

**ANKARA YILDIRIM BEYAZIT UNIVERSITY GRADUATE SCHOOL
OF NATURAL AND APPLIED SCIENCE**



**ELECTROPOLISHING OF TITANIUM GRADE-2 IN
HYDROFLUORIC ACID – ETHYLENE GLYCOL ELECTROLYTE**

M. Sc. Thesis by

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ANKARA

**ELECTROPOLISHING OF TITANIUM GRADE-2 IN
HYDROFLUORIC ACID – ETHYLENE GLYCOL
ELECTROLYTE**

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M. Sc. THESIS EXAMINATION RESULT FORM

We have read the thesis entitled “**ELECTROPOLISHING OF TITANIUM GRADE-2 IN HYDROFLUORIC ACID – ETHYLENE GLYCOL MIXED ELECTROLYTE**” completed by **Büşra KOZAN** under supervision of **Doç Dr. Metehan ERDOĞAN** and we certify that in our opinion it is fully adequate, in scope and in quality, as a thesis for the degree of Master of Science.

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ELECTROPOLISHING OF TITANIUM GRADE-2 IN HYDROFLUORIC ACID – ETHYLENE GLYCOL MIXED ELECTROLYTE

ABSTRACT

Electropolishing is a finishing process that selectively removes material from the surface of both simple or complex metal parts to produce a bright and smooth surface. In this study, the electropolishing process was applied to Titanium grade-2 material, known for its purity as alpha titanium and widespread utility, using an electrolyte composed of a mixture of hydrofluoric acid and ethylene glycol. The primary objective was to investigate the evolution of surface morphology through various analytical techniques such as surface roughness measurements, optical microscopy and scanning electron microscopy. The study delved into crucial parameters of the electropolishing process: current density, electrolyte temperature, electropolishing time, and initial surface roughness. Titanium grade-2 material demonstrated favorable electropolishing character in an electrolyte solution comprising 10.5 vol-% hydrofluoric acid and 89.5 vol-% ethylene glycol. Moreover, the study revealed that mechanical polishing, as a preparatory step, significantly influenced surface brightness, electropolishing duration and the lifetime of the electropolishing solution. The optimum surface finish for Titanium grade-2 was obtained at 20°C, with a current density of 22 A/dm², over a duration of 12 minutes, within the parameter ranges covered in this study.

Keywords: Titanium grade-2, electropolishing, surface finishing, mirror-like surface, hydrofluoric electrolyte.

TİTANYUM SINIF-2'NİN HİDROFLORİK ASİT – ETİLEN GLİKOL KARIŞIMLI ELEKTROLİT İÇİNDE ELEKTROPOLİSAJ

ÖZ

Elektro-parlatma, parlak ve pürüzsüz bir yüzey üretmek için hem basit hem de karmaşık metal parçaların yüzeyindeki malzemeyi seçici olarak kaldıran bir son işlemdir. Bu çalışmada, elektro-parlatma işlemi, hidroflorik asit ve etilen glikol karışımından oluşan bir elektrolit kullanılarak, alfa titanyum olarak saflığı ve yaygın kullanımı ile bilinen Titanyum sınıf-2 malzemesine uygulanmıştır. Birincil amaç, yüzey pürüzlülüğü ölçümleri, optik mikroskopi ve taramalı elektron mikroskobu gibi çeşitli analitik teknikler aracılığıyla yüzey morfolojisinin gelişimini araştırmaktır. Çalışma, elektro-parlatma işleminin önemli parametrelerini incelemiştir: akım yoğunluğu, elektrolit sıcaklığı, elektro-parlatma süresi ve başlangıç yüzey pürüzlülüğü. Özellikle, Titanyum sınıf-2 malzemesi, hacimce %10,5 hidroflorik asit ve hacimce %89,5 etilen glikol içeren bir elektrolit çözeltisinde olumlu elektropolisaj karakteri göstermiştir. Ayrıca çalışma, bir hazırlık adımı olarak mekanik parlatmanın yüzey parlaklığını, elektro-parlatma süresini ve elektro-parlatma çözeltisinin ömrünü önemli ölçüde etkilediğini ortaya koymuştur. Titanyum sınıf-2 için optimum yüzey kalitesi 20°C'de, 22 A/dm² akım yoğunluğunda, 12 dakikalık bir süre boyunca, bu çalışmada kapsanan parametre aralıkları dahilinde elde edilmiştir.

Anahtar Kelimeler: Titanyum sınıf-2, elektro-parlatma, yüzey bitirme, ayna benzeri yüzey, hidroflorik elektrolit

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NOMENCLATURE

Roman Letter Symbols

C	Coulomb
D	Diffusion Constant
D_0	Material Diffusion Constant
F	Faraday Constant
m	Mass
R	Gas Constant
V_0	Volume

Greek Letter Symbols

μm	Micrometer
ρ	Density

Subscripts

A	Amper
cm^3	centimeter cube
g	gram
GPa	Gigapascal
h	hour
I	Current
mm	millimeter
mm^3	millimeter cube
MPa	Megapascal
q	Electrical Charge
Qa	Activation Energy
s	second
T	Temperature
V	Volt
W	Watt
°	Degree

Acronyms

CP	Commercially pure
Cu	Copper

$\text{C}_3\text{H}_6\text{O}$	Acetone
$\text{C}_3\text{H}_8\text{O}$	Isopropyl alcohol
$\text{C}_2\text{H}_6\text{O}_2$	Mono-ethylene glycol
DC	Direct current
EP	Electropolishing
HCl	Hydrochloric acid
HF	Hydrofluoric acid
H_3PO_4	Phosphoric acid
H_2SO_4	Sulphuric acid
Ti	Titanium
TG-2	Titanium Grade-2
vol%	Volume percentage

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CHAPTER 1

INTRODUCTION

1.1 Electropolishing Process

Electropolishing (EP) is an electrochemical process. This process strips the surface from a metallic piece to polish and deburr the metal sample. EP is a special and sensitive polishing process. This process is the reverse of electroplating and it is also known as electrolytic polishing or electrochemical polishing [1]. Conventional mechanical polishing processes can cause deformed layer and residual stresses on the workpiece surface. During the processing of the workpiece, residues can cause contaminations and this may adversely affect the durability of the pieces. Electropolishing process clean the surface from contaminations and gives the stress free surface. Figure 1.1 shows the difference between surfaces which are raw, abrasive machined and electropolished.



Figure 1.1 Raw, abrasive machined, and electropolished metal surfaces

EP process has an important role for precision engineering and high-tech industry. This technique forms a thin inactive film on the material surface and this film increases the corrosion resistance. Electropolishing process can change the material surface

characteristics (from hydrophilic to hydrophobic). Modification of the material surface should be done according to the intended function and the place to be used. The main goal in this process is the brightening and flattening of surfaces. As a result of this process, the surface roughness decreases and a smoother surface is obtained [2] - [5].

1.2 Electropolishing Theory

In the system, there is an active surface between the electrolyte and the metal surface, which allows the metal material to dissolve. With the continuous dissolution of the metal workpiece in the form of metal ions, a viscous layer is formed and the dissolved mass is deposited in the bath, thus forming an ionic concentration on the rough workpiece surface as seen as in Figure 1.2. The current density passes through rough surfaces much higher than smooth surfaces and therefore dissolution of rough surfaces occurs much faster. Over time, the rough surface dissolves and a smooth surface is obtained [6].

