

Article

Investigations on the Surface Integrity of Ti6Al4V under Modified Dielectric(s)-Based Electric Discharge Machining Using Cryogenically Treated Electrodes

Muhammad Umar Farooq ^{1,*}  and Saqib Anwar ² ¹ School of Mechanical Engineering, University of Leeds, Leeds LS2 9JT, UK² Industrial Engineering Department, College of Engineering, King Saud University, P.O. Box 800, Riyadh 11421, Saudi Arabia

* Correspondence: mn21muf@leeds.ac.uk

Abstract: The surface integrity of machined components is considered to be an important part of the quality matrix for high-performance applications in the aviation industry. Therefore, close attention is given to the components made up of hard-to-cut materials such as Ti6Al4V, which face processability challenges. In this regard, among the non-conventional machining processes, electric discharge machining is widely preferred for cutting Ti6Al4V. In this study, the potentiality of cryogenic-treated tool electrodes (graphite and aluminum) with unmodified (kerosene) and modified (kerosene with Span 20, Span 60, and Span 80) dielectrics are comprehensively investigated. A three-phased experimentation framework is deployed based on the following process parameters, i.e., surfactant type, surfactant concentration, electrode material, and pulse ON:OFF time. Thorough statistical analyses are performed based on the full factorial design of experiments, and the results are characterized by process physics. It is found that the cutting mechanism is highly dependent on the surfactants, especially their hydrophilic–lipophilic balance in the dielectric. The desirability-based process optimization results show that the unmodified process (with kerosene) produced relatively higher roughness values of 7.5 μm and 5.8 μm for aluminum and graphite electrodes, respectively. However, the modified process (with surfactant) resulted in a lower degree of roughness on the workpiece. The graphite electrode using S-60 with a resulting R_a of 1.73 μm outperformed the aluminum electrode using S-20, yielding an R_a of 3.4 μm . The S-60 surfactant resulted in minimum roughness on the workpiece with the fewest surface defects at the 25 g/L concentration, 50:25 μSec pulse ON:OFF time, and with the cryogenically treated graphite electrode.

Keywords: sustainability; dielectric; Ti6Al4V; surface integrity; electric discharge machining; cryogenic; tool



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1. Introduction

The micro-goals of manufacturing sustainability include the efficient processability of hard-to-cut alloys used in the biomedical, aeronautical, automotive, and nuclear industries [1]. Titanium and its variants are used widely for these hard-to-cut alloys because of their exceptional engineering attributes. The properties include high strength, wear, cryogenic characteristics, and corrosion and wear resistance. Ti6Al4V is widely used in industries among the different titanium variants due to its applicability in high-performance applications [2]. However, the processing of the alloy remains challenging. Along with several engineering benefits, Ti6Al4V proposes challenges such as low thermal conductivity, high strength, and strain hardening, which undermine machining through conventional processes such as turning, milling, and drilling [3]. Therefore, non-conventional machining techniques, such as those using laser beams, electron beams, electric discharge, and plasma arc cutting, are commonly used. In addition to the processability of materials, the desired quality matrix includes surface integrity. For instance, electron beam melting is an additive

manufacturing process that fabricates complex shapes; however, inter-layer roughness reduces the product integrity by inducing the risk of fatigue cracks [4]. Moreover, the need for a surface modification remains. Therefore, the earlier processes are continuously evaluated to solve the challenges in generating a good surface finish and properties by devising efficient machine settings. However, electric discharge machining is the preferred choice for machining, which involves cutting the material into the desired shape and enhances the surface hardness, corrosion and fatigue resistance, and other mechanical properties [5]. In addition, the generated electric discharge-machined surface is enriched by oxides and carbides, which enhance the mechanical functionality [6]. The process functions in a narrow tolerance range for high-quality applications. Likewise, the surface integrity achieved through electric discharge machining (EDM) is comparable to that of the conventional grinding process, which contributes as a second-step solution to regular metal-cutting operations [7]. Therefore, electric discharge machining is a considerable choice for producing the work part intended for high-end applications.

The electric discharge machining process works by a spark erosion mechanism that can machine intricate shapes with high precision in hard-to-cut materials. Hard metals, composites, and difficult-to-cut aerospace alloys are processed without any contemplation of the physical or metallurgical properties. The process employs an electro-erosion mechanism with high-frequency electric sparks to erode the workpiece surface through a cycle of melting, vaporization, and solidification [8,9]. There is no physical contact between the workpiece and the tool electrode. However, both electrodes are entirely immersed in the dielectric medium. During discharge sparking, the dielectric medium becomes ionized to create a pathway for sparks forming a plasma channel in the inter-electrode gap [2]. The material surface goes through melting and vaporization phases because of the sparks' high energy, which leaves multiple craters behind. The tool electrode carries a mirror shape of the desired geometry. If the electric discharge machining process is categorized into three segments, namely spark initiation, melting and vaporization, and flushing, then one-third of the weightage is given to the latter part [10].

In the flushing mechanism, dielectric fluid performs various functions and is considered to be a vital part of the operation. The dielectric fluid controls the discharging process in the interaction regime. It helps to flush the debris and melt pool from the machined surface. Moreover, it contributes to heat dissipation and cooling down the workpiece during spark off-time [11]. The commonly used dielectric fluids include EDM oil, mineral oils, hydrocarbon-based oils such as kerosene, gases in dry machining, water, emulsion-based dielectrics, and micro- and nano-additives-mixed dielectrics. The dielectrics' different properties enhance the material removal rate, surface quality, wear characteristics, and accuracy control [12]. Kerosene is widely used as a dielectric in electric discharge machining. Since the electric discharge machining process has a low cutting rate, several developments have been made to enhance the process efficiency. Ahmed et al. [2] investigated large ranges of the electric parameters, such as the pulse current (more than 10 A) and pulse on- and off-time, to achieve higher productivity during the EDM of Ti6Al4V. The study reported a high carbon content (proportion of black spots) by graphite and copper electrodes. However, the resulting surface was coarser, producing $R_a = 8.85 \mu\text{m}$, which negates the goal of achieving a high surface integrity. The authors reported that the reasons for the deteriorated surface quality were the exotic pulse current levels and pulse on- and off-time. In addition to an excessive number of parametric settings, different additives are evaluated with kerosene oil to enhance the process yield. For instance, Farooq et al. [8] added silicon powder (5, 10, and 20 g/L) to dielectric fluid to generate a biomedically favorable surface while machining a biomedical-grade Ti alloy. The authors benefited from finishing process parametric levels of 100:100 μs , a pulse ON:OFF duration, and a 5 A pulse current with 5 g/L Si powder concentration to achieve a superior surface finish. Similarly, Mughal et al. [13] modified the surface of Ti6Al4V ELI using similar a method to achieve lower roughness levels. The underlying observation is that low energy transfer (5 A) on the surface resulted in a superior quality as a higher sparking intensity deteriorates the

surface. The powder acts as a bridge in the inter-electrode gap of the tool electrode and workpiece and facilitates achieving a suitable voltage. The powder particles promote the bridging effect, which enhances the spark gap because of the bond formation of powder particles in the machining regime [11]. The interconnected channels increase the number of sparks per unit time, thus enhancing workpiece material removal. In addition, the plasma channel is enlarged by adding powder with enhanced spark density. Moreover, the benefits include uniform sparking over the interaction area, helping to achieve a smoother surface and higher material removal rate [14].

On the other hand, powder-mixed dielectrics can severely deteriorate the performance of the electric discharge machining through agglomeration [13]. The electrostatic forces tend to promote agglomeration, which hampers EDM. In addition, this phenomenon decreases the number of sparks per unit time due to the poor sparking performance of available non-agglomerated powder. The problem was resolved through surfactants, another type of additive, to reduce the chances of agglomeration. The surfactants, known as surface-active agents, showcase hydrophilic and hydrophobic natures [15]. The surfactants are available in various types: ionic (anionic and cationic), non-ionic, and zwitterionic ones. Using surfactants lowers the surface tension in the dielectric, potentially avoiding the agglomeration of powder additives. In addition to surfactants, emulsifiers are also used to enhance the suspension rate to improve the sparking and machining actions [16]. Fundamentally, the surfactant enhances the conductivity of the dielectric medium in the inter-electrode interaction zone, minimizes surface tension of the overall dielectric, and avoids the accumulation of powder particles through better dissolvability and scattering. Consequently, these mechanisms improve the surface quality and enhance the cutting rate [14]. Reddy et al. [17] machined PH17-4 stainless steel using electric discharge machining oil. The dielectric was modified using Span 20 (S-20) surfactant and graphite powder to improve surface quality. The optimum process parameters, a pulse current of 10 A, a surfactant concentration of 4 g/L, and a powder concentration of 9 g/L, resulted in 3.17 μm roughness and 14.1 μm recast layer thickness.

Similarly, Kolli and Kumar [18] used 4 to 6 g/L surfactant concentrations in the dielectric to improve the surface quality. The study concluded that a low pulse current favored an improved surface finish in the modified process. Sugunakar et al. [19] resulted in a 5.49% decrease in surface roughness with the addition of 6 g/L S-20 surfactant with silicon powder. Ilani and Khoshnevisan [14] studied intermolecular forces by comparing dielectrics without a surfactant, with 21.81 g/L, and 43.35 g/L surfactant with Al and Al_2O_3 powders. The study showed that adding a surfactant significantly reduced the intermolecular forces in the inter-electrode machining zone, improving the process output and enhancing the surface finish. König and Jorres [20] evaluated organic compound-based aqueous emulsions as potential dielectrics in the electric discharge machining of steels. The study showed that adding glycerin reduced the tool wear under rough conditions (a high discharge current of 20 A). However, water-based dielectric degraded the metal characteristics and surface roughness (from 9 to 10 μm). Zhang et al. [21] evaluated water in an oil emulsion and obtained a higher relative electrode wear rate. The authors related the dielectric's high viscosity to limiting the spark channel expansion and reducing the processing efficiency. Moreover, the study highlighted the detrimental effects of a high discharge energy and impulsive forces on the surface.

In the same way, Wu et al. [22] evaluated the surface finish on SKD-61 steel using pure kerosene, aluminum mixed with kerosene, and aluminum and surfactant mixed with kerosene under extreme finishing parametric conditions (0.3 A pulse current). The dielectric conduction was increased with the addition of surfactant in the dielectric, which resulted in the uniform distribution of discharge energy. Dukhin and Goetz [23] investigated the ionic behavior of non-ionic surfactants such as S-20 and S-80. In comparison to pure water, both surfactants produced significantly higher dispersions of powder particles by employing the dissociation and dislocation mechanisms of surfactant molecules. Similarly, Wu et al. [24] investigated S-20 and S-80 with surfactant concentrations ranging from 10 to 100 g/L.

Nevertheless, pulse currents between 0.6 and 6 A were chosen for attaining a better surface quality during machining. The study showed an increase in conductivity of the dielectric through the hydrophilic–lipophilic balance (HLB) and resulted in a superior surface quality and discharge efficiency at S-20 as compared to that at S-80 [23]. Reddy et al. [25] used Span 20 because of its higher HLB value to machine PH 17-4 stainless steel. The authors discussed the effects of the hydrophilic group absorbing debris and carbon dregs and the hydrophobic tail extending towards dielectric fluid. The HLB value altered the dielectric conductivity and produced a superior surface quality.

It can be seen that surfactant and powder particles are used during electric discharge machining. However, the performance of standalone surfactants has not been comprehensively examined. It is well known that debris hinders the process efficiency during machining. Therefore, it is hypothesized that surfactants will help to achieve better debris dispersion and enhance the machinability performance of Ti6Al4V due to improved dielectric conductivity. Additionally, a surfactant-based modified dielectric during electric discharge machining has not been used to investigate the surface integrity of Ti6Al4V.

Along with the progress related to the dielectric, significant developments have been made in the tooling requirements of the electric discharge machining process. In this regard, the cryogenic treatment of materials such as tool electrodes is a viable alternative to improve the electric discharge machining process' efficiency. The cryogenic treatment enhances the electrical and thermal properties of the tool electrode by refining the microstructure [26] and improves wear resistance [27]. Singh and Singh [28] evaluated the performance of Ti, copper (Cu), and copper chromium (CuCr) electrodes during a cryogenic treatment while machining Titan 15 ASTM grade 2. The authors discussed a 58.77% improvement in the electrode wear ratio and a 7.99% reduction of the surface roughness. Sundaram et al. [29] analyzed the performance of cryogenically treated electrodes on a beryllium–copper alloy and showed a 4.5% reduction of tool wear. However, the surface integrity aspect was not studied. Grewal and Dhiman [30] investigated the performance of cryogenically treated Cu electrodes on EN24 steel. The cryogenic treatment reduced the surface roughness by 15.75% and improved the machined surface quality. Srivastava and Pandey [31] machined M2 high-speed steel using cryogenic-treated Cu electrodes and reduced the surface roughness more compared to that achieved with the non-treated electrodes. Ishfaq et al. [11] compared cryogenic-treated and non-cryogenic-treated copper, brass, and graphite electrodes during the machining of Inconel 617. The authors concluded that cryogenic-treated electrodes outperformed non-treated electrodes by several folds. Similarly, the cryogenic treatment reduced the surface defects on Ti6Al4V as per Abdulkareem et al. [27] while they were evaluating surface roughness. Therefore, much of the literature favors the cryogenic treatment of tool electrodes to achieve a better surface integrity.

