless steel from Ti6Al4V. Two types of electrolytes were investigated, which in one stainless steel is machined. In another, the layer created by steel-titanium phases is also machined [14].

Niu et al. [15] investigated the ECM of the nickel-based GH4169/Inconel 718 superalloy and resulted in the chipping rate, surface, and uniform Machining gap quality improved by increasing the progress rate. In addition, a method was provided for electrolyte current as jets for electrochemical milling on this nickel-based superalloy, which has good productivity and stability, especially for deep-depth chipping.

In another paper, the improvement of platform accuracy and the reduction of stray current in the ECM of blade profiles were investigated for three materials SS304 Inconel 718 and TiAl 4822 with auxiliary tools in the cathode. For these materials, cathode (tool) is designed in three conditions, without insulation and auxiliary tools, cathode insulation and a combination of cathode insulation, and ECM auxiliary tools in which the auxiliary tool improves platform accuracy and reduces extra machining [16].

Furthermore, the effect of how electrolyte flows on ECM performance of blisk was investigated numerically and experimentally [17]. This process for blisk machining is improved up to 70% by changing the electrolyte flow mode.

Evaluating the effect of scale in the ECM process of Nitinol alloy is the main goal of Mouliprasanth and his colleague in this research, which transfers machining parameters from micro to macro. Tool progress, duty ratio, and voltage are considered input parameters of the process, and machining removal rate and geometric properties are considered response variables [18].
Ge et al. [19] investigated the electrochemical dissolution behavior on K423A nickel-based superalloy in sodium nitrate electrolyte. It has been noted that ECM is an effective method for machining nickel-based superalloys on which little research has been performed. Sodium nitrate's performance is reported as a better electrolyte than sodium chloride, and the appropriate concentration for sodium nitrate electrolyte 10%. Finally, the ECM method was introduced as an effective and low-cost process for machining K423A nickel-based superalloy.

Wang et al. provided a method to improve local machining in the ECM process. In the proposed method, synchronization of pulse power supply and low-frequency oscillations were applied, in which the on/off positions of the pulse power supply are symmetric due to the feed wave. Simulations and experiments were performed on rhombus-shaped holes, which improved surface accuracy and quality [20].

Bilgi et al. [21] investigated drilling using the ECM method on nickel-base superalloy. Moreover, suitable solutions were proposed to remove obstacles to the ECM of large blades with two-way torsion angles and platforms. The optimal feedrate of the cathodes towards the blade is determined. Besides, a new method has been proposed for flushing of electrolyte in the machining gap on both sides of the blades and cathodes, which have excellent results in both simulations and experiments [22].

Huang and Liu [23] investigated micro-ECM to create complex shapes on the nickel-based superalloy. Complex micro-dimensional shapes were created on nickel-based superalloy by creating nanoscale pulses. The parameters of voltage, pulse on-time, and electrode diameter were investigated, and finally, complex shapes were machined.

Burger et al. [24] investigated the ECM of LEK94 single crystal superalloy, which is the only available report on single-crystal superalloys' ECM. The current density was introduced as the most important and effective machining parameter for improving surface quality. The current density is affected by the machining gap and voltage, and electrolyte conductivity. Based on the results, low current density leads to non-uniform dissolution in superalloy and high surface roughness. High current density should be created to achieve uniform electrochemical dissolution, which high machining rate and low surface roughness in high current density were created on this nickel-base superalloy.

Singh et al. [25] analyzed the microstructure and multi-objective optimization of ECM of Inconel 825 superalloy using a hybrid method. The voltage, electrolyte concentration, and tool feedrate parameters are considered as optimization inputs, machining removal rate, surface roughness, and extra machining are considered as objective functions. Finally, a voltage of 16 volts, a concentration of 45 g/l, and the tool feedrate of 0.3 mm/min are presented as the optimal machining conditions.