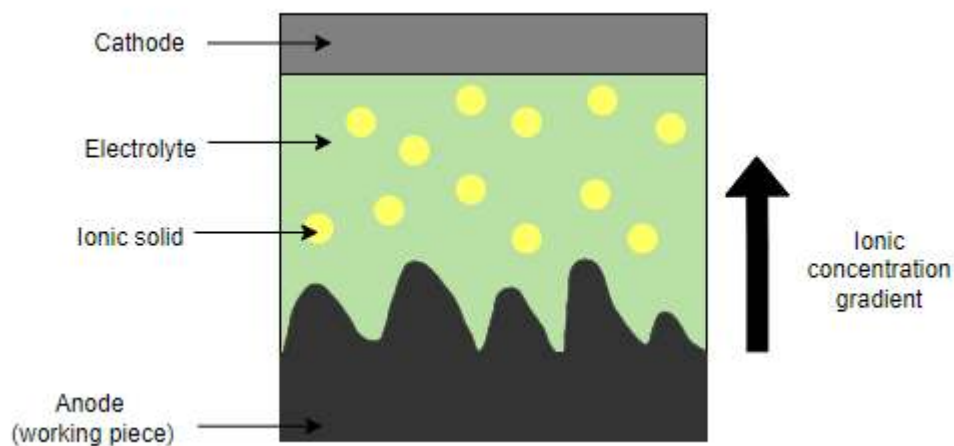


Figure 1.2 Schematic representation of ionic concentration gradient

Figure 1.3 shows the adsorbate-acceptor system model. It schematically shows the anodic dissolution of a metallic workpiece M in an electrolyte containing a small quantity A which is the acceptor species. This model is important for understanding

anodic dissolution in the electropolishing process. The electrolyte in question here may consist of a type of acid or mixtures of different acids and may be a type related to water, which is necessary for the dissolution of water or cations of A indicated as acceptor. In the electropolishing process the metal is dissolved and oxidized. As this happens, cations are released and adsorb to the surface. The surface is blocked from dissolution by the ions and high potential may be required for the reaction to continue. The acceptor ions remove the cations from the surface and the metal ions dissolve. When the concentration of the acceptor species approaches zero at the electrode-solution interface, the limiting current is reached [7].

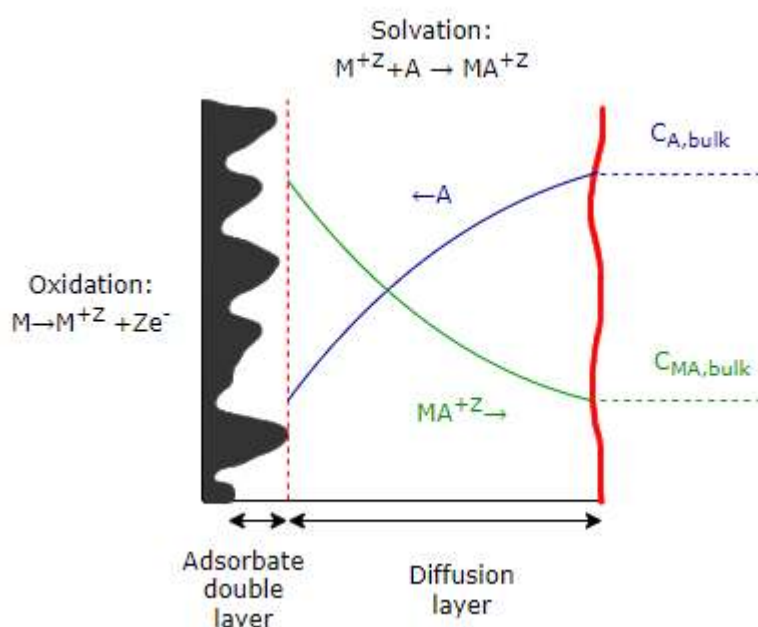


Figure 1.3 Adsorbate-acceptor model for electropolishing process

Faraday law, Jacquet theory, Edwards theory, Elmore theory, Hoar and Farthing theory explain electropolishing process. A single theory is insufficient to fully explain this process. Many different parameters play a role in the electropolishing and these theories need to be examined in order to fully understand it.

1.2.1 Faraday's Law

The electropolishing process theory can be explained by Faraday's law from 19th century. Faraday expressed the amount of electrochemically modified material in terms of electrical energy. This is called Faraday's laws of electrolysis. As the current passes through the electrolyte, matter accumulates on the electrode and the amount of matter accumulated is directly proportional to the amount of electricity passing through [8]. If the current I (A) is constant, the electrical charge q (C) is equal to the product of current and time t (s) as given in the equation 1.1 [9]. The mass of the dissolved metal W (g) can be calculated theoretically by this law. The amount of electricity required to dissolve 1 gram of an element with atomic value n and atomic weight M is nF/M coulombs. The total amount of metal dissolved over the elapsed time t (s) and applied current I (A) is calculated according to the formula 1.2 [10], [11].

$$q = I \times t \quad (1.1)$$

$$W = \frac{Mit}{nF} \quad (1.2)$$

F is the Faraday constant.

We can calculate the theoretical removal volume V_0 (mm³) with the density of the atom ρ [10];

$$V_0 = \frac{Mit}{nF\rho} \quad (1.3)$$

Also, the amount of the removed metal can be expressed in height h (mm) with the surface area A (mm²) of a metal part [6];

$$h = \frac{Mit}{nFA} \quad (1.4)$$

1.2.2 Jacquet's Theory

In Jacquet's viscous film theory, the electrolyte, which is the solvent, adheres to the surface of the workpiece and prevents excess dissolution by limiting the current. Jacquet's theory says that the viscous film does not spread completely evenly on the surface of the workpiece. The hollows and peaks of the rough surface are covered with a viscous layer of different thickness adjacent to the anode as illustrated in Figure 1.4 [12]. He stated that the viscous layer is thinner in the hills, which means that these areas dissolve faster. On the contrary, he said that the hollows are covered with a thicker viscous layer and the dissolution in these areas is quite slow. With the difference in the thickness of the viscous layer, the rough surface is smoothed [13].