Conclusively, the literature survey reveals that the potential of surfactant-mixed dielectrics has not been comprehensively evaluated in the electric discharge machining of Ti6Al4V. In addition, not a lot of content is available on performance comparisons of different surfactants of varying hydrophilic–lipophilic balances (HLB) and their concentration during machining. The process efficiency in terms of surface quality is not primarily evaluated using cryogenically treated tool electrodes, which has been the most recommended solution to achieve superior surface integrity. Therefore, a comparative assessment of cryogenically treated tool electrodes using different surfactant-mixed dielectrics of varying concentrations has been carried out regarding surface integrity. A thorough three-phase experimentation framework (including an extensive preliminary, systematic, and validation protocol) was deployed using two cryogenically treated electrodes of aluminum and graphite with four categories of dielectrics, i.e., kerosene, span 20 (S-20) mixed with kerosene, span 60 (S-60) mixed with kerosene, and span 80 (S-80) mixed with kerosene. Three concentrations (12.5, 25, and 37.5 g/L) were used for the surfactants. The investigations were conducted using optical microscopy, scanning electron microscopy, energy dispersive spectroscopy, and statistical analyses. The optimally performing electrode mate-

rial, dielectric type, and concentration are proposed through a desirability function and were validated experimentally.

2. Materials and Methods

2.1. Machine Tool

In the current research, Ti6Al4V titanium alloy was employed as the workpiece. A sheet of $300 \times 100 \text{ mm}^2$ (length $\times$ width) was utilized for experimentation, having a thickness of 2.5 mm. The alloy was manufactured by BaoJi Titanium Industry Co Ltd., China, and the composition was verified through optical spectroscopy (as mentioned in Table 1) with the manufacturer sheet.

Table 1. Composition of the workpiece Ti6Al4V alloy.

Manufacturer Composition		Measured Composition	
Element	Weight (%)	Element	Weight (%)
C	<0.08	C	0.01
Al	5.5–6.75	Al	5.52
Fe	<0.4	Fe	0.20
V	3.5–4.5	V	4.18
H	<0.05	H	0.0042
N	<0.01	N	<0.01
O	<0.2	O	0.14
Ti	Bal	Ti	Bal

Electric discharge machining is an electro-thermal process where a series of electrical discharges melt and vaporize the material. Therefore, material removal is majorly dependent on the thermal and electrical properties of the workpiece and tool materials. The physical, electrical, and thermal attributes of the workpiece supporting the machinability through electric discharge machining and making the alloy preferable for various high-performance applications are presented in Table 2.

Table 2. Physical, electrical, and thermal properties of the workpiece Ti6Al4V alloy (open access CC BY-NC-ND 4.0 [3]).

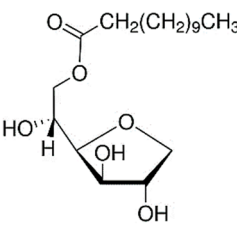
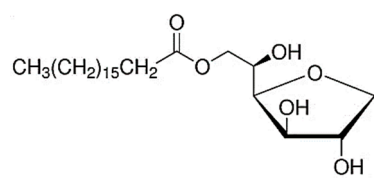
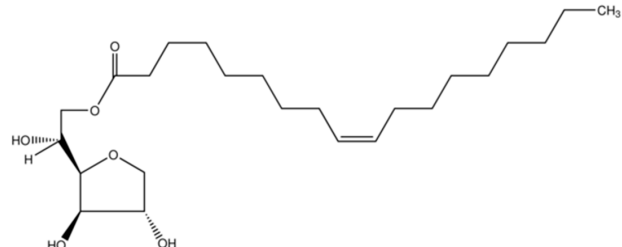
Mechanical Properties		Thermal Properties		Electrical Properties	
Fracture toughness (MPa m ^{1/2})	84	Thermal conductivity (W/mK)	6.7	Conductivity ($\Omega^{-1} \text{ m}^{-1}$)	5.61×10^5
Elastic modulus (MPa)	114	Thermal expansion coefficient (K^{-1})	8.7×10^{-6}	Resistivity (Ωm)	1.78×10^{-6}
Percentage elongation (%)	14	Specific heat (J/kg °C)	553		
Ultimate tensile strength (MPa)	862	Melting temperature (°C)	1604–1660		
Hardness (HRC)	36	Density (g/cm ³)	4.43		

In this research study, two electrode materials, aluminum (Al) and graphite (Gr), were employed to machine the Ti6Al4V workpiece because of their relative properties such as mechanical, thermal, and electrical ones. The choice of graphite as an electrode is made with a rationale to improve the surface integrity of the workpiece. Kashif et al. [11] demonstrated the excellent material removal performance of cryogenically treated graphite electrodes. However, as per the physics of the EDM process, a higher material removal rate is associated with poor surface integrity. Therefore, a thorough investigation of surface integrity is focused on herein. On the other hand, the study by Ahmed et al. [2] resulted

in a superior surface integrity using aluminum electrodes compared to that of graphite, leading to the choice of aluminum as an electrode for a comprehensive comparison.

The circular cavity depth was kept constant (200 μm) to investigate the electro-thermal erosion process. The electrodes were cryogenically treated to enhance the electro-thermal properties. Cryogenic treatment refines the microstructure and helps in enhancing the machining performance [11,27]. The cryogenic treatment was carried out at a temperature condition of 185 $^{\circ}\text{C}$. The treatment was conducted with liquid nitrogen as a cooling agent in an enclosed chamber for 24 h. It is anticipated that the cryogenic treatment affects the electro-thermal properties of the material, which significantly influence the discharge energy transfer. In addition to treated electrodes, a further step was taken to enhance the surface integrity by modifying the dielectric. Three hydro-carbon-based surfactants, namely span 20 (S-20), span 60 (S-60), and span 80 (S-80), manufactured by Hefei TNJ Chemical Co., Ltd., were used in different concentrations to enhance the dielectric properties. The surfactants lower the surface tension of the liquid and react with the dielectric as per the HLB value. The primary use of surfactants is in emulsifiers where high solution stability is required. However, the potential in EDM is unexplored, where debris removal is a prime focus in the tool–electrode interaction area. The physical properties of the surfactants that affect the dielectric properties and contribute to the EDM process are mentioned in Table 3.

Table 3. Physical properties of the surfactants (open access webpage [32]).

Properties	S-20	S-60	S-80
Chemical formula	C ₁₈ H ₃₄ O ₆	C ₂₄ H ₄₆ O ₆	C ₂₄ H ₄₄ O ₆
Molecular weight (g/mol)	346.46	430.62	428.60
Density (g/cm ³ at 25 °C)	1.032	1.056	1.068
Flashpoint (°C)	>110	185.3	186.2
HLB value	8.6	4.7	4.3
S-20	S-60	S-80	
			

A dedicated die-sinking electric discharge machine (Model: Rj-230 Creator, Taiwan) was used for the experiments with a customized flushing mechanism to ensure adequate dielectric emulsion with surfactants. The process schematic is shown in Figure 1, where a series of discharges melt and evaporate (plasma generation and removal) the material, leaving a circular cavity behind. The debris, bubbles, and melt material are present in the interaction area during machining action. During machining, the tool electrode and workpiece were immersed entirely in the dielectric. In addition, a consistent experimental protocol ensuring constant pressure, dielectric conductivity, and workpiece/tool positioning was used to rule out undesired variability in the evaluation process.

2.2. Design and Response Measurement

A three-phase experimentation plan (as presented in Figure 2) was formulated to investigate the process capability and surface formation. Initially, the first experimentation phase included pilot experiments (without a design of experiments—DOE). In this phase, an extensive number of trial experiments were carried out to highlight the suitability of constant and variable parameters. In addition, several parametric conditions were identified that were ineffective on the surface formation. The inappropriate settings resulted

in extensive spark interruption and irregular spark generation. Likewise, the excessive sparking at a concentrated surface area resulted in burn marks, which halted the machining cycle. The range of the machining conditions was tuned systematically to distinguish the effect of cryogenically treated electrodes with no apparent evidence of burn marks on the eroded surface. Among the electrical parameters, the pulse ON:OFF time performed well in controlling the discharge energy transfer to the surface. The rest of the electrical parameters were kept constant. It is pertinent to mention that pulse current is also a major factor that helps to rule out the productivity limitations of the process. Therefore, the roughening level of pulse current 15 A was considered to achieve the desired surface integrity with the modified process.

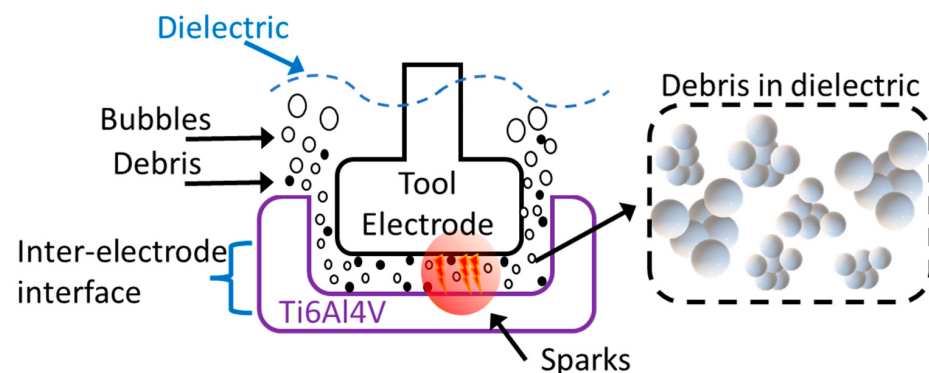


Figure 1. Schematic of the electric discharge machining process.

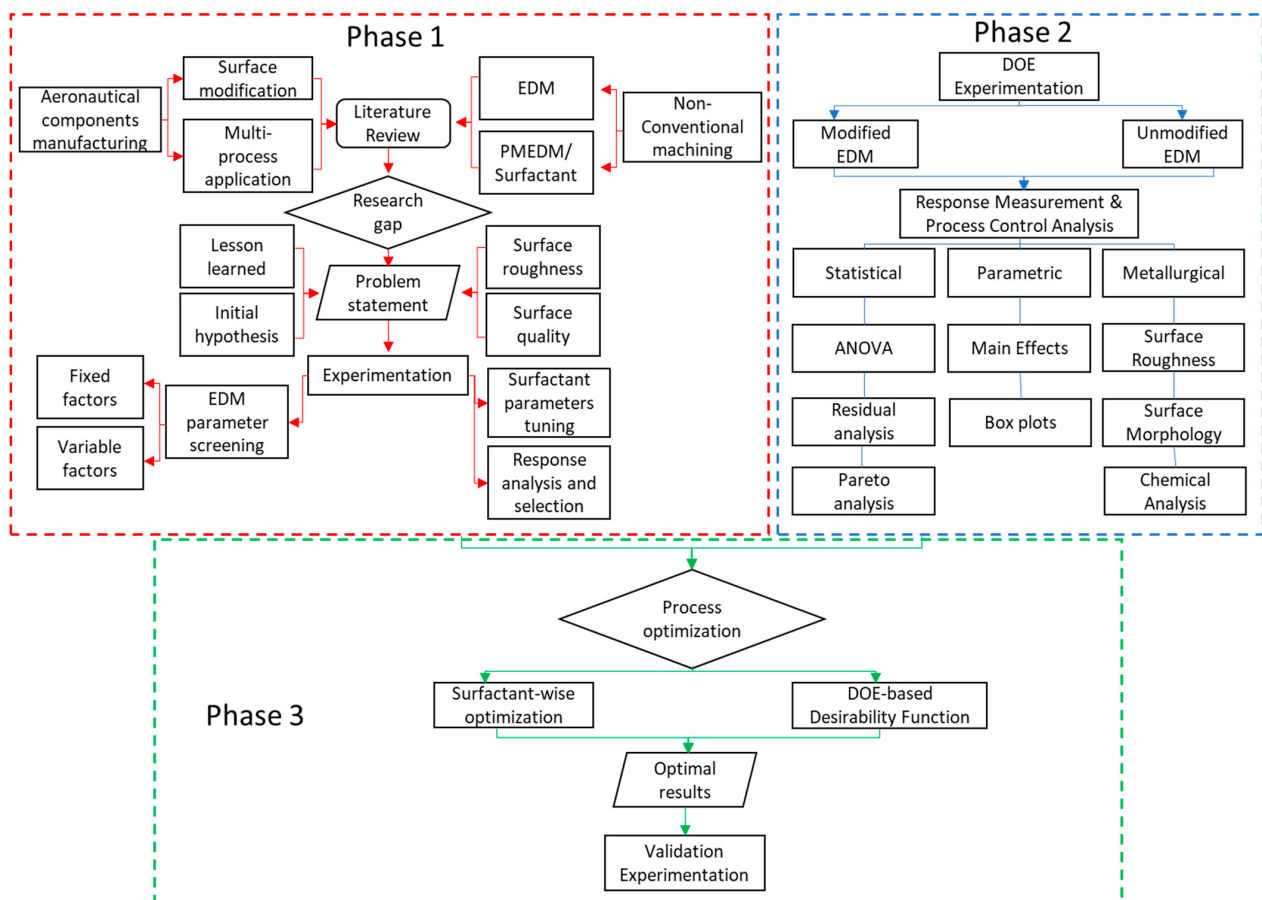


Figure 2. Three-phase experimentation framework.

As per the results from the first phase, the second experimental phase was planned. In addition to electrode types and electrical parameters, the surfactant type and their concentration were considered to be variable factors. The concentration was subdivided into three levels for a comprehensive exploration of dielectric chemistry.

In the second phase, a full factorial design of experiments (DOE) approach was applied, where six experiments were performed with the electrode, and the pulse ON:OFF time was taken as a baseline key performance indicator for surface integrity. With each electrode, 27 experiments were carried out. Each concentration of a specific surfactant included six experiments, making 18 experiments using each surfactant type. Thus, 60 experiments with two repetitions were performed to rule out the process variability. In addition, box plots are shown in the parametric control analysis to enhance the confidence of analysis. The input variables are shown in Table 4.