The surface integrity of three γ-TiAl alloy models was investigated after ECM by Wang et al. Two levels of current densities were used to compare the low and high levels of process current densities' surface properties after ECM. Local corrosion and rough surfaces result from machining at low flow densities, while smooth surfaces (polishing) are created at high current densities of all three alloys [26].

In the present research, the microstructure of CMSX-4 nickel-based single-crystal superalloy was investigated before and after ECM. Scanning electron microscopy front and side images of the workpiece surface indicated that the single-crystal microstructure does not change due to electrochemical machining.

### 2. Methods

#### 2.1. Workpiece

The workpiece is made of nickel-based single-crystal superalloy, which has unique properties, and its chemical composition is shown in Table 1. This material has high hardness and strength, and their machining using other traditional and modern methods has challenges such as wear in tools, residual thermal and mechanical stresses, heat affected zone (HAZ), and the accuracy and quality of the surface. CMSX-4 nickel-based superalloy is created by vacuum induction melting (VIM). A laboratory Bridgman furnace with a water cooling mechanism is used for directional freezing. Then, single crystal samples are subjected to precipitation hardening heat treatment, including dissolution annealing and aging steps. Figure 1 shows a casted ingot of this material, which was grinding and then cut into tablets of this material with a diameter of 2 mm.

#### 2.2. Tool and electrolyte

In the initial experiments, tools made of copper and brass were prepared, but brass tools were used due to easier construction, suitable electrical conductivity, and better results in implementing the initial experiments. The tool's diameter is 6 mm, and the tip of the tool should be polished and grinding to remove any turning effects and create a smooth and polished surface. Three of this tool was made, which there should be an alternative tool if there is damage to the tool during the experiments. Figure 2 shows the used tools in this research. The side of the tool is insulated to prevent the negative impact of stray currents. Figure 3 shows how the negative pole is connected to the tool and the
power supply's positive pole to the workpiece. The tool was placed at a distance from the workpiece in the work compartment known as the machining gap.

This machine can flush the electrolyte parallel (from the working chamber's side walls) or perpendicular to the workpiece surface (inside the tool). The electrolyte is one of the main factors in ECM. The uniformity and purity of the electrolyte have great importance during the machining process. The metals removed from the workpiece and the impurities in the electrolyte increased the risk of sparks in addition to changing the rate of machining, which a proper purification and filtration system should be considered in the machine. The used electrolyte is sodium nitrate solution with a concentration of 80 g/l.

3. Experimental Details

The experiments were performed according to the range specified for the ECM process's input parameters based on previous activities [11, 12]. An ECM machine with high flexibility and reproducibility and suitable for experiments is needed to perform practical testing and develop electrochemical machining technology. A device with a 30 Volts voltage and a 108 Amps current was used to direct and pulse machining. This machine consists of four separate units; machine, power supply, electrolyte, and control units that interact with each other with a special integration. This machine is constructed by considering the measurement units and suitable monitoring for performing experiments and acquiring technical knowledge for electrochemical machining. Figure 4 shows an image of this machine.

3.1. Sample and fixture preparations

Unfortunately, this material has no magnetic properties, so blocking and grinding it has been difficult and costly. The workpiece's thickness was 2 mm, which is cut by the wirecut method and then grinned. Grinding and creating a smooth and polished surface is necessary to eliminate machining and cutting effects. A fixture is designed, and the workpiece is placed in a working chamber and connected to the power supply using a screw to this fixture which is shown in Figure 5. The chamber is made of Plexiglas, which is both electrically insulating and resistant to chemicals. Plexiglas’ transparency makes the process better, in addition to being beautiful and visible inside the chamber.

3.2. Implementation of experiments

Finally, machining has performed on the workpiece after adjusting the input parameters of the process such as voltage, machining time, and initial machining gap. The experimental parameters were adjusted according to Table 2.