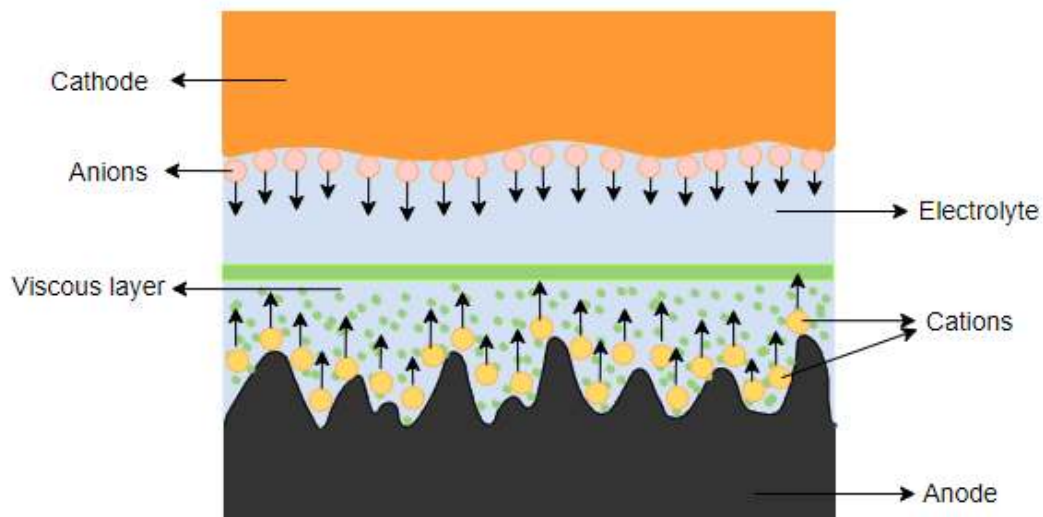


Figure 1.4 Schematic drawing of viscous film theory

1.2.3 Elmore Theory

Elmore analyzed Jacquet's theory in his study and said that this theory is quite simple. In addition to this theory, he proposed his own diffusion theory. In his work in 1939-1940, he applied the electropolishing process on copper using an electrolyte composed of orthophosphoric acid and examined the current-voltage relationship also he made some assumptions in his diffusion theory. As the current passes through the system, the metal dissolves and leaves the anode surface by diffusion. The concentration gradient at the anode should be proportional to the current density over the whole time. In the case where the area of the metal part is A and $x=0$, the current is as in formula 1.5 [14], [15].

$$i = -AFD\left(\frac{\partial c}{\partial x}\right)_{x=0} \quad (1.5)$$

Here F is the Faraday constant, c is the metal concentration in the equivalents, D is the dissolved metal diffusion coefficient.

Current flows through the cell for some time and the concentration on the metal anode surface reaches a maximum value. If no new reaction appears, the current is limited by the concentration gradient. In this case, a limiting current is generated and the concentration gradient is higher at the protrusions on the metal surface, so that it dissolves faster than in the cavities. Elmore says that the polishing effect is due to differences in the concentration gradients of the solute metal ions [15].

1.2.4 Edwards Theory

Edward proposed the diffusion theory of receptors to replace Elmore's theory. According to the acceptor theory, the dissolution rate of the anode metal depends on the rate at which acceptor ions in the electrolyte reach the metal surface. These ions reach the surface by diffusion. The polishing effect is realized by the diffusion layer having a steeper concentration gradient at the peaks of the surface. In other words, the polishing process is realized from the changes in the concentration gradient along the

diffusion layer on the metal anode surface. Instead of the diffusion of ions from the anode metal given in the basic electropolishing theory, this theory says that solvent ions (acceptors) in the solvent move towards the anode [16], [17].

1.2.5 Hoar and Farthing Theory

In the study by Hoar and Mowat [18] using urea-ammonium chloride solution on nickel anode, it was observed that nickel anode showed the same electrolytic behavior as other studies. They addressed the issue of anodic passivation, which was not mentioned in previous studies and also, proposed the passivation film theory with this experimental study. The theory states that a thin film is continuously formed between the electrolyte and the metal anode but does not thicken; the electrolyte solution dissolves it immediately. The foundation of solid film theory was laid with this concept. Hoar and Farthing [19] proved this theory with their electropolishing study on α -brass and copper using orthophosphoric acid. When mercury was dropped on the anode, the surface became wet in a very short time and then spread. Taking advantage of mercury's ability to diffuse on smooth, clean solid metals, the authors observed the presence of a continuously forming and dissolving solid film on copper and α -brass anodes.

1.3 Electropolishing Mechanism

Electropolishing is the type of electrolysis that includes a direct electric current passing along an electrolyte in an electrolytic cell. The metal sample to be polished used as anode and it is positive terminal and the negative terminal is the cathode of a DC (direct current) power supply. The sample is dipped into a formulated and temperature adjusted electrolytic solution. Electrical current produced by the power supply passes from the electrolyte, the oxidation reaction is occurred on the surface of metal sample and the protrusions dissolve quickly, at the end a smooth metal surface is obtained (Figure 1.3) [1]. As a result of this reaction, impurities and irregularities on the metal surface are removed.

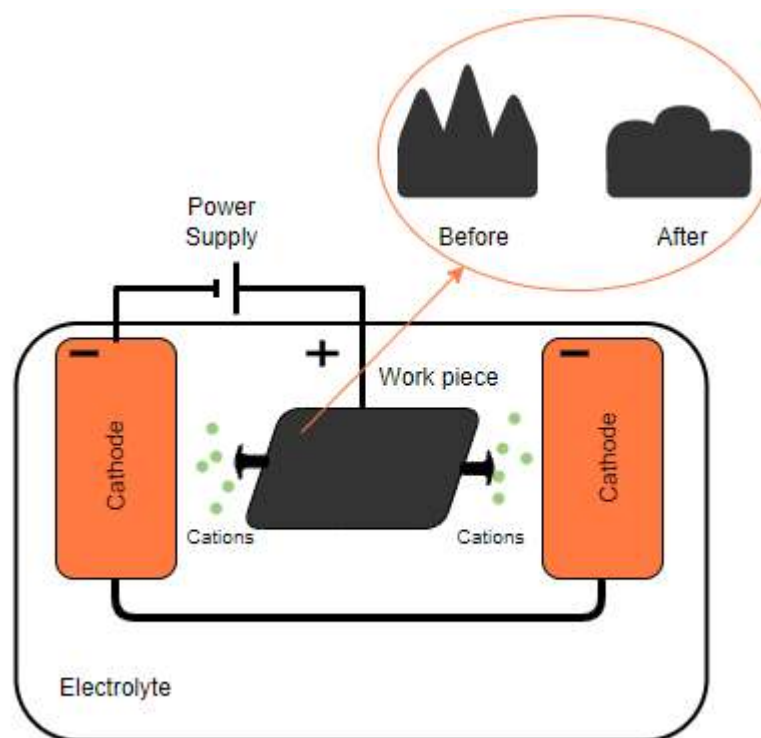


Figure 1.5 A typical setup of electropolishing mechanism and surface illustration before and after process

On the cathode side, a reduction reaction is observed and hydrogen is produced, on the anode side, the oxygen gas is produced along the metallic dissolution. Bath composition, temperature, current density and the metal piece affects the amount of metal removing. In this process generally, time and current are the controlled variables. Solution which is generally acid solutions such as sulphuric, chloric and phosphoric acids or their mixtures with water, alcohol or acetic acid are used as electrolyte. Chemicals such as glycerol, ethylene glycol, butyl glycol are used to increase electrolyte viscosity. The metal part dissolves in the electrolyte and metal ions move from the metal surface into the electrolyte. This ion movement continues continuously throughout the process. As seen as in the Figure 1.3, an ionic concentration gradient is formed on the rough part surface and with high current density passing through the roughness, these areas dissolve much faster, resulting in a smooth surface. Chosen

electrolyte and current density determines the amount of material removal in electropolishing process [1], [3], [20], [21].

1.3.1 Current Density-Voltage Curve of Electropolishing

Jacquet, used the current density-voltage curve to analyze the dissolution of anodic material in the electropolishing process. Figure 1.4 shows the current density-voltage plot for the electropolishing process. During the anodic dissolution process, etching, passivation, polishing and gas formation reactions occur. In the etching zone, the metal surface directly dissolves, creating pits in this area. In the passivation region, the current density decreases with increasing voltage due to the formation of a passive oxide layer on the metal surface. In the region of the limiting current plateau, the current density remains almost constant with increasing voltage, and the electropolishing process occurs at a high rate in this region. However, a current higher than the applied current is needed in this region. In order to obtain the best surface in the electropolishing process, the plateau region is not the best range, the applied current density is a more important parameter. In the gas formation region, the passive oxide layer breaks down with increasing voltage and anodic dissolution occurs. Pitting is observed due to oxygen bubbles remaining on the surface of the workpiece. Therefore, the gas formation region is also called the pitting region [22], [23].