Table 4. Input control parameters for machining of Ti6Al4V.

Input Parameters	Symbols	Units	Levels		
			−1	0	+1
Surfactant type	S	-	S-20	S-60	S-80
Surfactant concentration	S _c	g/L	12.5	25	37.5
Pulse ON:OFF time	P _r	μSec	50:100	50:50	50:25
Electrode material	E _m	-	Aluminum	Graphite	

The surface roughness of the machined surfaces was measured in terms of the Ra parameter by a surface texture meter (Surtronic S-128, Taylor Hobson, Leicester, UK). For each sample, five consecutive values were recorded and averaged out. The cutoff length of 0.8 mm and an evaluation length of 4 mm were used for measuring the roughness. The statistical analyses include an analysis of variance, an analysis of means, and an analysis of residuals. The microscopical analyses include optical microscopic analysis, scanning electron microscopy, and compositional analysis. The surface morphology and composition were assessed using a Quanta 450 Field Emission Gun (FEG) scanning electron microscope equipped with Energy Dispersive Spectrometer (INSPECT S50). The performance analysis of the cryogenically treated electrodes was carried out using an OLYMPUS STM6-LM optical microscope. In the third phase, validity experiments were carried out to support statistical analysis. Scanning electron microscopic analysis is carried out based on surfactant type resulting in a superior surface.

3. Results and Discussions

3.1. Statistical Analysis

The analysis of variance is performed to investigate the influence of electric discharge machining parameters, tool electrode material, and surfactant characteristics. As per the quantitative methods, the data were thoroughly examined using the constant variance results, independence, and assumptions of normality. The linearity of the data was evaluated using the normal probability plot. The normality of the residuals is shown in Figure 3a. Furthermore, the coefficient of correlation was computed as per Looney and Gulledge (1985) at a 95% confidence level [29] and resulted in $R^2 = 98.59\%$ for 54 experiments with two repetitions. The correlation coefficient is significantly higher than the critical value of confidence level, indicating the satisfaction of the normal distribution of residuals.

Similarly, the residuals as a function of the order of data collection are shown in Figure 3a, which shows the randomness of the process and parametric effects on the design of experiments. The method also helps evaluate the residual's independence assumption, which is a baseline for the analysis of variance [33,34]. The desired outcome of the visualization is to identify any obvious patterns subjective to the experimenter's experience, which may potentially generate bias in data. Therefore, the irregularity affirms

the independence of data and satisfies the condition (also affirmed by the Durbin–Watson test of 54 residuals).

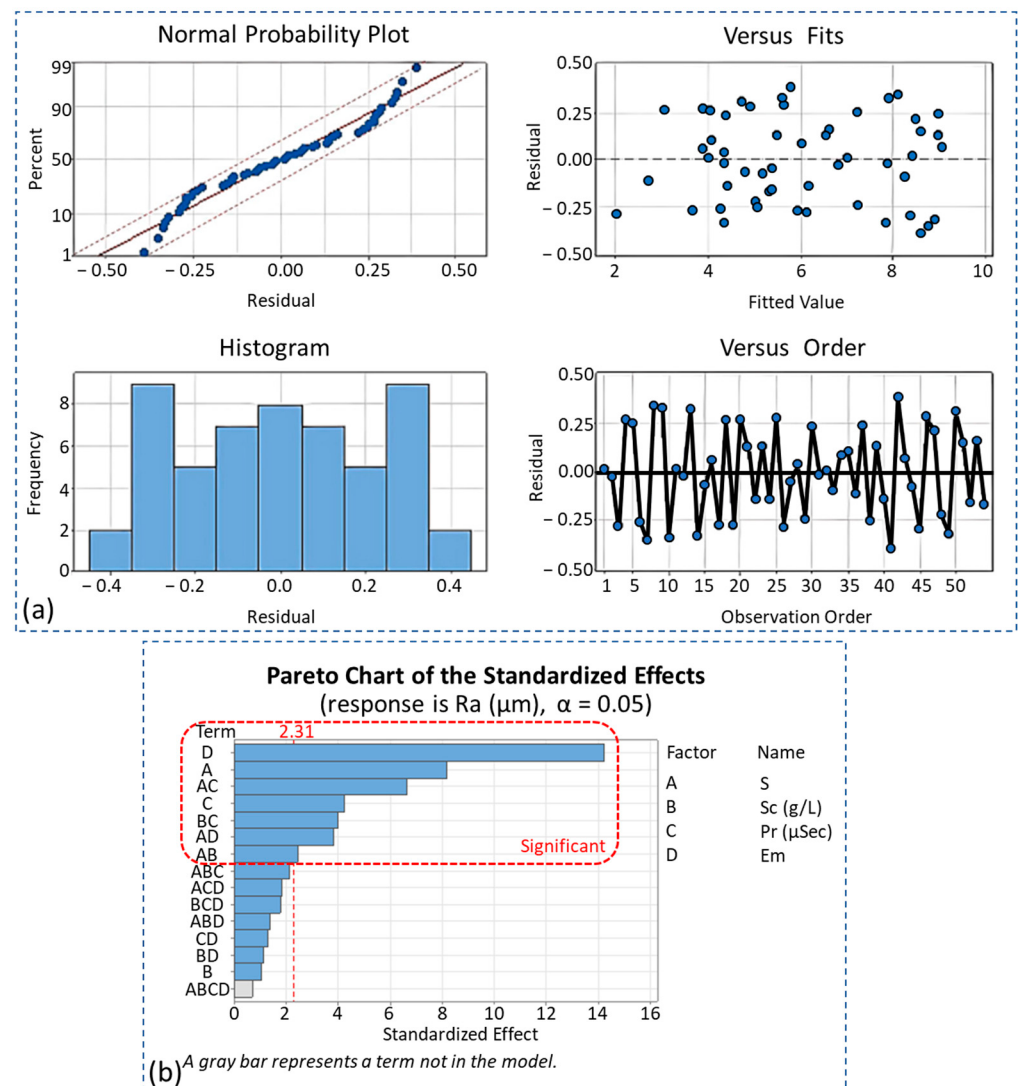


Figure 3. (a) Quantitative-based analysis on model adequacy and statistical confidence, and (b) Pareto chart for the significance of process parameters.

The residual versus predicted value plot is shown in Figure 3a. The plot typically tests the assumption of constant variance in data. The residual plot must be structureless to satisfy the assumption. The Brown–Forsythe test at a 95% confidence interval and the visual understanding of the plot affirms the satisfaction of the constant variance assumption.

The analysis of variance, a key statistical tool in assessing the significance of process variables on response characteristics, is carried out at a 95% confidence interval ($\alpha = 0.05$). The variable with a higher confidence than 95% (lower p -value than $\alpha = 0.05$) was considered to be significant, followed by two other criteria of 90% and 85% confidence in determining the weak significance. The variable with less than 85% confidence is classified as not significant. In addition to the p -value, the F -value is evaluated to determine the control factors. Moreover, the percentage contribution of the process variables is also computed (Equation (1)) to characterize the relative degree of significance.

$$\text{Percentage contribution} = \frac{\text{Adj SS}}{\text{Total SS}} \times 100 \quad (1)$$

The analysis of the variance of surface roughness obtained using cryogenically treated tools and the surfactant-dielectric-based EDM process is shown in Table 5. The significance is determined based on the p -value, F -value, and percentage contribution (PCR). The high F -value shows the sensitivity of the variable to the response characteristics.

Table 5. Analysis of variance of cryogenically treated tools and surfactant-dielectric based electric discharge machining process.

Source	DF	Contribution	F-Value	p-Value
Model	45	98.59%	12.46	<0.001 *
Linear	7	57.45%	46.68	<0.001 *
S	2	16.60%	47.22	<0.001 *
Sc (g/L)	2	0.46%	1.32	0.320
Pr (μ Sec)	2	4.75%	13.49	0.003 *
Em	1	35.64%	202.71	<0.001 *
2-Way Interactions	18	31.57%	9.97	0.001 *
S*Sc (g/L)	4	2.96%	4.21	0.040 *
S*Pr (μ Sec)	4	16.92%	24.06	<0.001 *
S*Em	2	3.91%	11.11	0.005 *
Sc (g/L)*Pr (μ Sec)	4	6.63%	9.42	0.004 *
Sc (g/L)*Em	2	0.51%	1.45	0.291
Pr (μ Sec)*Em	2	0.64%	1.82	0.224
3-Way Interactions	20	9.57%	2.72	0.074 **
S*Sc (g/L)*Pr (μ Sec)	8	4.40%	3.13	0.063 **
S*Sc (g/L)*Em	4	1.34%	1.90	0.203
S*Pr (μ Sec)*Em	4	1.94%	2.76	0.103 ***
Sc (g/L)*Pr (μ Sec)*Em	4	1.89%	2.68	0.109 ***
Error	8	1.41%		
Total	53	100.00%		

* 95% Confidence interval. ** 90% Confidence interval. *** 85% Confidence interval.

It is evident that the control variables such as surfactant type (p -value < 0.001), pulse ON:OFF time (p -value 0.003), and electrode material (p -value 0.001) are significant parameters with 16.60%, 4.75%, and 35.64% of the contributions, respectively. There are certain interactions of the process variables that have a significant effect on the response, as shown in Figure 3b. Mainly, two-way interactions such as surfactant type with its concentration (p -value 0.040), pulse ON:OFF time (p -value < 0.001), and electrode material (p -value 0.005) significantly control the response. Similarly, the joint effect of surfactant type and pulse ON:OFF time (p -value 0.004) significantly controls the surface roughness. The joint influences of the surfactant with electrode material controls and pulse ON:OFF time are 3.91% and 32.51%, respectively. The three-way interactions of surfactant type and its concentration with other variables were also analyzed, but these were found to have weak significance. However, these interactions affirm the combined effect of process variables on the responses. It is pertinent to mention that the accumulative influence of the surfactant type, concentration, and electrode material is fairly noticeable. Therefore, an optimized selection should be carried out while designing the system.

3.2. Process Control Analysis

3.2.1. Unmodified Process Analysis

The surface integrity produced through the electric discharge machine is a function of crater characteristics such as shape and size. These overlapping craters result in the overall roughness of the machined surface. A better surface roughness results from shallow craters as compared to that caused by deep craters. Furthermore, the formation of surface texture differs with the engagement of dissimilar materials as electrodes. This engagement results

in varying micro-depth impressions [35]. During the electro-erosion process, poor debris evacuation also contributes to the arcing phenomenon, which deteriorates the work and tool surfaces. The discharge strength is affected by the presence of a sedimentary layer of debris in the interaction zone. Therefore, this results in a significant variation in surface roughness. This highlights the importance of having an efficient/balanced flushing time (pulse OFF time) during which the debris (minute particles) is flushed away. The pulse-off time is linked to the process' flushing attribute, which is crucial for energy transfer [34]. If the off-time is shorter, the incomplete insulation recovery and discharge instability will result in deteriorated results. The parametric effect of pulse ON:OFF time on surface roughness (R_a) is shown in Figure 4.

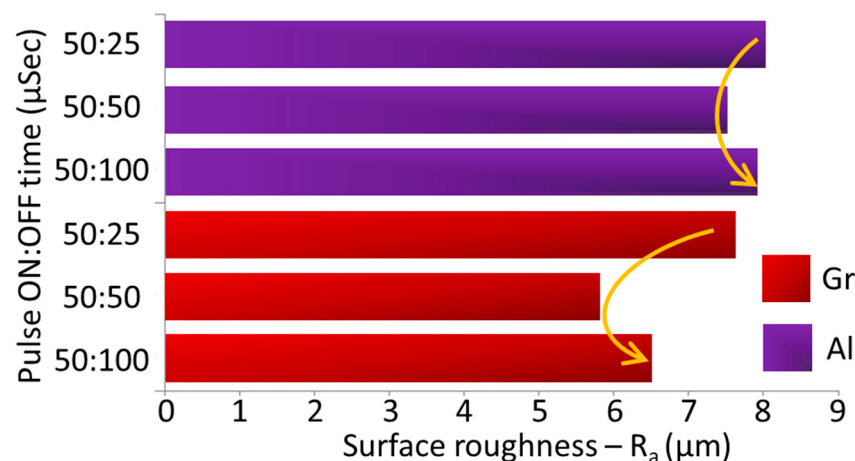


Figure 4. The parametric trend of surface roughness with kerosene as dielectric.

In the case of graphite, it can be observed from the trend line that a decrease in the pulse-OFF time results in a coarser impression compared to that of a higher pulse-off time. The balanced pulse ratio (50:50 μSec) resulted in minimum roughness with both electrodes ($\sim 7.5 \mu\text{m}$ for aluminum and $\sim 5.8 \mu\text{m}$ for graphite). The aluminum electrode produced more roughness as compared to that of the graphite electrode. The longer pulse-ON time (stimulated by the high electrical conductivity of aluminum $35 \times 10^6 \text{ S/m}$) introduces a high density of electric sparks, inducing heat energy transfer to the surface. Since both of the tools are cryogenically treated, which assists in thermal conduction, the graphite (400 W/m K) behaves better in thermal conduction than aluminum does (205 W/m K), resulting in a lower degree of roughness. The electrical energy generates heat on the surface, which melts the material. Thermal conduction inside the tool results in the dissipation of heat energy, which is not efficiently carried out in aluminum. Therefore, an increased material melt pool is created, leaving a deeper crater behind upon flushing the melt. A similar physical mechanism is endorsed by Kumar et al. [26]. The aluminum generally produced a coarser surface at all pulse ratios. The flushing action improved by decreasing the pulse ratio, resulting in a superior surface finish.