3.3. SEM / EDS testing

After polishing, the samples were polished with 0.2 to 0.05 μm alumina powders to observe the cross section's dendritic microstructure, and an etch marble solution was used to observe the microstructure. The samples' microscopic structure was evaluated using a Nikon EPISHOT 300 optical microscope equipped with a SONY camera and SEM PHILIPS XL30 and FE-SEM TESCAN MIRA3 electron microscopes.

4. Results And Discussion

Figure 6 is prepared using a VMM (Video Measuring Machine) to measure and plot how the workpiece moves during the experiments. The workpiece is cut from the middle with wire-cut after ECM for 30 seconds, and Figure 6 shows the cut section's width. In the present research, the workpiece's microstructure has been investigated from two sides using scanning electron microscopy and EDS in two modes without electrochemical machining and after that. One side is the top view (from sides A and B in Figure 6), and the other side is the front view (perpendicular to the surfaces C and D in Figure 6).

4.1. Investigation of the machining surface’s microstructure before and after ECM

The microstructure was studied in two different sections before and after ECM operations. First, the surface was analyzed using scanning electron microscope images before and after machining. Figure 7 indicates the pre-machined surface before machining (from view A according to Figure 6) and Figure 8 microstructure images after ECM (from view B according to Figure 6). All scanning electron microscope images show that there is no grain boundary. Based on the previous research, this alloy is mainly composed of three phases, gamma field phase γ, strengthening gamma prime phase γ', and a small carbide phase. Large and white particles indicate the carbide phase, which is dispersed heterogeneously.
Tables 3 and 4 show the EDS analysis of points A and B in Figure 7 and Figure 8. According to the EDS analysis, the white dots A represent the carbide phases in the scanning electron microscope images, including titanium and tantalum carbides. In addition, the gamma prime -shaped cubic phases are distributed homogeneously and at very short distances from each other within the gamma field phase. According to EDS analysis, point B has more nickel, which indicates the gamma field phase. The microstructure has not changed due to the machining process, and the three main phases of this superalloy are present in the microstructure by comparing the images of a light electron microscope before and after ECM on this superalloy. EDS analysis indicated that the microstructure’s chemical composition in both states includes three phases: gamma, gamma prime, and carbide.

4.2. Investigation of the lateral surface’s microstructure before and after ECM

In the following, scanning electron microscope images of the lateral surface before (Figure 9) and after (Figure 10) of ECM are presented to study the microstructures. As can be seen, in both cases, the microstructure includes the gamma phase and gamma prime phases. Also, some carbide phases are present in the form of large and heterogeneous particles in the microstructure. According to the EDS analysis in Table 5, points A and B indicate the carbide phase in microstructure before ECM, which is the same phase seen in Table 6 at point A. Point C in Table 5 and point B in Table 6 represent the gamma phase, and since nickel point C in Table 5 is less than point B in that table, point C represents the gamma-prime phase.

5. Conclusion

Machining surface and lateral surface were observed before and after the electrochemical machining process from two viewpoints according to scanning electron microscopy and EDS analysis for CMSX-4 superalloy. In addition, the large white particles in these images are carbide phases that are dispersed heterogeneously before and after the ECM process. The black parts indicate the gamma prime strengthening phase and the gamma field phase, which are observed as the field gamma phase and the cubical gamma prime phase. In scanning electron microscope images, the sample is free of grain boundaries before and after machining. The gamma-prime cubic phases are homogeneously distributed at very short distances from each other within the gamma field phase. Based on comparing the images, ECM does not affect the microstructure, and the sample is still a single crystal with gamma-prime phases distributed in the gamma field. Also, scanning electron microscopy images show that the sample under machining conditions lack new grain germination or a significant defect in the microstructure of the CMSX-4 nickel-based single-crystal superalloy.