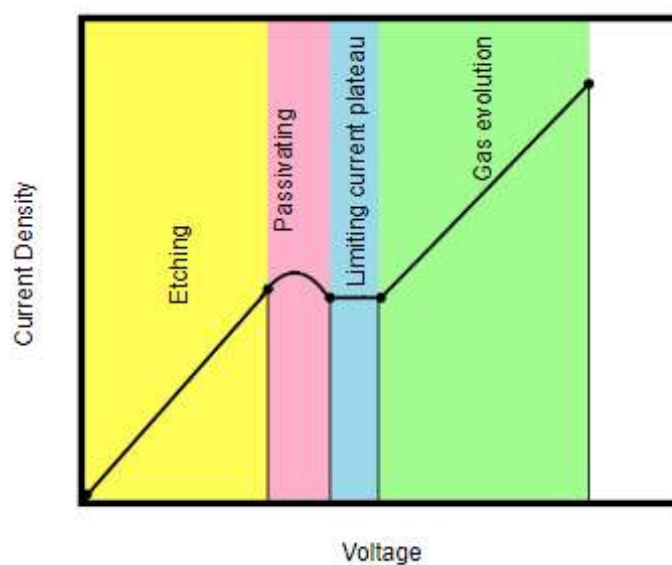


Figure 1.6 The current density-voltage curve of EP process

1.4 Properties of Electropolished Material

The surface roughness of a metal part can affect its performance in the application in which it will be used. Surface roughness, even at a low level, has been shown to affect the tensile force with another object and interfere with surface adhesion. In applications where these parts will be used, insecurities may arise regarding the connection in the assembly part. Various metal parts are used as needed in areas such as aerospace, automotive, biomedical. For the parts to be used in these fields, material properties such as durability, biocompatibility and corrosion resistance are very important [24]. It has been shown in many different studies that these properties are improved by surface finishing techniques especially by electropolishing [25], [26].

1.4.1 Mechanical Properties

Surface roughness plays an important role on the fatigue properties of a material. Maiya and Bush [27] proved in their study that fatigue life is directly related to surface roughness. The fatigue life of 304 stainless steel decreases with increasing surface roughness and it is thought that roughness may have an effect on crack formation and propagation rate. In the study by Cheung et al. [28], on fatigue behavior, a different result was obtained. In this study, electropolishing with hypochlorite solution was applied on nickel-titanium material to investigate the low-cycle fatigue behavior. Fracture cycle number, crack initiation locations, crack elongation and surface stress were investigated. The electropolished parts showed at most one crack source, whereas the non-electropolished parts showed more of these sources. The electropolishing process reduced the roughness of the parts but did not affect the low cycle fatigue failure. In another study [29], while the durability of the laser doped Ti-6Al-4V part was 210 MPa, the durability of the same polished part increased to 510 MPa. This has been associated with smoother surface properties and removal of surface defects on the part as cracks.

1.4.2 Corrosion Resistance

The deterioration or damage of the surface of a material by being exposed to chemical or electrochemical reactions under the influence of environmental factors is called

corrosion. Corrosion resistance refers to how resistant the material is to rusting, abrasion or chemical reactions over time. Corrosion resistance can change according to the chemical composition, microstructure and surface properties of material. The surfaces of materials that have not been electropolished are often damaged and contain impurities. These cause corrosion to begin and rust and once corrosion begins, it continues to spread. The electropolishing process provides corrosion resistance to the part by removing impurities and damaged layers on the material surface. Faust made a study on corrosion resistance of different stainless steel test samples which were mechanically polished and electropolished. The mechanically polished sample surface was covered with corrosion, but the electropolished sample surface showed a mirror-like finish [22], [30]. Viera ZATKALÍKOVÁ and others study on the AISI 316L austenitic stainless-steel shows that, corrosion pits on the untreated surface are spread upon the surface in irregular shapes. It is mostly oval or round shape on electropolished surfaces and these pits are found in mechanical grinding lines [31]. Melchers and Jeffrey [32] examined the effect of surface finishing techniques on corrosion resistance in their study on steel. It was observed that the corrosion rate of acid cleaned and electropolished surface finished parts was less than those without surface finishing after a period of 2 months. It was stated that surface roughness decreased as a result of the electropolishing process, and rough surfaces are more favorable to corrosion formation.

1.5 Important Parameters in Electropolishing Process

The surface smoothening process in the electropolishing process consists of anodic smoothening. Anodic smoothening consists of two stages, one is macro-smoothening (anodic levelling), the other is micro-smoothening (brightening). Smoothness of the surface is the result of macro-smoothening and the brightness of the surface is the result of micro-smoothening. In an electropolishing process, many parameters play an important role in smoothing and polishing the surface. Factors such as temperature, current density, bath composition, polishing time are the basic factors for a successful surface polishing process and are crucial for an accomplished surface treatment [33].

1.5.1 Electrolyte Composition

An electrolyte is act as a bearer of reactions, heat and current. Different chemical mixtures such as organic or inorganic can be used for electrolyte compositions. Different acid types and concentrations are used for different metals and alloys. The bath composition directly affects the final surface properties of target material therefore, electrolyte composition should have some characteristic properties; (i) it should be viscous, (ii) should be good solvent, (iii) shouldn't react with the sample when the current off, (iv) should include large ions or organic molecules, (v) should be safe and stable, (vi) should work at room temperature conditions. The electrolyte composition has been the subject of a lot electropolishing studies. In the literature, a big part of electrolyte formulas is for stainless-steel polishing. In the electropolishing study conducted on niobium samples, baths with different HF and H₂SO₄ concentrations were used and as a result of the study, the process speed and the service life of the solution increased with the increase in the amount of HF [34], [35]. Rokosz et al. applied an electrochemical polishing process on AISI 316L stainless steel. H₃PO₄ and H₂SO₄ were used in different concentrations and it was observed that their volumetric ratios had an effect on the chemical composition of the passive film formed after the electrochemical polishing process. The best corrosion resistance was obtained as a result of experiments using 40% H₃PO₄ and 60% H₂SO₄ by volume. The volume ratio of acid plays an essential role in EP [36].

1.5.2 Temperature

One of the most significant factors for electropolishing process is temperature. The quality of the finishing surface depends on electrolyte temperature. The biggest effect on the diffusion constant and speed is temperature. Lower temperature means slower diffusion of metal ions. The solubility of metal ions is affected from the solution temperature. The low temperature also means slower metal ion solubility. However, it is difficult to control the reaction at high temperatures and accumulation of impurities at the anode can be observed. The reaction rate is greatly affected by temperature and a homogeneous surface may not be obtained [37], [38]. The relationship between diffusion constant and temperature is as in the formula below;

$$D = D_0 \exp\left(-\frac{Q_a}{RT}\right) \quad (1.4)$$

D_0 is the material constant (m^2/s), Q_a is the activation energy for the diffusion (J/mol), R is the gas constant (8.31 J/mol.K) and K is the absolute temperature (K) [39].