The surface morphology resulted from pure kerosene dielectric (Figure 5) at a pulse ON:OFF time of 50:50 μSec using the graphite electrode shows evidence of debris redeposition (especially carbon due to inefficient flushing [2]), craters, and spherical modules. The redeposition of debris results in a recast layer on the surface, limiting the surface integrity. As established earlier, the crater size drives surface integrity, which is a function of the melting point of the tool electrode [35]. Therefore, higher melting point results in a smoother surface with a relatively smaller crater volume. The mid values of pulse ON:OFF time and graphite's higher melting point and thermal conductivity resulted in better surface characteristics at the same melting and flushing timings.

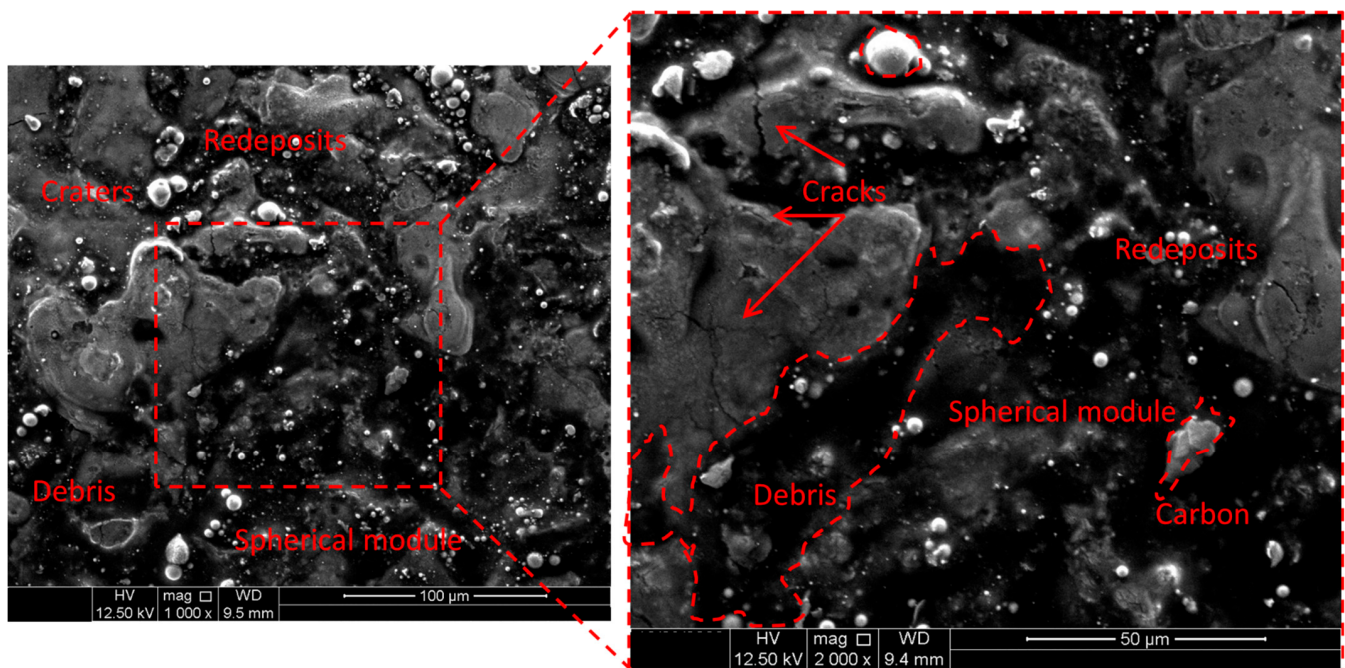


Figure 5. Surface morphology of kerosene-based dielectric at pulse ON:OFF time of 50:50 μ Sec and using graphite electrode.

3.2.2. Modified Process Analysis

The modified process is based on the kerosene mixed with surfactants. The single-factor analysis of variance shows that electrode material, surfactant type, and pulse ratio are the most significant parameters in controlling surface roughness. The percentage contributions of the process parameters are mapped in Figure 6a, with tool electrode material and surfactant type showing 35.64% and 16.60% control of the surface roughness, respectively.

Tool Electrode Analysis

The analysis of electric discharge-eroded craters using different electrode materials enrich the understanding of the machining mechanism. It is evident, in Figure 6b, that the surface roughness values obtained by the aluminum electrode and graphite electrode materials are around $\sim 7.8 \mu\text{m}$ and $\sim 4.5 \mu\text{m}$, respectively. The microstructures of the machined impressions on specific tool electrodes are shown in Figure 6c,d. The stochastic nature of the process forms a plasma channel, generating a collapsing effect of the craters. As a result, circular-shaped and interconnected craters are formed because of the ejection of molten metal from the center. The craters' formation and overlapping result in a unique surface texture using varying parametric conditions, specifically tool electrodes [2]. The discharge energy transferred to the workpiece corresponding to the electrode material (Al or Gr) significantly affects the surface texture, as evident from Figure 6c,d.

The cryogenic treatment refines the microstructure and improves mechanical properties such as fracture toughness and wear properties [30]. The improvement is driven by the elimination of residual stresses produced during the production of tool electrodes [11,31]. Therefore, the intrinsic properties of the tool electrode are important in generating a uniform surface texture. Several features of craters are presented in Figure 6, such as large-sized, deep, or shallow craters and small-sized, deep, or shallow craters. The collapsing of plasma and the overlapping of craters, making a spongy-like structure, is evident in Figure 6d, which were processed using the graphite electrode. The shallowness of small-sized craters and redeposited debris resulted in less surface roughness. On the other hand, the deep and large-sized craters are evident on the surface (Figure 6c), which were processed using an Al electrode, due to excess heat energy stimulated by the high electrical conductivity of aluminum ($35 \times 10^6 \text{ S/m}$). In addition, the Al electrode possesses a low melting tempera-

ture of 660 °C, which translates into the erosion of the electrode surface, which is copied on the workpiece surface, leading to poor surface integrity. However, in the case of the Gr electrode, a higher melting point drives the erosion process to a small crater volume and superior surface finish. The interconnected craters (in the case of the Gr electrode) result from uniform sparking, where a spongy surface texture causes less machined surface roughness. It is evident from Figure 6, the cryogenic-treated Gr electrode results in a better surface texture ($\sim 4.5 \mu\text{m}$) with smaller and interconnected crater boundaries.

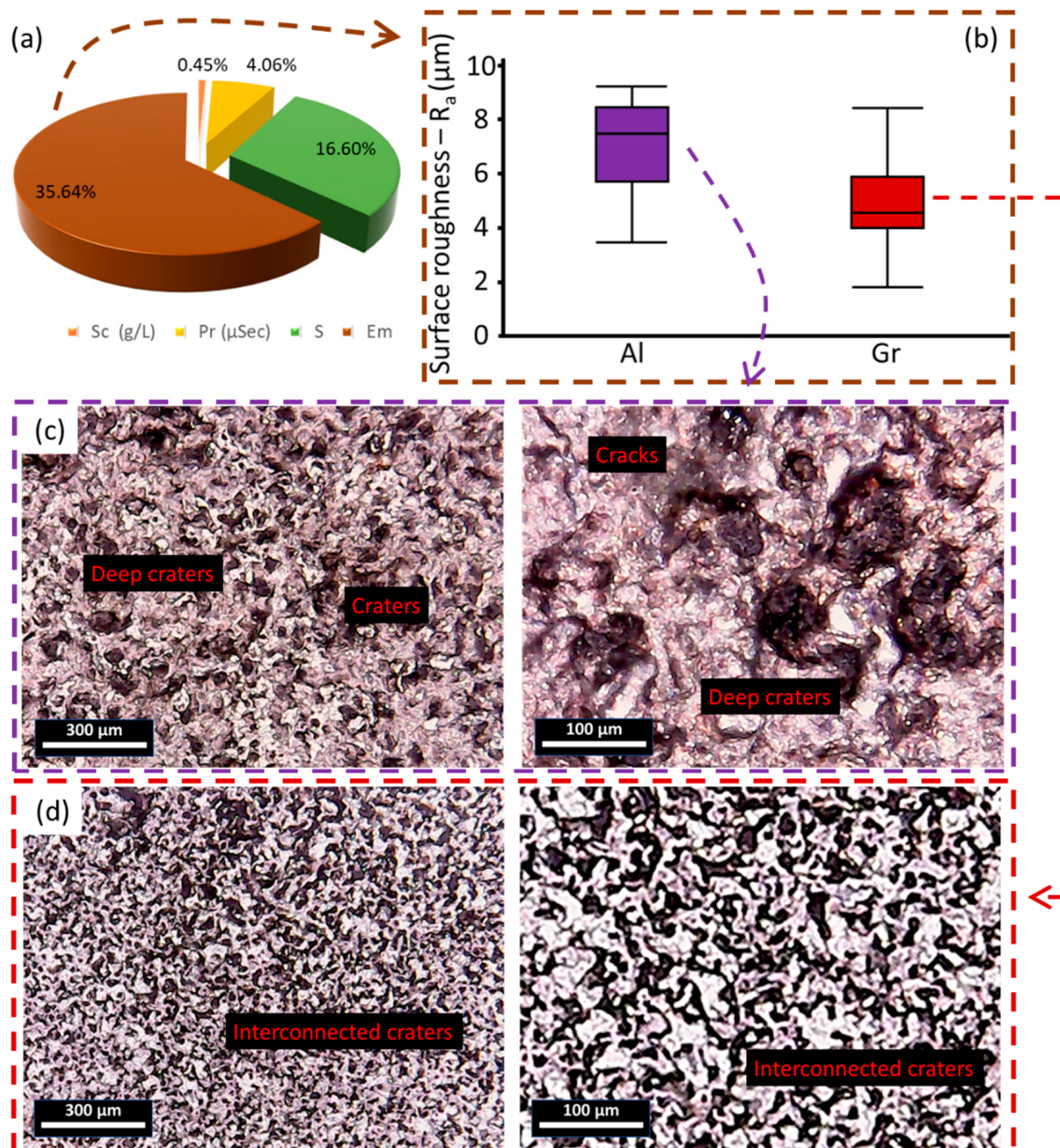


Figure 6. Tool electrode analysis during modified process; (a) percentage contribution for controlling modified process; (b) effect of tool electrode material; (c) micrograph of kerosene with 12.5 g/L of S-60 using Al electrode; (d) micrograph of kerosene with 12.5 g/L of S-60 using Gr electrode.

The surfactant-mixed dielectric increases the flushing ability because of the effects of the hydrophilic group absorbing debris and carbon dregs and the hydrophobic tail extending towards the dielectric fluid [25]. The pulse ratio also controls the surface texture along with the electrode material, as evident in Figure 7. The increase in OFF time is beneficial in terms of reducing surface roughness. The increase in pulse ratio (50:25 μSec ,

where the pulse on-time is twice as long as the pulse off-time) yields a higher amount of discharge energy. A higher discharge energy with a shorter off-time results in an excessive liquid phase at the periphery of the crater, thus increasing the plasma channel volume. Thus, deeper craters are formed, which are translated into a poor surface finish. It is pertinent to mention that the surface quality produced with a cryogenic-treated graphite electrode is relatively better than that achieved with an aluminum electrode. This behavior is attributed to the higher thermal conductivity of the graphite electrode (400 W/m K) in comparison to that of the aluminum electrode (205 W/m K), which plays a pivotal role in defining the electrode wear characteristics. Less thermal conductivity promotes the electrode's surface erosion because of inadequate dissipation of the inter-electrode interface energy. This poor dissipation results in intense localized heating, generating deep craters. Ishfaq et al. [35] experienced a similar phenomenon in the machining of D2 steel.

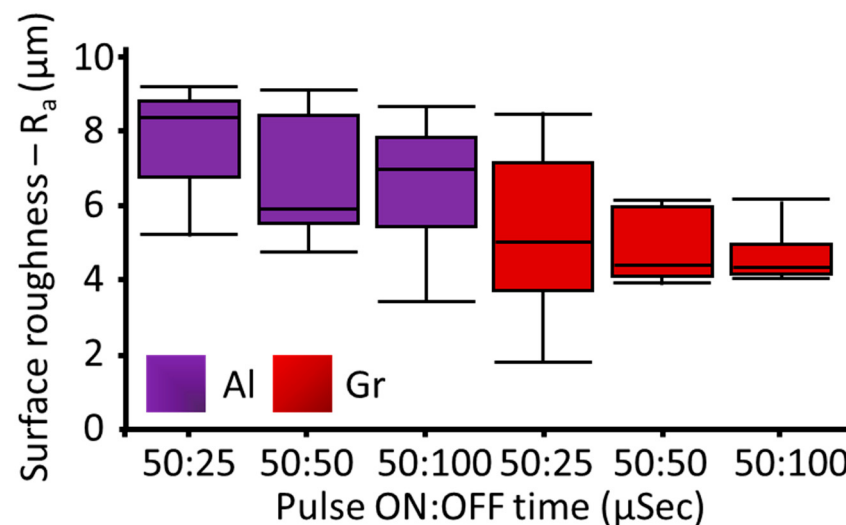


Figure 7. The parametric effect of cryogenic-treated electrodes and pulse ON:OFF time on surface roughness of surfactant-mixed dielectrics.