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**Tables**

**Table 1 Chemical composition of CMSX-4 workpiece**

| Cu  | Fe  | P   | V   | Mn | Si  | C   | Nb | Hf | Mo | Ti  | Re | Al | W | Ta | Cr | Co | Ni |
|-----|-----|-----|-----|----|-----|-----|----|----|----|-----|----|----|---|----|----|----|----|
| 0.07| 0.5 | 0.9 | 2.8 | 5.45| 6.2 | 6.3 | 6.4| 9.3| Rem.|     |    |    |   |    |    |    |    
| 0.005| 0.015| 0.015| 0.01| 0.04| 0.08| 0.1 | 0.12| 0.7 | 1.1 | 3.1 | 5.75| 6.6 | 6.7 | 6.6 | 10 |

**Table 2 The ECM process parameters**

| Tables |
| Value     | Process parameter          |
|-----------|-----------------------------|
| 25 V      | Voltage                     |
| 150 micron| Machining gap               |
| 2 µm/s    | Tool feed rate              |
| 80 gr/liter| Electrolyte Concentration  |
| 8.88 S/m  | Electrolyte Conductivity    |
| 30 s      | Time of machining           |

Table 3 The EDS analysis of points A and B in Figure 7

| Point B [wt.-%] | Point A [wt.-%] | Series | Element |
|-----------------|-----------------|--------|---------|
| -               | 8.16            | K series | Carbon |
| 1.16            | 17.52           | K series | Titanium |
| 5.43            | 5.21            | K series | Chromium |
| 7.63            | 4.21            | K series | Cobalt |
| 61.94           | 27.49           | K series | Nickel |
| 8.25            | 37.42           | L series | Tantalum |
| 5.53            | -               | K series | Aluminum |
| 0.31            | -               | K series | Iron |
| 4.46            | -               | L series | Tungsten |

Table 4 The EDS analysis of points A and B in Figure 8

| Point B [wt.-%] | Point A [wt.-%] | Series | Element |
|-----------------|-----------------|--------|---------|
| -               | 5.15            | K series | Carbon |
| 0.73            | 11.76           | K series | Titanium |
| 4.75            | 6.54            | K series | Chromium |
| 8.22            | 5.37            | K series | Cobalt |
| 58.48           | 36.78           | K series | Nickel |
| 11.55           | 34.41           | L series | Tantalum |
| 3.02            | -               | K series | Aluminum |
| 0.71            | -               | K series | Iron |
| 2.04            | -               | L series | Molybdenum |
| 10.48           | -               | L series | Tungsten |

Table 5 The EDS analysis of points A, B and C in Figure 9
| Point C [wt.-%] | Point B [wt.-%] | Point A [wt.-%] | Series  | Element    |
|----------------|-----------------|-----------------|---------|------------|
|                | 4.18            | 5.59            | K series| Carbon     |
| 0.21           | 14.42           | 23.99           | K series| Titanium   |
| 4.68           | 7.45            | -               | K series| Chromium   |
| 6.25           | 6.32            | -               | K series| Cobalt     |
| 64.01          | 35.67           | 6.31            | K series| Nickel     |
| 5.96           | 31.96           | 64.11           | L series| Tantalum   |
| 5.51           | -               | -               | K series| Aluminum   |
| 3.71           | -               | -               | L series| Molybdenum |
| 9.67           | -               | -               | L series| Tungsten   |

Table 6 The EDS analysis of points A, B and C in Figure 10

| Point C [wt.-%] | Point B [wt.-%] | Point A [wt.-%] | Series  | Element    |
|----------------|-----------------|-----------------|---------|------------|
|                | -               | 10.29           | K series| Carbon     |
| 2.51           | 0.54            | 13.47           | K series| Titanium   |
| 6.05           | 5.08            | -               | K series| Chromium   |
| 7.90           | 8.94            | -               | K series| Cobalt     |
| 56.90          | 62.79           | 21.02           | K series| Nickel     |
| 9.18           | 4.75            | 55.22           | L series| Tantalum   |
| 5.37           | 7.55            | -               | K series| Aluminum   |
| 4.06           | -               | -               | L series| Molybdenum |
| 8.04           | 10.36           | -               | L series| Tungsten   |