1.5.2 Electropolishing Time and Initial Surface Roughness

Electropolish time is a key parameter in electropolishing processes. The studies have shown that the longer the time, more anodic dissolution and the part surface becomes smoother. It has been observed that the irregularities of the metal surface decrease, homogeneity increases and becomes more accurate as the working time increases and this effect was reported by Haidopoulos et al [40]. In another study using sulfuric acid and phosphoric acid, it was observed that the roughness decreased abruptly at the beginning and continued to decrease with increasing time [41]. Another important parameter in electropolishing process is initial roughness, because the ability of this process to reduce roughness and polish is limited. In the electropolishing study on the shape memory alloy, the part with an initial roughness of $2 \text{ }\mu\text{m}$ was reduced to a minimum of $0.98 \text{ }\mu\text{m}$, but the roughness of the part with an initial roughness of $1 \text{ }\mu\text{m}$ was reduced to below $0.5 \text{ }\mu\text{m}$. In the study, no significant decrease in surface roughness was observed after 3 minutes. For a successful electropolishing process, the initial surface roughness of the part must be taken into account [10].

1.5.3 Other Parameters

The distance between anode and cathode is also a factor affecting the electropolishing process. The greater the distance between them, the more voltage the system needs to pass to maintain the current density, and the higher the voltage, the more likely it is to cause unwanted pitting and a poor surface appearance on the part surface. Conversely, if the distance between the anode and cathode is too small, the voltage value drops considerably and the desired good surface finish is not achieved. For a successful surface finish, the best distance between anode and cathode must be determined and maintained [42]. The rotational speed of the electrolyte or workpiece is another factor

affecting the electropolishing process. As the rotational speed increases, the thickness of the diffusion layer on the workpiece decreases, ion diffusion takes a shorter path and thus dissolution occurs faster [43]. The age of the electrolyte used in the electropolishing process is a very important parameter. The reuse of the bath is very important in the industrial dimension in terms of both reducing the harmful effect on the environment and minimizing the cost. However, if the bath is used repeatedly, the limiting current density decreases over time with cation consumption. In this case again, the chemical chosen is very important and a reasonable value should be set for the reuse of the bath [1].



1.6 Possible Problems in Electropolishing Process

Table 1.1 Solutions suggestions to possible problems and causes in electropolishing process [44]

Problem	Probable cause	Solution suggestion
Center of the specimen deeply etched	No polishing film at the center of the specimen	- Increase voltage - Decrease agitation - Use more viscous electrolyte
Pitting or etching at edges of specimen	Too viscous or thick film	- Decrease voltage - Increase agitation - Use less viscous electrolyte
Sludge settling on the surface	Insoluble anode product	- Try new electrolyte - Increase temperature - Increase voltage
Roughness or matte surface	Insufficient or no polishing film	- Increase voltage - Use more viscous electrolyte
Waviness or streaks on the polished surface	Insufficient time Incorrect agitation Inadequate preparation Too long time	- Increase or decrease agitation - Use better preparation - Increase voltage or decrease time
Stains on polished surface	Attack after polishing current is off	- Remove specimen while current is still on - Try less corrosive electrolyte
Unpolished spots (bullseyes)	Gas bubbles	- Increase agitation - Decrease voltage
Phases in relief	Insufficient polishing film	- Increase voltage - Use better preparation - Decrease time
Pitting	Polishing too long Voltage too high	- Use better preparation - Decrease voltage - Decrease time - Try different electrolyte

There are many independent variables that affect the electropolishing process. For this reason, a universal theory for this process has not been established. ASTM standard is presented on the possible problems that may arise during the process and the cause and solution of these problems as seen as in Table 1.1. It can be said that the most important variable here is temperature. With an increase in temperature, there is a decrease in bath resistance, plateau current density and electrolyte viscosity. Thus, the anode surface cannot be obtained in the desired quality. High plateau current density generally shortens the electropolishing time. A long electropolishing time may cause undesirable distorted images on the surface [44].

1.7 Titanium

William Gregor discovered the element titanium in England in 1790. Titanium and its alloys are used in many fields due to their advanced physical and mechanical properties (Table 1.1, 1.3). It has properties such as biocompatibility, low density, high corrosion resistance, high strength. It has applications in almost every aspect of our lives, in automotive industry, architecture, aerospace industry, sports equipment, chemical industry, marine applications, safety and security, and medical applications. Titanium and its alloys are widely used in biomaterials. Various implants such as cardiovascular stents, spinal fixation devices are made from pure or alloyed forms of titanium. Their high level of biocompatibility and corrosion resistance properties increase their use in this field. When examined in terms of mechanical behavior, these metals show very similar behavior to bone movement and are one of the rare materials that can adapt to the human body and meet the desired properties. Implants made from commercially pure titanium with low iron content are better adapted to the human body. However, the mechanical properties of pure titanium are not as good as desired. This group of titanium with low mechanical strength is insufficient in hard tissue applications. Since more strength is required in biological devices, grade 4 commercially pure (CP) titanium was used. However, Mahmoodian et al's [45] severe plastic deformation study on CP titanium resulted in an ultrafine grained structure, which gave CP Ti an improved mechanical strength. And the biocompatibility of this

type of material with the human body was found to be considerably better than alloys. Table 1.3 shows the mechanical properties of different titanium grades. Each property of titanium and its alloys determines where and how they are used [46], [47], [48].

Table 1.2 Physical properties of Titanium [49]

Density	4.54 g/cm ³
Electrical Conductivity	2.34×10 ⁶ S/m
Thermal Conductivity	17.0 W/mK
Boiling Temperature	3287 °C
Linear Thermal Expansion Coefficient	8.6×10 ⁻⁶ (°C) ⁻¹

From a scientific perspective, titanium atoms are observed in two fundamentally different structures: the central cubic body (bcc) and the hexagonal tight packing (hcp). These crystal structures are known as alpha (α) and beta (β). In its alloyed or pure form, hexagonal structure indicates alpha and cubic structure indicates beta. There are four grades of titanium alloys: alpha, near alpha, alpha-beta and beta. Commercially pure (CP) titanium has an alpha structure. Adding different elements to this titanium structure produces titanium alloys. With sufficient alloying elements, the beta phase is produced during the heating phase and converted by cooling. These structures are called alpha-beta alloys. According to the ASTM standard, grade 1, 2, 3 and 4 commercially pure (CP) titanium metals are unalloyed. Classification of CP Ti alloys are given in Table 1.2. The difference between the CP Ti grades is the oxygen and iron content. In CP Ti materials, the elements nitrogen and oxygen are present for solid solution hardening. The presence of oxygen is associated with strength and strength

values vary according to the oxygen level. CP Ti materials have different values ranging from 170-480 MPa (Table 1.3).