In the case of the Gr electrode, a higher melting temperature (3300 °C) of the electrode melts a larger proportion of the workpiece (which has a lower melting temperature (1604 °C)). At higher pulse ratio and current values, intense heating causes melting and vaporization at the inter-electrode. Therefore, more of the workpiece material is eroded compared to that of the electrode due to differences in the melting points. At the same time, a lower pulse ratio drives low energy transfer and more flushing activity. This balanced approach (aided with a refined microstructure, where the grain size supports the sparking attributes) results in superior surface roughness, yielding connected craters. The third level of significance of the pulse ratio and electrode material's combined effect is evident in Table 5 for controlling the output. A comparison of the roughness obtained at a low pulse ratio (50:100 μSec) with the aluminum electrode and the high pulse ratio (50:25 μSec) with the graphite electrode shows that they are similar to each other. Aluminum has a high density, a low melting temperature, and poor thermal conductivity. At the same time, graphite has loosely compacted atoms (a lower density) and a higher thermal conductivity and melting temperature, which support the electro-erosion process of generating a superior surface integrity. A similar scientific process is endorsed by Ahmed et al. [2] during the machining of Ti6Al4V for selecting a preferable electrode made from graphite, aluminum, brass, and copper. Therefore, a low pulse ratio and graphite electrodes are preferred to produce minimum roughness on the workpiece.

Surfactant-Mixed Dielectric Analysis

The process is modified by employing surfactant-mixed dielectrics. The performance of each dielectric using both electrodes and the effect of concentration are evaluated. In

comparison to the SEM micrograph obtained of the kerosene dielectric (Figure 5), the amount of debris and carbon deposition are reduced for the modified dielectrics, as shown in Figure 8. The surface contains significant contents of debris (carbon content), redeposits, and spherical modules, which are slightly reduced as compared to those of the pure kerosene-based dielectric. The improvement of surface integrity by using a surfactant is also endorsed by Wu et al. [24] while machining SKD61 steel. The range of surface roughness obtained with modified dielectrics is smaller than that of kerosene-based processing (evident from the comparison of Figure 9 with Figure 4). The cryogenic-treated graphite electrode produced a superior surface finish as compared to that of cryogenic-treated aluminum for all surfactant types. For instance, the S-20-mixed dielectric made of aluminum resulted in $\sim 7 \mu\text{m}$ roughness, whereas graphite resulted in $\sim 4.3 \mu\text{m}$ roughness in the same conditions. However, both electrodes in the modified dielectrics presented a $\sim 25\%$ improvement compared to that of the kerosene dielectric. The surfactant enhanced the dielectric's conductivity and decreased the surface tension for adequate melt and debris removal. In addition, the surfactant's presence caused debris dispersion and restricted agglomeration [18]. This mechanism contributes to improving the surface characteristics. In addition to the improvement in dielectric attributes, the properties of electrodes also affect the surface, as their two-way interaction is significant, with a p -value of 0.005.

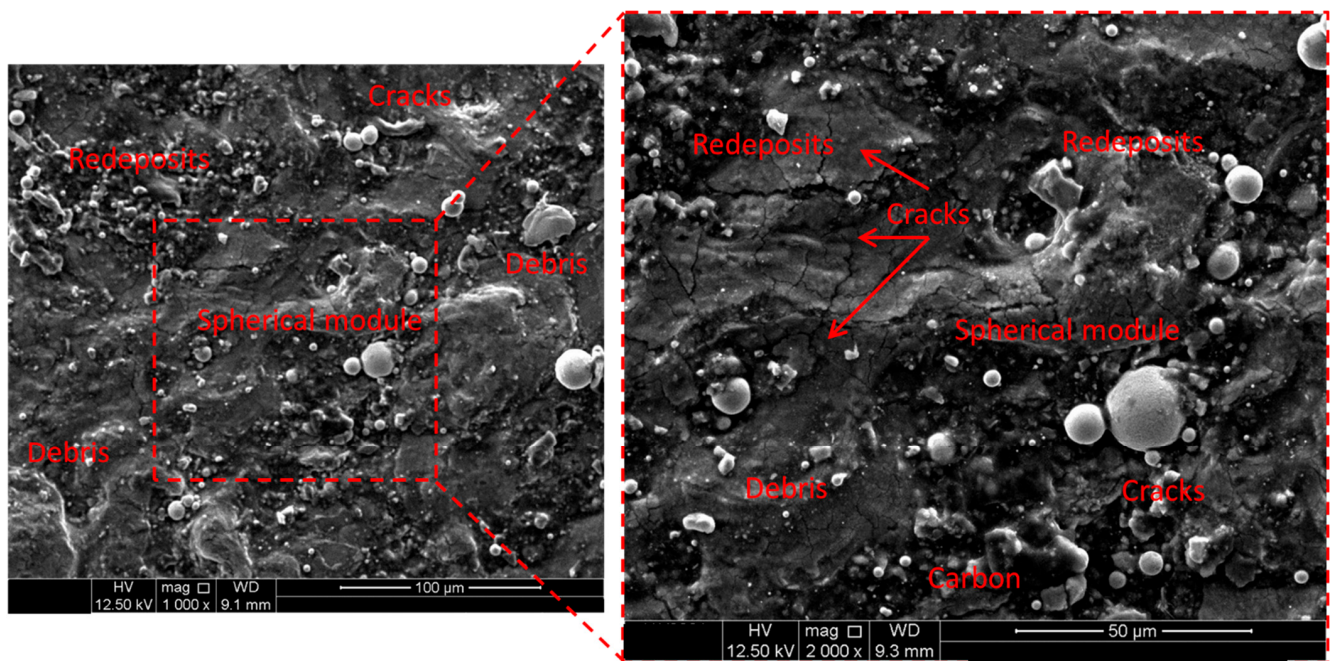


Figure 8. Surface morphology of kerosene-mixed S-20 dielectric at 37.5 g/L, pulse ON:OFF time of 50:100 μSec , and using the aluminum electrode.

A visible difference is apparent in the surface roughness in Figure 9 for each surfactant type. The performance of the surfactant during machining significantly depends on the hydrophilic–lipophilic balance, which determines the EDM-assisting mechanisms. These mechanisms include lowering the surface tension for better debris evacuation and reacting with debris. The S-60-mixed dielectric produced better results as compared to those of the other surfactant types because of the synergistic effect of HLB and parametric conditions. The aluminum electrode produced $\sim 6 \mu\text{m}$ roughness, and graphite resulted in $\sim 4 \mu\text{m}$ roughness with S-60. The machined surface obtained using this dielectric is shown in Figure 10. The influence of lowering the surface tension of the dielectric medium facilitates an effective cutting mechanism. Such as S-60, the cryogenic-treated graphite resulted in $\sim 5.3 \mu\text{m}$ roughness, and the cryogenic-treated aluminum electrode produced $\sim 8.2 \mu\text{m}$ using the S-80-mixed dielectric. Overall, the graphite electrode showed better results in

all modified dielectrics because of the balanced energy transfer resulting from low density (1.7 g/cm^3) and electrical conductivity ($0.3 \times 10^6 \text{ S/m}$). It is pertinent to mention that the performance of both graphite and aluminum electrodes is comparatively better with the S-60 surfactant. The surface integrity is highly affected by the melting and vaporization of the metal and the removal of unwanted debris and melt. In this regard, the effect of the tool material, heat treatment in refining the grain size, and surfactant properties altogether enhanced the surface quality. The effect of surfactants on material removal has been supported by Dzulkifli et al. [36].

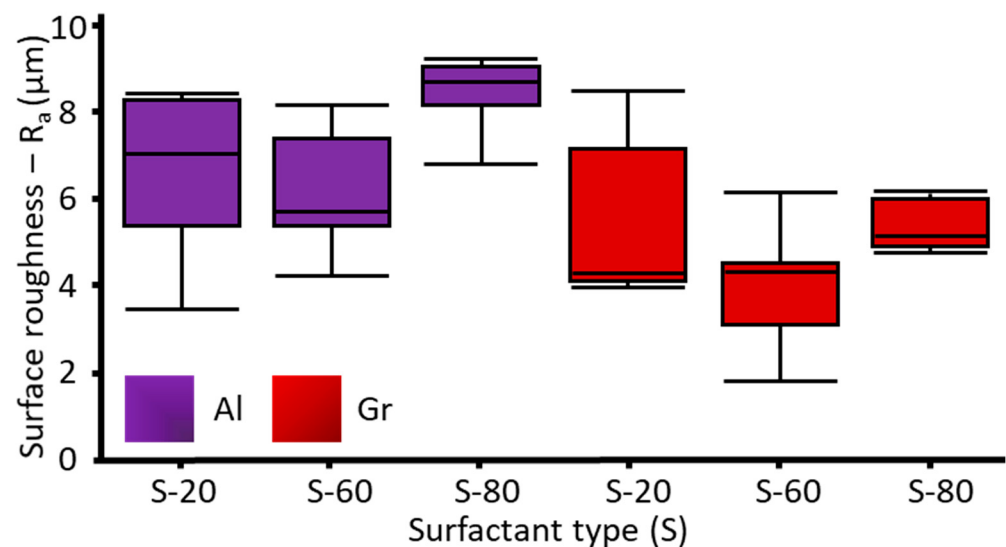


Figure 9. The parametric trend of surface roughness of surfactant type and electrode material (analysis of means of concentrations).

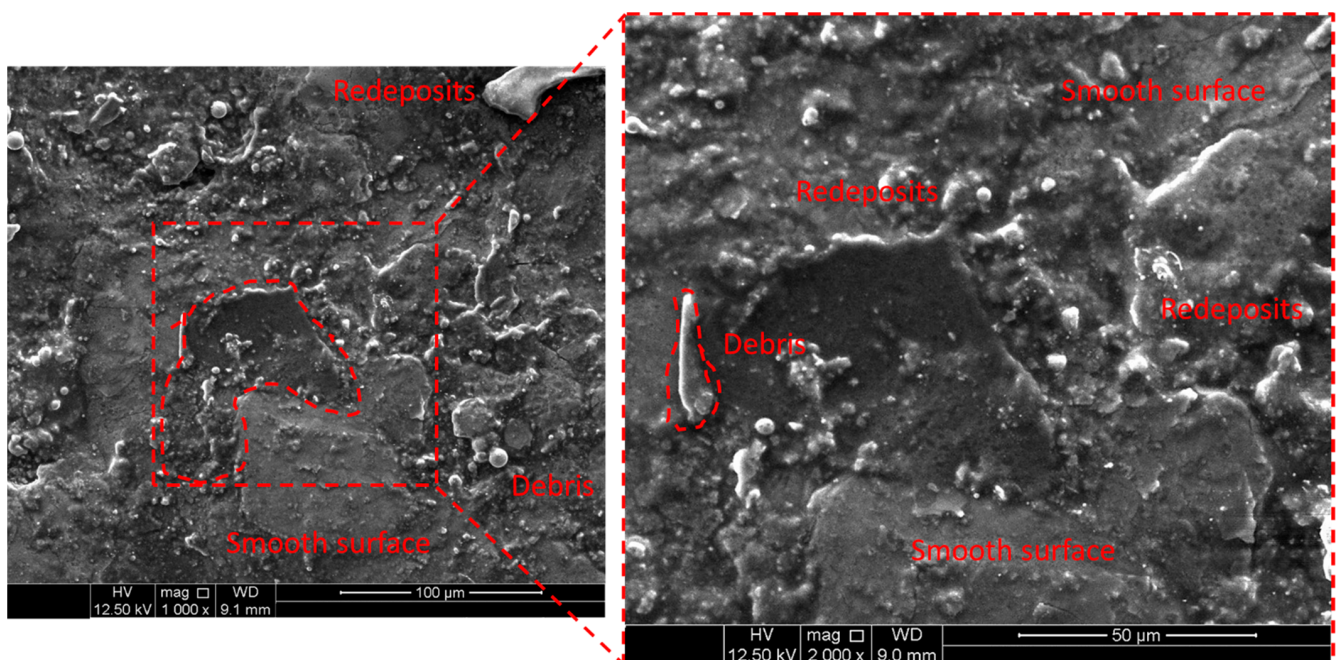


Figure 10. Surface morphology of kerosene-mixed S-60 dielectric at 25 g/L, pulse ON:OFF time of 50:100 μSec , and using graphite electrode.

The roughing parametric conditions (high value of pulse current) are the root cause of the surface defects. Since electric discharge machining is a slow process [35], aggressive parametric conditions are used to achieve a higher MRR, which deteriorate the surface.

The dielectric's function is not only to remove the melt material, but also to facilitate the machining process through improved conductivity. The kerosene-based dielectric possesses low viscosity and thermal conductivity, limiting the process efficiency and lowering the machined surface quality [36]. This is why more surface roughness is achieved using the kerosene-based dielectric with both electrode materials. Surface defects such as deep craters, excessive cracks, debris redeposition, carbon content, and spherical modules are visible in Figure 5. Chen et al. [37] highlighted that the dissemination of carbon occurs because of the use of kerosene for machining. The elevated temperature during the machining action decomposed kerosene into carbon that stuck to the workpiece and tool electrode, leading to compromised EDM performance. On the other hand, the surfactant-mixed dielectric produced a superior surface quality with reduced numbers of cracks, craters, and less redeposited debris, as evident in Figure 8 for S-20, Figure 10 for S-60, and Figure 11 for S-80.