Table 1.3 Commercially pure Titanium grades alloy compositions [50]

Commercially Pure Titanium Grades	Alloy Composition
CP Ti Grade-1	Ti-0.5Fe-0.4O-0.05N-0.08C-0.015H
CP Ti Grade-2	Ti-0.25Fe-0.08C-0.2O-0.03N-0.015H
CP Ti Grade-3	Ti-0.03Fe-0.2O-0.05N-0.08C-0.015H
CP Ti Grade-4	Ti-0.2Fe-0.15O-0.08C-0.015H

With aluminum (Al) and vanadium (V) added to titanium, a two-phase alloy titanium metal consisting of alpha and beta structure is obtained. This structure contains Ti-6% Al-4% V and is the most commonly used alloy titanium in dental implant applications. With the addition of aluminum and vanadium elements, the strength of the metal is greatly increased compared to unalloyed pure titanium metal. For applications where more hardness is required, Ti-4% Mo-4% Al-2% Sn alloy is used, which is formed by adding molybdenum (Mo), aluminum (Al) and tin (Sn) [50], [51], [52].

The corrosion resistance property is based on a single phase in the alpha structure. To the phase are added small amounts of solid solution strengthening and alpha phase stabilizing elements such as palladium (Pd), aluminum (Al), and oxygen (O). High corrosion resistant alloys are used in industries such as chemical, energy and food to produce corrosion resistant pipes, containers and heat exchangers. Their formability is limited due to their crystalline structure and high work hardening properties. These non-heat-treated alloys have good weldability. However, these alloys are inferior to other alloys in terms of strength properties. With titanium alloys, the desired mechanical properties for the application can be achieved and thus they are one of the main materials used for aerospace and other commercial applications. Ti-5Al-2.5Sn is

one of the most commonly used alpha alloys and is widely preferred for applications such as liquid hydrogen storage tanks found in spacecraft [53].

Table 1.4 Mechanical properties of ASTM Titanium Grades [51], [54]

Grade	Elongation (%)	Yield Strength (MPa)	Elastic Modulus (GPa)	Ultimate Tensile Strength (MPa)
1	24	170	103-107	240
2	20	275	103-107	345
3	18	380	103-107	450
4	15	483	103-107	550
5	10	795	114-120	860

*Grade 5; Ti-6Al-4V

1.8 The Review of Literature

Electropolishing process is an electrochemical treatment process which eliminates the irregularities and gives a smooth, shiny and clean material surface. This process can be used for large or small parts with poor surface finish, simple or complex structure [8]. Patents for electropolishing pure titanium are available from U.S. companies, but not much technical detail is given. Electrolyte compositions generally include sulfuric acid, hydrofluoric acid, acetic acid, and other additional chemicals. The bath temperature was applied in the range of 20-25°C and the current density was 7 A/dm². The studies focused on metal dissolution rate and surface roughness parameters [55]. Some reactions take place during the titanium electropolishing process. Three reactions are observed as given below:

Anodic dissolution: $\text{Ti} = \text{Ti}^{4+} + 4\text{e}$

Oxygen evolution: $4\text{OH}^- = \text{O}_2 + 2\text{H}_2\text{O} + 2\text{e}$

Formation of passive oxide film: $\text{Ti} + 2\text{OH}^- = \text{Ti oxide} + \text{H}_2\text{O} + 2\text{e}$

The oxide layer formed on titanium can vary in thickness and stoichiometry. It is mostly in the form of titanium dioxide, TiO_2 [55].

To date, there have been many electropolishing studies in the literature for pure titanium and its alloys. Koji Fushimi et al. [56] studied on the titanium surface using NaCl-ethylene glycol mixture for investigate the anodic dissolution behavior of titanium. Measurements by surface profilometer showed that repeated surface treatment reduced roughness and increased brightening. The high potential was found to be necessary for the removal of the oxide layer on the surface but did not work well for the brightness effect. Working with an electrolyte solution consisting of choline chloride-ethylene glycol mixture to obtain a mirror-like Ti surface, Wrya O. Karim et al. [57] obtained a very well-finished Ti surface in 30 minutes at low voltage value (10 V). The electrolyte used provided the viscous property necessary for mass transport of ions. It was emphasized that the problems encountered in studies using aqueous electrolyte solution were not observed in the study using ionic liquid. J. B. Mathieu and D. Landolt [58] investigated the anodic polarization behavior of titanium material during electropolishing in perchloric acid-acetic acid solution. The required critical breakdown potential was found and it was observed that below this potential a thick passivating film of TiO_2 was formed, which prevented the metal from dissolving. When the part was polarized at a value above the breakdown potential, pitting was observed on the surface. During the electropolishing process, perchlorate ion was reduced to chloride and this reduction was observed as a heterogeneous reaction on the titanium surface. In a study on commercially pure Ti-Grade2, mechanical polishing, electrolytic polishing and magneto electrolytic polishing were applied and the effect of different polishing processes on corrosion and fatigue behavior was investigated. The smoothest surface with the roughness value $0.13 \mu\text{m}$ was obtained by magneto electrolytic polishing. In the study in which the surface characteristics of the pure titanium part subjected to electropolishing process were investigated, colored anodic film formation was observed at different voltage values. At different voltage values, films of different colors were formed and electrochemical, optical micrograph

tests showed that the titanium oxide layer was broken when 90 V was applied. In the experiment conducted by adding hydrofluoric acid to the electrolyte solution of sulfuric acid, the blue color formed on the part became transparent and a film layer consisting of porous nano-channels was formed [59]. It was clearly stated that the low voltage was not effective on the entire sample in the electropolishing process of commercially pure titanium, and that a polishing effect was observed at the edges of the part and an oxide layer was formed on the surface. Even if the time was increased, a yellowish oxide layer was observed at low voltage value [60]. In a study using sulfuric acid electrolyte with very low temperature parameters (20°C to -70°C), a sharp decrease in current density was observed as the temperature decreased. It was noted that the pitting region was observed at high voltage values as the temperature decreased, but was not observed even at high voltage values at -70°C. It was found that as the acid molarity increased, the current density decreased and the voltage range widened. At -70°C, they stated that salt film was formed on the metal surface also oxygen and sulfur content was found [61].

It was confirmed that titanium can be polished by using sulfuric acid-ethanol mixture instead of sulfuric acid-methanol electrolyte in the study by Ching An Huang et al [62]. In order to increase the efficiency of the surface polishing process, water was added to the electrolyte solution at different ratios, and good results were obtained in studies with composition of 0.1% and 1% H₂O by volume. Electrolyte solutions consisting of alcoholic ingredients are safe for use. Studies have shown that alcohol-containing electrolytes are effective in removing the viscous thickness in the electropolishing process of Ti surfaces. In the study using ethyl alcohol, isopropyl alcohol, aluminum chloride and zinc chloride electrolyte solution, the viscous layer was formed on the titanium part during the electropolishing process and it was emphasized that shaking was necessary to remove this layer. The recommended surface roughness value of 0.09-0.21 µm was achieved to minimize bacteria and plaque retention of titanium implants used in dental treatments [63]. In the study conducted by adding different percentages of ethanol to the electrolyte solution, the SEM image (Figure 1.6) obtained showed how important the electrolyte composition is. By adding 20% ethanol by volume to the ethylene glycol-NaCl electrolyte solution, the Ti sheet was electropolished and a mirror-like surface was obtained. At ethanol

additions above this ratio, non-uniform electron exchanges between ions occurred [64].