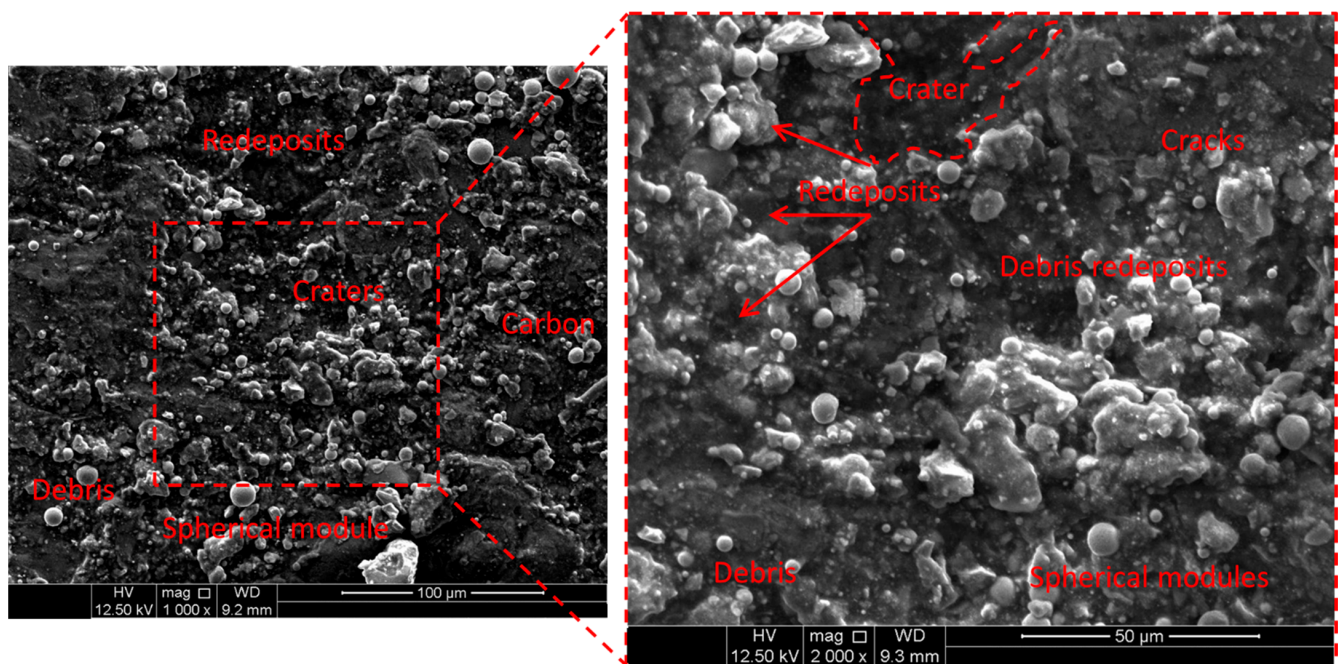


Figure 11. Surface morphology of kerosene-mixed S-80 dielectric at 37.5 g/L, pulse ON:OFF time of 50:100 μ Sec, and using graphite electrode.

For the S-20-mixed dielectric, the flashpoint is low $<110^{\circ}\text{C}$, which reduces the functionality in terms of melt removal. However, it has higher conductivity as compared to that of kerosene, which helps in achieving a superior surface quality. On the other hand, S-60 and S-80 have higher flashpoints, 185.3°C and 186.2°C , respectively, which help in improving both the functions of debris removal and enhancing thermal conductivity. The S-60 dielectric resulted in around $\sim 18\%$ improvement of the surface finish compared to that of S-20. Similarly, S-60 resulted in $\sim 35\%$ reduction of roughness compared to that of S-80 in terms of the aluminum electrode. With the reduction of the hydrophilic–lipophilic balance, the functionality of the surfactant increases in an oil-based environment. S-20 has the highest hydrophilic–lipophilic balance (HLB) of 8.6, which makes it partially soluble in oil. However, S-60 and S-80 have lower HLB values in the range of four to five, which enhances the solubility in oil. Solubility in oil is directly linked to a reduction of surface tension and enhancing the properties of oil. S-80 has the lowest HLB value of 4.3, which assures the mixing of surfactant at a higher degree than those of other alternatives. However, the solubility of S-20 surfactants is lower in kerosene-based dielectrics, producing excessive defects on the surface.

Compared to the surface produced by the S-20, S-60 achieved a better surface finish. Due to the imbalanced EDM process, a high number of cracks, craters, and more redeposited

debris are evident on the S-20-based machined surface. The performance of S-20 is limited by a low flash point in the machining zone and a high HLB value (which is favorable for water solubility), thus compromising the thermal conductivity of the dielectric. When the graphite tool electrode is engaged, the surface roughness obtained using S-20 is $\sim 4.5 \mu\text{m}$, which is $\sim 35\%$ lower than that which is achieved using the aluminum electrode. Since the functionality of S-20 is partially effective in kerosene, the refined grain structure of graphite helps in reducing the energy transfer on the surface for material removal. In addition, graphite, a relatively more thermo-conductive electrode, disseminates the energy from the inter-electrode interface. This dissemination of energy reduces the intensity of sparking and enhances the surface quality. The reduction of spark intensity by cryogenically refined grains was experienced during the machining of Inconel 617 by Ishfaq et al. [11]. Similarly, graphite electrodes achieved almost similar results using the S-60-mixed dielectric. However, the surface roughness obtained using S-60 was 11% lower than that achieved using S-80.

Micrographs of the surface morphology of the kerosene-mixed S-60 dielectric at 25 g/L concentration, 50:100 μSec pulse ON:OFF time, and using the graphite electrode are shown in Figure 10. An improved surface was obtained using S-60 with a reduced degree of redeposited debris, spherical modules, and cracks as compared to those when S-80 was utilized (see Figures 10 and 11). This is because S-60 improves the dielectric conductivity because of the adequate HLB value. In contrast, a lower HLB value (4.3) of S-80 creates an imbalanced machining action mechanism due to the excessive thermal conductivity of the dielectric. The process requires a synergistic relationship between the discharge energy and dielectric conditions. Excessive thermal conductivity also reduces the surfactant's efficacy [15,23]. For instance, in the case of the Al electrode, S-80 resulted in $\sim 22\%$ and 34% increases in the Ra values compared to those of the S-20 and S-60 dielectrics, respectively. Additionally, in the case of the Gr electrode, a 15% deteriorated surface roughness was recorded for S-80 compared to that of the S-60-based dielectric. Therefore, a balanced choice is preferred with an optimized combination of electrode material and surfactant type. This is because when the surface tension of a dielectric is adequately reduced, a balanced sparking phenomenon occurs in the inter-electrode interface due to higher conductivity and efficient debris removal.

A high number of spherical modules are evident in Figure 11 on the machined surface using the kerosene-mixed S-80 dielectric at 37.5 g/L, pulse ON:OFF time of 50:100 μSec , and using a graphite electrode. The spherical modules are caused by the low surface tension of melt liquid. The S-80 surfactant possesses the lowest HLB value (4.3), which makes it highly dissolvable in kerosene dielectric to ease debris movement. However, this phenomenon leads to the removal of solidified melt material as well. Since the flashpoint of 186.2°C is significantly lower than the melt temperature, most of the surfactant is evaporated without reacting with the melt. However, molecules of the surfactant react with the solid debris and discourage their agglomeration. Wu et al. highlighted the phenomenon of reaction between the surfactant and debris [22]. Similarly, the findings are supported by Ishfaq et al. [11]. The removal of solid debris from the surface increases free space for melt material movement, enhancing the plasma channel. Therefore, this movement advances the possibility of spherical module formation, which is evident in Figure 11.

The agglomeration and dispersion phenomena of debris are illustrated in Figure 12, where kerosene-based dielectric debris tends to agglomerate and negatively affect the process. This agglomeration promotes the redeposition of debris and carbon attachment on the electrode and workpiece surface, thus affecting the sparking phenomenon. The imbalanced sparking phenomenon reduces the discharge density and energy transfer on the surface, which deteriorates the EDM surface. On the other hand, the surfactant-mixed dielectric reacts with debris and avoids accumulation [24]. Moreover, the conductivity of the dielectric is improved by the surfactant's inherent properties, and the surface integrity is positively affected. The surfactant's HLB value and flashpoint are pivotal in defining the machined sample's surface characteristics. The temperature at the interface of electrodes

during machining is significantly high, which limits the efficiency of surfactants in that region. Therefore, to eradicate the limitation of flashpoint, cryogenic-treated electrodes are used, which generate balanced discharges and transfer heat energy to the inter-electrode interface during machining action.

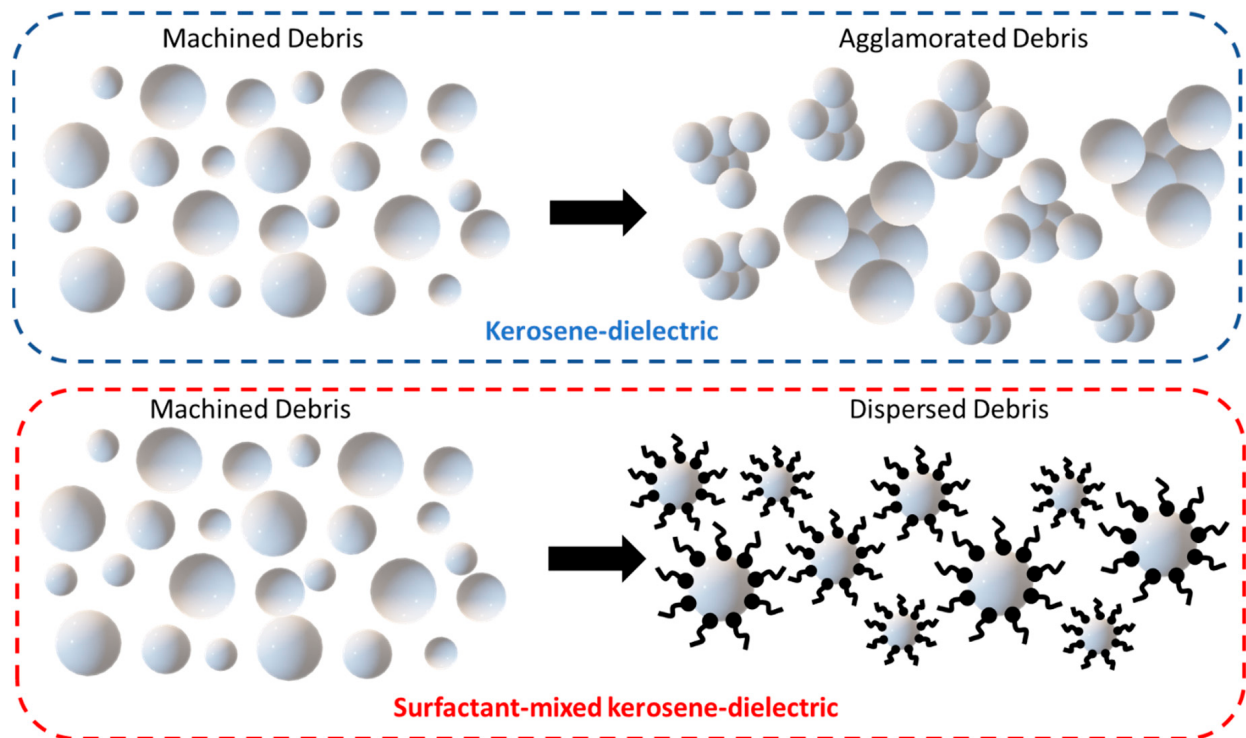


Figure 12. The agglomeration and dispersion phenomena of debris using kerosene dielectric vs. surfactant-mixed kerosene dielectric.

The parametric effect of the surfactant concentration on surface roughness is shown in Figure 13. S-60 has achieved a better surface finish at 12.5, 25, and 37.5 g/L concentrations as compared to those of the other surfactants. It is evident from the statistical analysis that surfactant type affects surface roughness significantly, with a p -value < 0.001 . Moreover, the two-way interaction of surfactant type with its concentration also significantly controls the surface roughness, with a p -value of 0.040 and a 95% confidence interval. The roughness values obtained at 12.5 g/L and 25 g/L of S-20 lie in a similar range of $\sim 6 \mu\text{m}$. However, a further increase in concentration to 37.5 g/L reduced the surface roughness by around 30%, making it $\sim 4.2 \mu\text{m}$. The decrease is caused by increased S-20 proportion in the dielectric, which facilitated the conductivity improvement. In addition, a higher surfactant content enhanced the oil–surfactant emulsion, which aided in debris removal, as evident in Figure 12.

The surface roughness achieved using S-60 is in a similar range of that of the higher concentration (37.5 g/L) of S-20. However, the mean roughness at the 12.5 g/L and 25 g/L concentrations is significantly higher than the roughness at the 37.5 g/L concentration with S-20. These precise results highlight the significance of the results and the supremacy of surfactant properties (surfactant type and concentration affecting dielectric properties). On the other hand, the increase in the concentration of S-60 from 12.5 g/L and 25 g/L resulted in similar roughness values. However, the variability of roughness values increased at higher concentrations 37.5 g/L because of the instability of the process. A similar trend was observed in the case of S-80, where a lower concentration of 12.5 g/L produced more surface roughness. The increase in S-80 concentration from 12.5 to 37.5 g/L resulted in a 17% reduction of surface roughness. This reduction is because of the increased content of a low-HLB surfactant in the dielectric. The upsurge of 200% (from 12.5 to 37.5 g/L) in

the concentration of low-HLB (4.3) S80 increased the conductivity of the dielectric and improved the reaction tendency with debris. The presence of a surfactant enhances the flow characteristics for the efficient removal of melted droplets, generating fewer surface asperities and defects [22]. Conclusively, a lower HLB value (for better solubility in oil) and the surfactant's concentration affect the processing. At higher surfactant concentrations, the surfactant molecules react with debris in a high proportion, enhance the conductivity of dielectric by several folds, and decrease the surface tension for better melt-flushing action. However, an unbalanced increase in dielectric properties promotes the arcing phenomenon, which deteriorates sparking activity. Therefore, a balanced approach is required to select the surfactant type considering the flashpoint and HLB value [11] (because the hydrophilic group attaches to debris, as shown in Figure 12). The HLB value directly affects conductivity and dissolubility in kerosene oil.