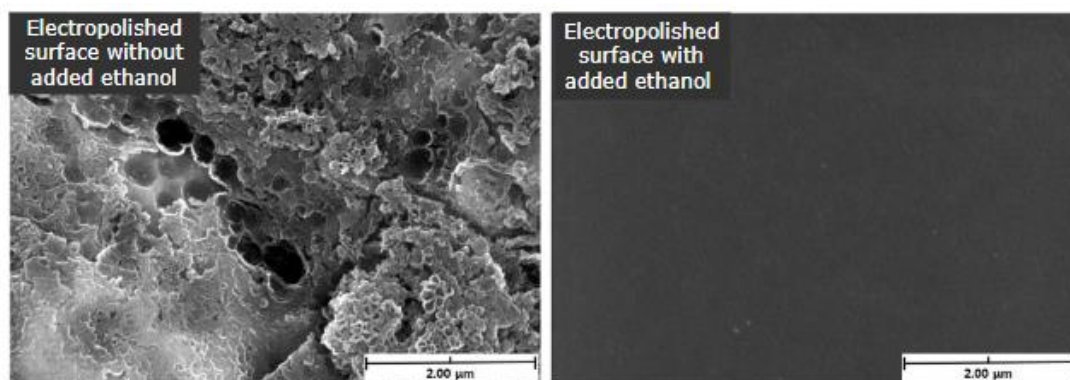


Figure 1.7 SEM images of Ti sheets after electropolishing with and without ethanol addition [64]

CHAPTER 2

EXPERIMENTAL PROCEDURE

2.1 Preparation

The experiments were performed on a commercial TG-2 rod which has 5 mm diameter. The TG-2 material element analysis is given in the Table 2.1. Before the experiments TG-2 rod was mechanically polished using lathe with emery papers 360, 400, 600, 800 and 1200 grit. For pre-cleaning process, the TG-2 rod cleaned in ultrasonic bath with HCl and C_3H_6O for 60 s, rinsed with distilled water and dried. In the experiments with non-mechanically polished parts, only the cleaning process was applied as the pre-treatment. As the cathode material a copper (Cu) folio is used.

Table 2.1 Element analysis of the TG-2 rod used in the experiments

Element	Ti	Al	Fe	Cr	Pd
Weight %	>99.67	0.18	0.06	0.05	0.02

The electrochemical cell contained 2 liters of electrolyte solution. The electrolyte solution content was constant throughout the entire study. Electrolyte solution contained 10.5% HF and 89.5% $C_2H_6O_2$ by volume percentage [65]. 50% HF acid was used and prepared from 70% HF using distilled water.

2.2 Experimental Set-Up

The experimental setup is given in Figure 2.1. Different electrolyte temperatures, current density values, and polishing time were applied to the mechanically polished and non-polished TG-2 specimens. The agitation speed, electrolyte composition, area

of the anode material, electrode gap and cathode material were constant throughout the whole study. A summary of the study and its fixed and control parameters are given in Table 2.2.

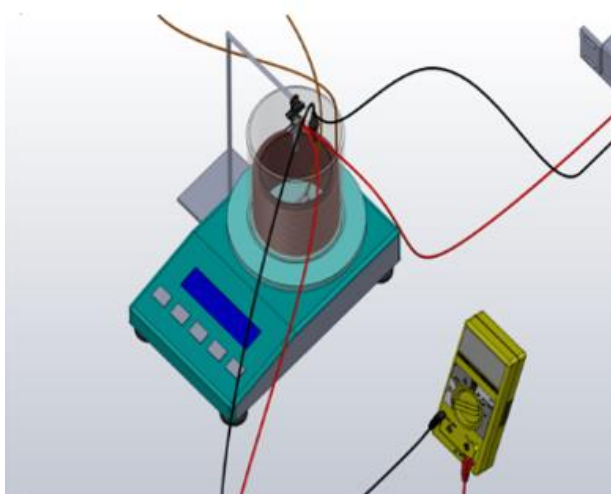
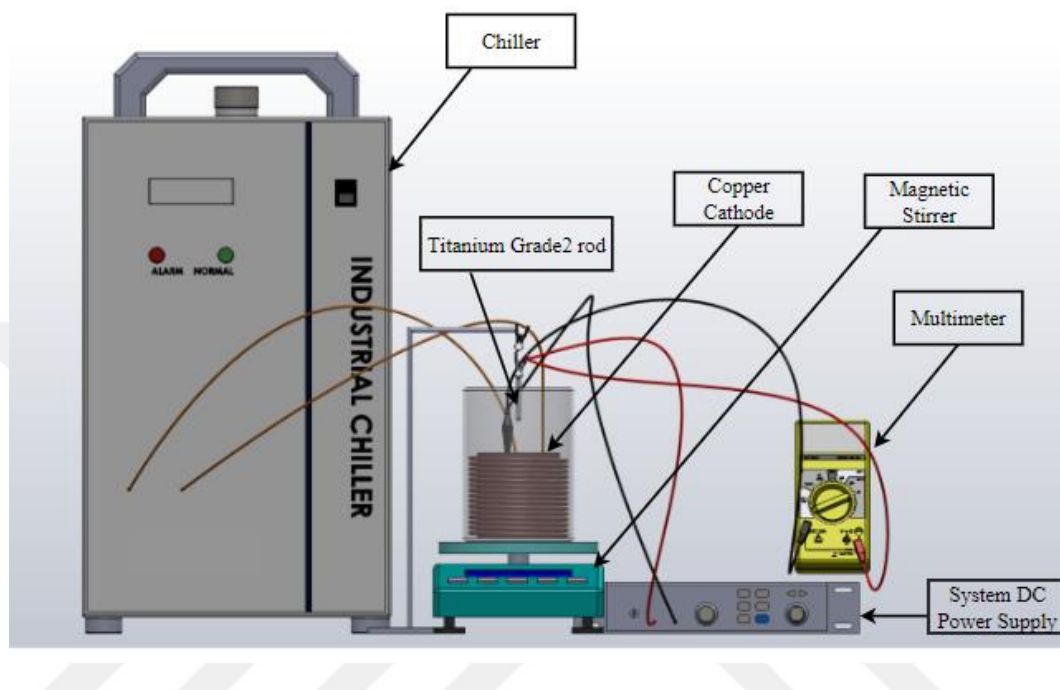


Figure 2.1 Experimental set-up drawing

Table 2.2 Fixed and control parameters of the experimental study

Variable	Parameters	Settings of The Parameters
Fixed Parameters	Electrolyte Composition	10.5% HF 89.5% C ₂ H ₆ O ₂
	Agitation Speed (rpm)	500
	Electrode Gap (mm)	50
	Cathode Material	Copper Folio
	Anode Material	Titanium Grade-2
	Area (dm ²)	0.0157
Control Parameters	Anode Material Initial Roughness (μm)	Ra ≅ 0.68 Ra ≅ 0.15
	Current Density (A/dm ²)	11, 22, 33, 44
	Polishing Time (min.)	3, 6, 9, 12
	Temperature (°C)	20, 30, 40

At low temperature studies (20°C), a chiller (CLS Scientific CRLC-08C) was used for cooling the electrolyte. For the experiments at and above the room temperature and the agitation of the electrolyte solution, a digital hotplate with magnetic stirrer (JSR JSHS-18D) was used. The current/voltage was applied using a DC power supply (Agilent Technologies N8761A) and the voltage was also confirmed by a multimeter (Keysight U1240). Surface roughness determination was performed with the surface roughness tester (Mitutoyo, SJ-400). Optical microscopy (OM) (Nikon, Eclipse E200) and

Scanning electron microscopy (SEM) (Zeiss, EVO MA10) were used to examine the surface analysis of the TG-2 samples.