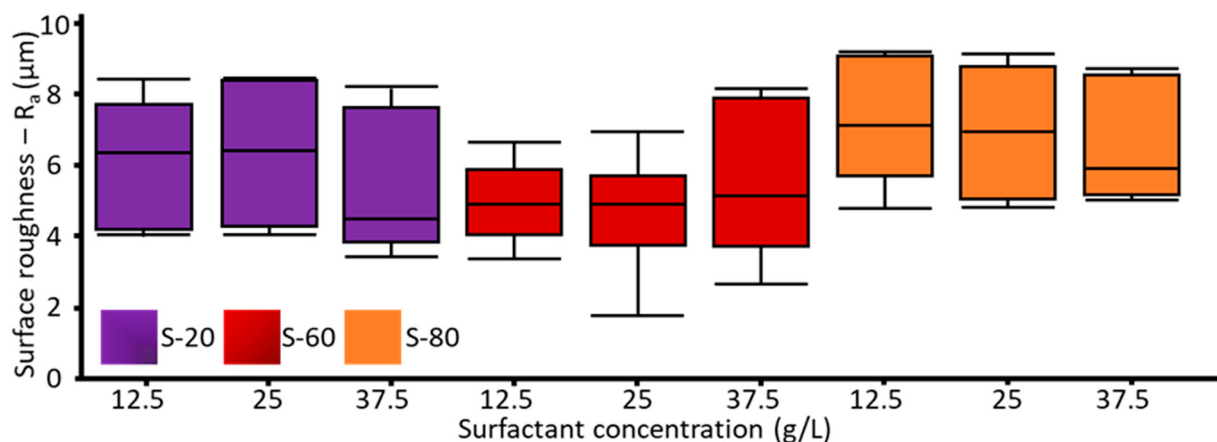


Figure 13. The parametric trend of surface roughness concerning surfactant type and concentration.

SEM analysis of the machined surfaces shows evidence of spherical modules and redeposited debris for both kerosene-based and S-60-based dielectric conditions (as evident in Figures 5 and 10, respectively). The energy dispersive spectroscopy (EDS) analysis results in Figure 14a highlights the deposition of around 38% carbon and around 39% oxygen on the surface due to the carbides and oxides generated during machining action using the kerosene-based dielectric. The carbon content significantly differs from the base material, as presented in Table 1, where it was merely 0.01%. Carbon originates from the decomposition of kerosene and surfactant, as both of them are hydrocarbon-based dielectrics. However, using the S-60-based dielectric (see Figure 14b), the carbon and oxygen contents are significantly reduced to 24% and 17%, respectively. The significant reduction of carbon content supports the effective removal of debris and improvement of the electro-erosion process.

3.3. Process Optimization and Validation

The process optimization was carried out through a desirability function, where a certain objective was defined within the available experimental design. Since a full factorial design is employed in this research, all possible alternatives were tested experimentally. The performance of the electrode material achieved using each surfactant type is shown in Figure 15. It is evident that cryogenic-treated graphite electrodes outperformed all the systems, except S-20. The parametric optimization was carried out with the individual desirability function, which predicts the response based on the data properties shown in Figure 2.

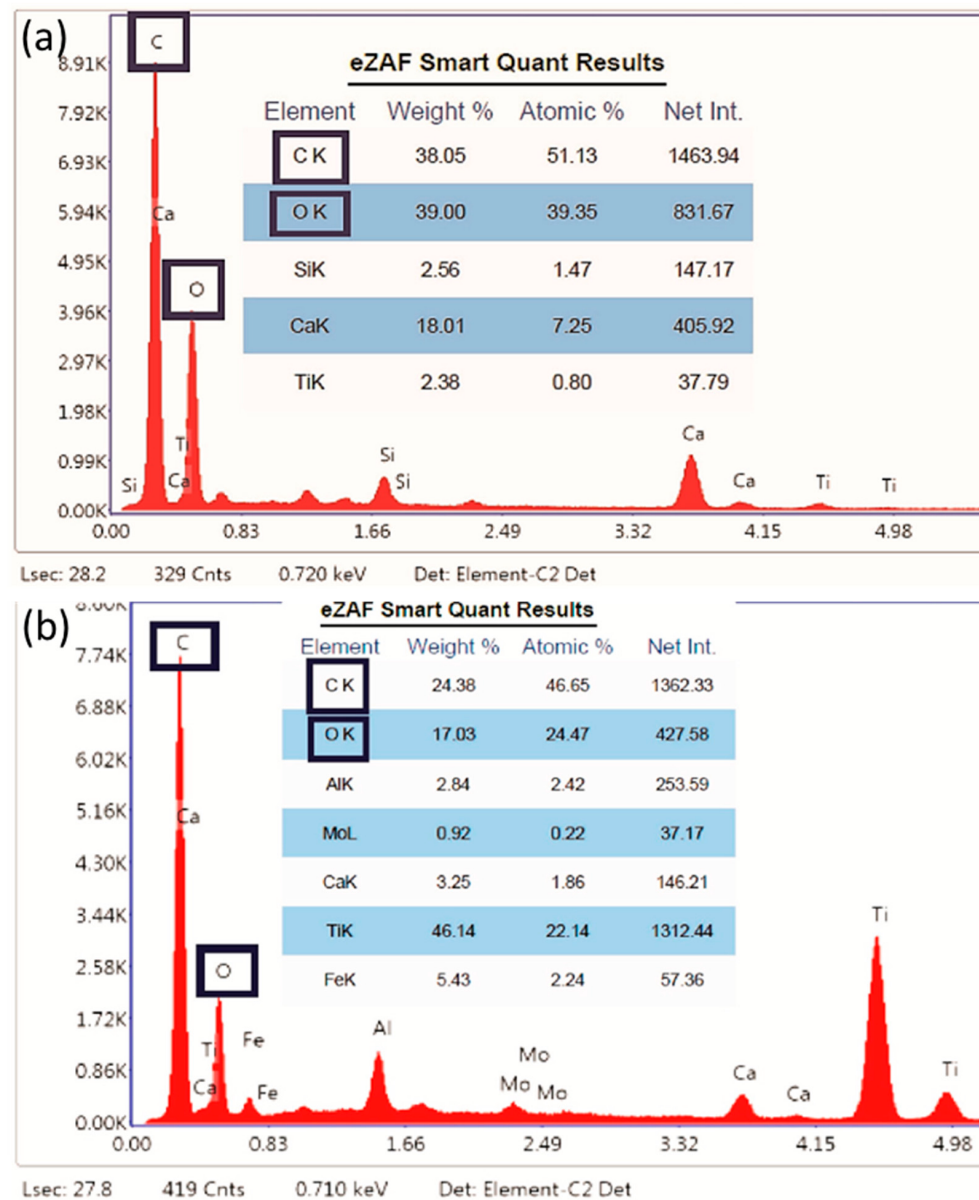


Figure 14. Chemical analysis of (a) kerosene-based dielectric at pulse ON:OFF time of 50:50 μ Sec, and using a graphite electrode and (b) S-60-based kerosene dielectric at 25 g/L, pulse ON:OFF time of 50:100 μ Sec, and using a graphite electrode.

Desirability (d_i) is based on the most desired outcome (achieving large values), which could be optimized through the “larger the better” function. The computation of index d_i using the “larger the better” function with experimental values is carried out through Equation (2).

$$d_i = \begin{cases} 0, & x_i \leq x_{min} \\ \left(\frac{x_i - x_{min}}{x_{max} - x_{min}} \right)^r, & x_{min} \leq x_i \leq x_{max}, r \geq 0 \\ 1, & x_i \geq x_{max} \end{cases} \quad (2)$$

where:

response desirability is indicated by index d_i ;

index x_i shows the expected outcome;

lower value is indicated by index x_{min} ;

higher value is indicated by index x_{max} ;

weight is shown by r .

Index x_i exceeding a certain threshold results in a desirability of 1, and this is 0 otherwise (which is an undesirable event). Similar to the case presented above, the acquisition of the smallest value as a highly desired objective is realized through the “smaller the better” function, as presented in Equation (3).

$$d_i = \begin{cases} 1, & x_i \leq x_{min} \\ \left(\frac{x_i - x_{max}}{x_{min} - x_{max}} \right)^r, & x_{min} \leq x_i \leq x_{max}, r \geq 0 \\ 0, & x_i \geq x_{max} \end{cases} \quad (3)$$

Index x_i overstepping with defined criteria results in 0 desirability, whereas the achievement of the desired outcome reflects a desirability of 1. Since the objective of the current study is to minimize roughness, Equation (3) was used as an optimization function.

On the other hand, the desired outcome with a nominal value (neither minimum nor maximum) requires the “nominal the best” desirability function to be deployed, as mentioned in Equation (4).

$$d_i = \begin{cases} \left(\frac{x_i - x_{min}}{T - x_{min}} \right)^s, & x_{min} \leq x_i \leq T, s \geq 0 \\ \left(\frac{x_i - x_{min}}{T - x_{min}} \right)^t, & T \leq x_i \leq x_{max}, t \geq 0 \\ 0, & \text{otherwise} \end{cases} \quad (4)$$

where:

T is the targeting value;

t and s are weights.

The outcome of the experiment was that x_i index is categorized as the most desirable equal value with a threshold. The desirability becomes 1 in the above-mentioned case and 0 otherwise.

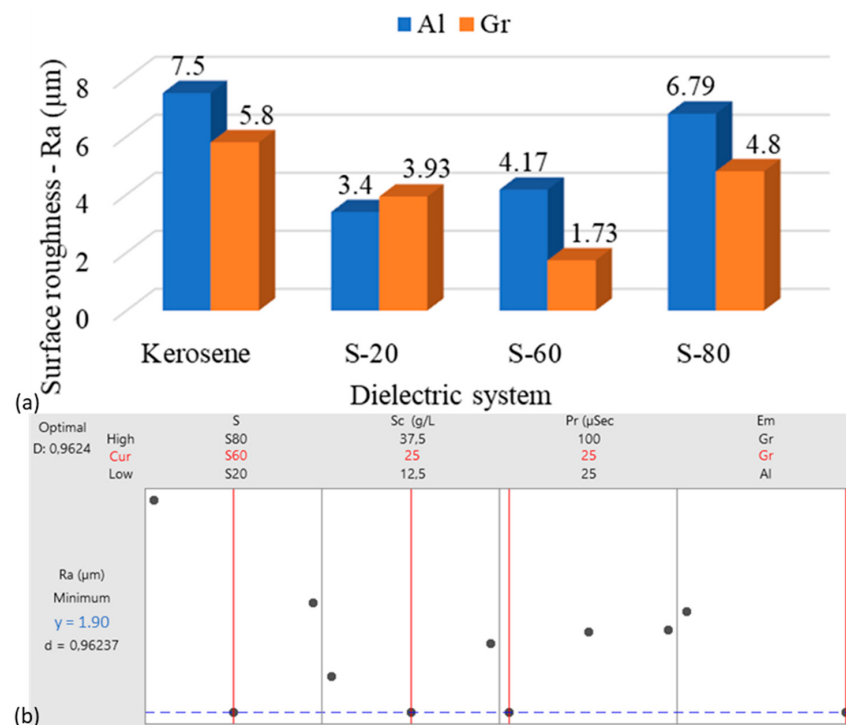


Figure 15. Experimental results of process of the optimization of surface roughness; (a) optimized results for each dielectric type; (b) optimized results from all variables.

It is evident that the unmodified process (with kerosene only) resulted (experimentally) in the relatively highest surface roughness values of 7.5 μm and 5.8 μm for aluminum

and graphite, respectively. However, the modified process (with surfactants) resulted in a lower degree of roughness on the workpiece with both electrodes. The graphite electrode ($1.73\text{ }\mu\text{m}$) performed well when we were using S-60, followed by the aluminum electrode ($3.4\text{ }\mu\text{m}$) using S-20. The resulting roughness for S-80 is increased than those for S-20 and S-60.

The desirability function results in 96.237% achievement of the desired outcome of minimum roughness (as shown in Figure 15). The result is based on the relative evaluation of all the experiments. The S-60 surfactant at an 25 g/L concentration, $50:25\text{ }\mu\text{Sec}$ pulse ON:OFF time, and using the graphite electrode produced the least amount of roughness ($1.73\text{ }\mu\text{m}$) on the workpiece with the slightest surface defects. The validation experiments performed with optimized settings show conformity with the analysis and a low standard deviation, as shown in Table 6.

Table 6. Confirmatory experiments on the S-60.

Sr. No	Process Parameters			Response Indicator	
	S	S _c (g/L)	P _r (μSec)	E _m	R _a (μm)
1	S-60	25	50:25	Gr	1.73
2	S-60	25	50:25	Gr	1.69
3	S-60	25	50:25	Gr	1.77
		Avg. experimental value			1.73
		Standard deviation			0.033

4. Conclusions

The potentiality of various surfactants at different concentrations is comprehensively evaluated with cryogenic-treated electrodes during the EDM of difficult-to-cut aeronautical alloy Ti6Al4V. The findings are examined through process physics and related evidence to improve the process' efficiency in terms of surface integrity. Based on the results, the following conclusions are drawn:

- Based on the analysis of variance, control variables such as the surfactant type (p -value < 0.001), pulse ON:OFF time (p -value 0.003), and electrode material (p -value 0.001) are found to be influential parameters with 16.60% , 4.75% , and 35.64% of the contributions, respectively. The interaction-based effect of surfactant type on its concentration (p -value 0.040), pulse ON:OFF time (p -value < 0.001), and electrode material (p -value 0.005) significantly control the surface quality.
- In the unmodified process, the balanced pulse ratio ($50:50\text{ }\mu\text{Sec}$) resulted in the least amount of roughness for both electrodes ($R_a \sim 7.5\text{ }\mu\text{m}$ for aluminum and $R_a \sim 5.8\text{ }\mu\text{m}$ for graphite).
- The unmodified process-based surface morphology at a pulse ON:OFF time of $50:50\text{ }\mu\text{Sec}$ using the graphite electrode showed a high number of surface defects, such as craters, spherical modules, and debris redeposition such as carbon, due to inefficient flushing.
- The Gr electrode resulted in a better surface texture ($\sim 4.5\text{ }\mu\text{m}$) with smaller, connected craters. On the other hand, deep and large-sized craters were obtained on the surface processed by the Al electrode due to excess heat energy stimulated by the high electrical conductivity of aluminum ($35 \times 10^6\text{ S/m}$).
- Both electrodes presented $\sim 25\%$ improvement of the surfactant-based modified dielectrics compared to that of the kerosene-based dielectric. The S-60-mixed dielectric produced better results than other surfactant types did (S-20 and S-80) owing to its suitable HLB value. The aluminum electrode produced $\sim 6\text{ }\mu\text{m}$ average roughness, and graphite resulted in $\sim 4\text{ }\mu\text{m}$ average roughness using the S-60-mixed dielectric.
- EDS analysis shows significantly less carbide and oxide accumulations on the machined surface in the modified dielectric (S-60) case than in those of the kerosene-based dielectric, attributing to the effective removal of debris.

- The desirability-based process optimization showed that the unmodified process (with kerosene only) produced higher roughness values of 7.5 μm and 5.8 μm for aluminum and graphite, respectively. However, the surfactant-based modified process resulted in a lower degree of roughness on the workpiece. The graphite electrode (1.73 μm) outperformed when we were using S-60, followed by the aluminum electrode (3.4 μm) using S-20.

The study is limited to the evaluation of the surface integrity of Ti6Al4V using modified dielectric(s)-based electric discharge machining. The chemical surface analysis through XRD can potentially be the immediate next step to evaluate element compositions on the surface and their effects on mechanical and tribological properties. In addition, a thorough comparative analysis of tool wear, the material removal rate, and other key machinability indicators are recommended for the titanium alloy while using the various surfactants.

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References

1. Farooq, M.U.; Hussain, A.; Masood, T.; Habib, M.S. Supply Chain Operations Management in Pandemics: A State-of-the-Art Review Inspired by COVID-19. *Sustainability* **2021**, *13*, 2504. [\[CrossRef\]](#)
2. Ahmed, N.; Anwar, S.; Ishfaq, K.; Rafaqat, M.; Saleh, M.; Ahmad, S. The Potentiality of Sinking EDM for Micro-Impressions on Ti-6Al-4V: Keeping the Geometrical Errors (Axial and Radial) and Other Machining Measures (Tool Erosion and Work Roughness) at Minimum. *Sci. Rep.* **2019**, *9*, 17218. [\[CrossRef\]](#) [\[PubMed\]](#)
3. Farooq, M.U.; Ali, M.A.; He, Y.; Khan, A.M.; Pruncu, C.I.; Kashif, M.; Ahmed, N.; Asif, N. Curved Profiles Machining of Ti6Al4V Alloy through WEDM: Investigations on Geometrical Errors. *J. Mater. Res. Technol.* **2020**, *9*, 16186–16201. [\[CrossRef\]](#)
4. Seifi, M.; Salem, A.; Satko, D.; Shaffer, J.; Lewandowski, J.J. Defect Distribution and Microstructure Heterogeneity Effects on Fracture Resistance and Fatigue Behavior of EBM Ti-6Al-4V. *Int. J. Fatigue* **2017**, *94*, 263–287. [\[CrossRef\]](#)
5. Farooq, M.U.; Bhatti, H.A.; Asad, M.; Kumar, M.S.; Zahoor, S.; Khan, A.M. Surface Generation on Titanium Alloy through Powder-Mixed Electric Discharge Machining with the Focus on Bioimplant Applications. *Int. J. Adv. Manuf. Technol.* **2022**, *122*, 1395–1411. [\[CrossRef\]](#)
6. Ehsan, S.; Rehman, M.; Mughal, M.P.; Farooq, M.U.; Ali, M.A. Machinability Investigations through Novel Controlled Flushing Characteristics in Wire Electric Discharge Machining of M42 High-Speed Steel. *Int. J. Adv. Manuf. Technol.* **2022**, *120*, 1315–1332. [\[CrossRef\]](#)
7. Mahajan, A.; Sidhu, S.S.; Devgan, S. MRR and Surface Morphological Analysis of Electrical-Discharge-Machined Co-Cr Alloy. *Emerg. Mater. Res.* **2019**, *9*, 42–46.
8. Umar Farooq, M.; Pervez Mughal, M.; Ahmed, N.; Ahmad Mufti, N.; Al-Ahmari, A.M.; He, Y. On the Investigation of Surface Integrity of Ti6Al4V ELI Using Si-Mixed Electric Discharge Machining. *Materials* **2020**, *13*, 1549. [\[CrossRef\]](#)
9. Farooq, M.U.; Anwar, S.; Kumar, M.S.; AlFaify, A.; Ali, M.A.; Kumar, R.; Haber, R. A Novel Flushing Mechanism to Minimize Roughness and Dimensional Errors during Wire Electric Discharge Machining of Complex Profiles on Inconel 718. *Materials* **2022**, *15*, 7330. [\[CrossRef\]](#)
10. Mahajan, A.; Singh, G.; Devgan, S.; Sidhu, S.S. EDM Performance Characteristics and Electrochemical Corrosion Analysis of Co-Cr Alloy and Duplex Stainless Steel: A Comparative Study. *Proc. Inst. Mech. Eng. Part E J. Process Mech. Eng.* **2021**, *235*, 812–823. [\[CrossRef\]](#)
11. Ishfaq, K.; Waseem, M.U.; Sana, M. Investigating Cryogenically Treated Electrodes' Performance under Modified Dielectric (s) for EDM of Inconel (617). *Mater. Manuf. Process.* **2022**, *37*, 1902–1911. [\[CrossRef\]](#)

12. Philip, J.T.; Mathew, J.; Kuriachen, B. Transition from EDM to PMEDM—Impact of Suspended Particulates in the Dielectric on Ti6Al4V and Other Distinct Material Surfaces: A Review. *J. Manuf. Process.* **2021**, *64*, 1105–1142. [\[CrossRef\]](#)
13. Mughal, M.P.; Farooq, M.U.; Mumtaz, J.; Mia, M.; Shareef, M.; Javed, M.; Jamil, M.; Pruncu, C.I. Surface Modification for Osseointegration of Ti6Al4V ELI Using Powder Mixed Sinking EDM. *J. Mech. Behav. Biomed. Mater.* **2021**, *113*, 104145. [\[CrossRef\]](#) [\[PubMed\]](#)
14. Ilani, M.A.; Khoshnevisan, M. Study of Surfactant Effects on Intermolecular Forces (IMF) in Powder-Mixed Electrical Discharge Machining (EDM) of Ti-6Al-4V. *Int. J. Adv. Manuf. Technol.* **2021**, *116*, 1763–1782. [\[CrossRef\]](#)
15. Schramm, L.L.; Stasiuk, E.N.; Marangoni, D.G. Surfactants and Their Applications. *Annu. Rep. Prog. Chem. Sect. C Phys. Chem.* **2003**, *99*, 3–48. [\[CrossRef\]](#)
16. Bart, J.C.J.; Gucciardi, E.; Cavallaro, S. Formulating Lubricating Oils. In *Biolubricants*; Bart, J.C.J., Gucciardi, E., Cavallaro, S., Eds.; Woodhead Publishing Series in Energy; Woodhead Publishing: Sawston, UK, 2013; pp. 351–395. ISBN 978-0-85709-263-2.
17. Reddy, V.V.; Kumar, A.; Valli, P.M.; Reddy, C.S. Influence of Surfactant and Graphite Powder Concentration on Electrical Discharge Machining of PH17-4 Stainless Steel. *J. Braz. Soc. Mech. Sci. Eng.* **2015**, *37*, 641–655. [\[CrossRef\]](#)
18. Kolli, M.; Kumar, A. Effect of Dielectric Fluid with Surfactant and Graphite Powder on Electrical Discharge Machining of Titanium Alloy Using Taguchi Method. *Eng. Sci. Technol. Int. J.* **2015**, *18*, 524–535. [\[CrossRef\]](#)
19. Sugunakar, A.; Kumar, A.; Reddy, V.V. Effect of Graphite Powder and Surfactant Mixed Dielectric in Electrical Discharge Machining of Nickel Alloy. *Zeichen. J.* **2021**, *7*, 328–336.
20. König, W.; Jörres, L. Aqueous Solutions of Organic Compounds as Dielectrics for EDM Sinking. *CIRP Ann.* **1987**, *36*, 105–109. [\[CrossRef\]](#)
21. Zhang, Y.; Liu, Y.; Ji, R.; Cai, B.; Shen, Y. Sinking EDM in Water-in-Oil Emulsion. *Int. J. Adv. Manuf. Technol.* **2013**, *65*, 705–716. [\[CrossRef\]](#)
22. Wu, K.L.; Yan, B.H.; Huang, F.Y.; Chen, S.C. Improvement of Surface Finish on SKD Steel Using Electro-Discharge Machining with Aluminum and Surfactant Added Dielectric. *Int. J. Mach. Tools Manuf.* **2005**, *45*, 1195–1201. [\[CrossRef\]](#)
23. Dukhin, A.S.; Goetz, P.J. *Ionic Properties of So-Called “Non-Ionic” Surfactants in Non-Polar Liquids*; Dispersion Technology Inc.: Bedford Hills, NY, USA, 2005; Volume 15, pp. 151–157.
24. Wu, K.L.; Yan, B.H.; Lee, J.-W.; Ding, C.G. Study on the Characteristics of Electrical Discharge Machining Using Dielectric with Surfactant. *J. Mater. Process. Technol.* **2009**, *209*, 3783–3789. [\[CrossRef\]](#)
25. Reddy, V.V.; Valli, P.M.; Kumar, A.; Reddy, C.S. Multi-Objective Optimization of Electrical Discharge Machining of PH17-4 Stainless Steel with Surfactant-Mixed and Graphite Powder-Mixed Dielectric Using Taguchi-Data Envelopment Analysis-Based Ranking Method. *Proc. Inst. Mech. Eng. Part B J. Eng. Manuf.* **2015**, *229*, 487–494. [\[CrossRef\]](#)
26. Kumar, S.; Khedkar, N.K.; Jagtap, B.; Singh, T.P. The Effects of Cryogenic Treatment on Cutting Tools. *IOP Conf. Ser. Mater. Sci. Eng.* **2017**, *225*, 012104. [\[CrossRef\]](#)
27. Abdulkareem, S.; Khan, A.A.; Konneh, M. Reducing Electrode Wear Ratio Using Cryogenic Cooling during Electrical Discharge Machining. *Int. J. Adv. Manuf. Technol.* **2009**, *45*, 1146. [\[CrossRef\]](#)
28. Singh, R.; Singh, B. Comparison of Cryo-Treatment Effect on Machining Characteristics of Titanium in Electric Discharge Machining. *Int. J. Automot. Mech. Eng.* **2011**, *3*, 239–248. [\[CrossRef\]](#)
29. Sundaram, M.M.; Yildiz, Y.; Rajurkar, K.P. Experimental Study of the Effect of Cryogenic Treatment on the Performance of Electro Discharge Machining. In Proceedings of the ASME 2009 International Manufacturing Science and Engineering Conference, West Lafayette, IN, USA, 4–7 October 2009; American Society of Mechanical Engineers Digital Collection. pp. 215–222.
30. Grewal, G.S.; Dhiman, D.P. Effect of Deep Cryogenic Treatment on Copper Electrode for Non-Traditional Electric Discharge Machining (EDM). *Mech. Sci.* **2019**, *10*, 413–427. [\[CrossRef\]](#)
31. Srivastava, V.; Pandey, P.M. Performance Evaluation of Electrical Discharge Machining (EDM) Process Using Cryogenically Cooled Electrode. *Mater. Manuf. Process.* **2012**, *27*, 683–688. [\[CrossRef\]](#)
32. Croda Home|Span Series Surfactants Physical Properties. Available online: <https://www.croda.com/en-gb> (accessed on 22 February 2023).
33. Raza, S.M.; Khan, A.M.; Farooq, M.U.; Iqbal, A.; Pimenov, D.Y.; Giasin, K.; Leksycki, K. Modelling and Analysis of Surface Evolution on Turning of Hard-to-Cut CLARM 30NiCrMoV14 Steel Alloy. *Metals* **2021**, *11*, 1751. [\[CrossRef\]](#)
34. Kanlayasiri, K.; Boonmung, S. Effects of Wire-EDM Machining Variables on Surface Roughness of Newly Developed DC 53 Die Steel: Design of Experiments and Regression Model. *J. Mater. Process. Technol.* **2007**, *192*, 459–464. [\[CrossRef\]](#)
35. Ishfaq, K.; Farooq, M.U.; Pruncu, C.I. Reducing the Geometrical Machining Errors Incurred during Die Repair and Maintenance through Electric Discharge Machining (EDM). *Int. J. Adv. Manuf. Technol.* **2021**, *117*, 3153–3168. [\[CrossRef\]](#)
36. Dzulkifli, N.F.; Mamat, A.; Choudhury, I.A. The Potential of Water-In-Oil Emulsion of Canola Oil as Dielectric Fluid for EDM Process. *Arch. Akad. Baru Artic.* **2021**, *72*, 129–141. [\[CrossRef\]](#)
37. Chen, S.L.; Yan, B.H.; Huang, F.Y. Influence of Kerosene and Distilled Water as Dielectrics on the Electric Discharge Machining Characteristics of Ti-6Al-4V. *J. Mater. Process. Technol.* **1999**, *87*, 107–111. [\[CrossRef\]](#)

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