After electro-polishing, the part was quickly removed and washed thoroughly with distilled water. As a final step, it was washed with C_3H_8O to prevent water marks from remaining on the surface of the part and left to dry.

2.3 Surface Quality Analysis

The change in the roughness value of the sample after each experiment was analyzed by surface roughness test. For each surface, 5 measurements were taken and the average roughness value was calculated. The samples with the best results were analyzed by optical microscopy to obtain detailed surface images. A summary of all the steps of the study is shown in the flowchart below (Figure 2.2).

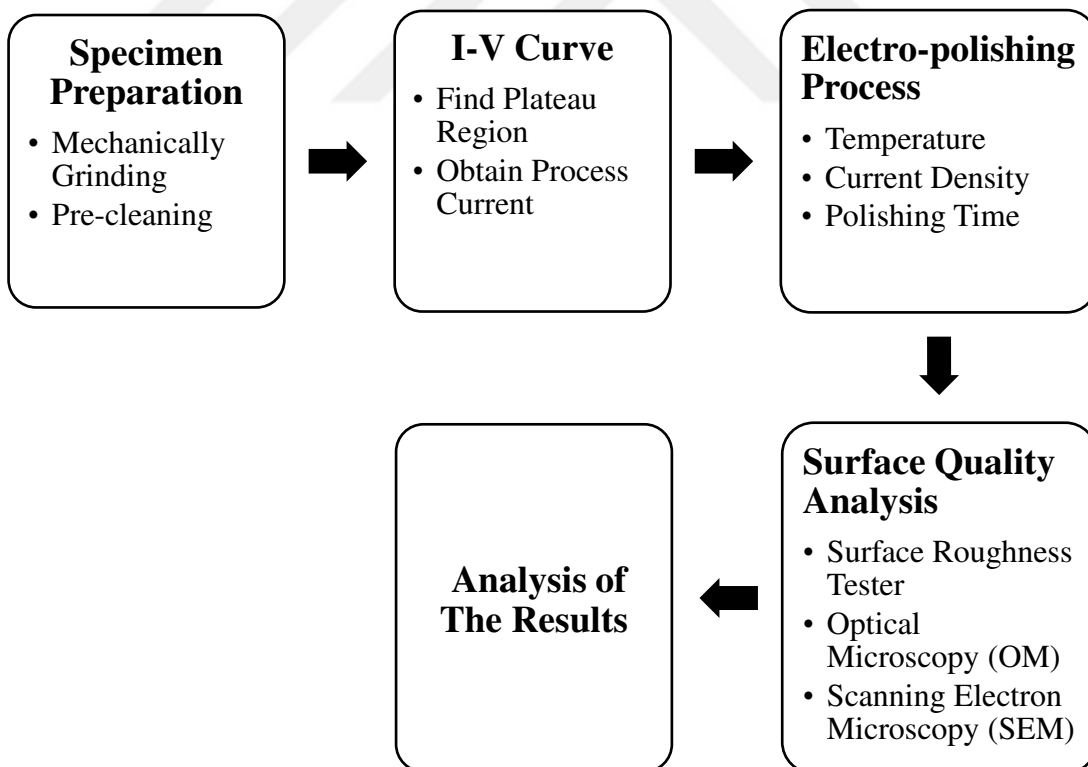


Figure 2.2 A summary of the experimental study

CHAPTER 3

RESULTS AND DISCUSSION

3.1 Voltage-Current Density Relationship

Figure 3.1 shows the relationship between electropolishing voltage and current density at different temperatures. As we can see from the voltage-current density graph, the voltage values increased with increasing current density but decreased with increasing temperature. With an increase in temperature, the number of free carriers increases and this leads to a lower voltage [66].

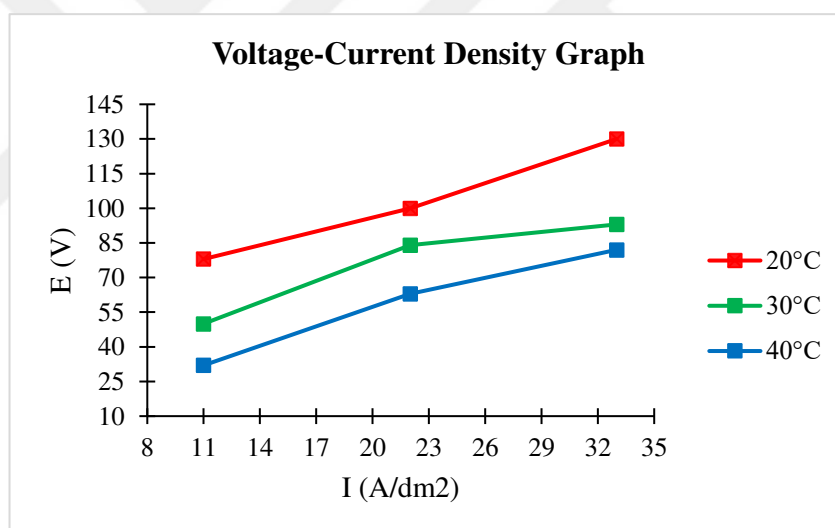


Figure 3.1 Electropolishing voltage and current density relationship of TG-2 sample at different temperatures.

3.2 Roughness vs. Current Density Relationships

In experiments performed at 44 A/dm^2 condition, the samples showed poor surface properties such as high roughness value and burnt surface (Figure 3.2). Because of the results, all the experiments for 44 A/dm^2 condition were not completed.



Figure 3.2 Electropolished TG-2 sample at 20°C , 44 A/dm^2 , 3 min. conditions

Two different surfaces, mechanically non-polished and polished samples were used in this study. Different roughness values were obtained for two surfaces which have different initial roughness values. Figure 3.3 and Figure 3.4 shows the roughness values of mechanically non-polished TG-2 rod at different temperature and current density conditions. As seen as in the graphs, when the applied current density is increased, the surface roughness is decreased. In literature, it was found that high potential is not suitable for polishing but is necessary to remove the first oxide layer. Polarization at lower potential where the limiting current flows followed by initial polarization is shown to be effective for polishing the surface [56]. When the experimental results at 20°C is examined, it is seen that high current density has a great effect on roughness. Increasing current density had a positive effect on roughness at

all temperatures. The best surface result for mechanically non-polished TG-2 rod was obtained in the 12 min. of the experiment at 33 A/dm² current density and 20°C.

3.3 Roughness vs. Temperature Relationships

In a study on the titanium part, it was reported that the temperature increase (25°C) formed a regular nanostructure and contributed to the reduction of surface roughness [67]. In this study, no positive correlation was observed between the temperature and the roughness of the surface as can be seen in Figure 3.3. Almost, at all temperature and current density values, surface roughness values increased with increasing time. Temperature accelerates metal removing rate, but the results show that the electropolishing effect was poor. As the temperature increased, the decrease in roughness value slowed down. This is because, excessive temperature accelerates anodic dissolution and chemical attack during polishing and an inhomogeneous polishing process takes place on the surface. This causes pits and distortions on the surface. During the electropolishing process, an exothermic reaction occurs and the already high temperature continues to rise with the heat released. The overheated electrolyte solution, together with the heat that is not distributed homogeneously, becomes aggressive on the metal surface and accelerates local dissolution [68]. At this point, finding the temperature at which the process is most efficient is very important for a quality surface.

Roughness – Temperature Graphs

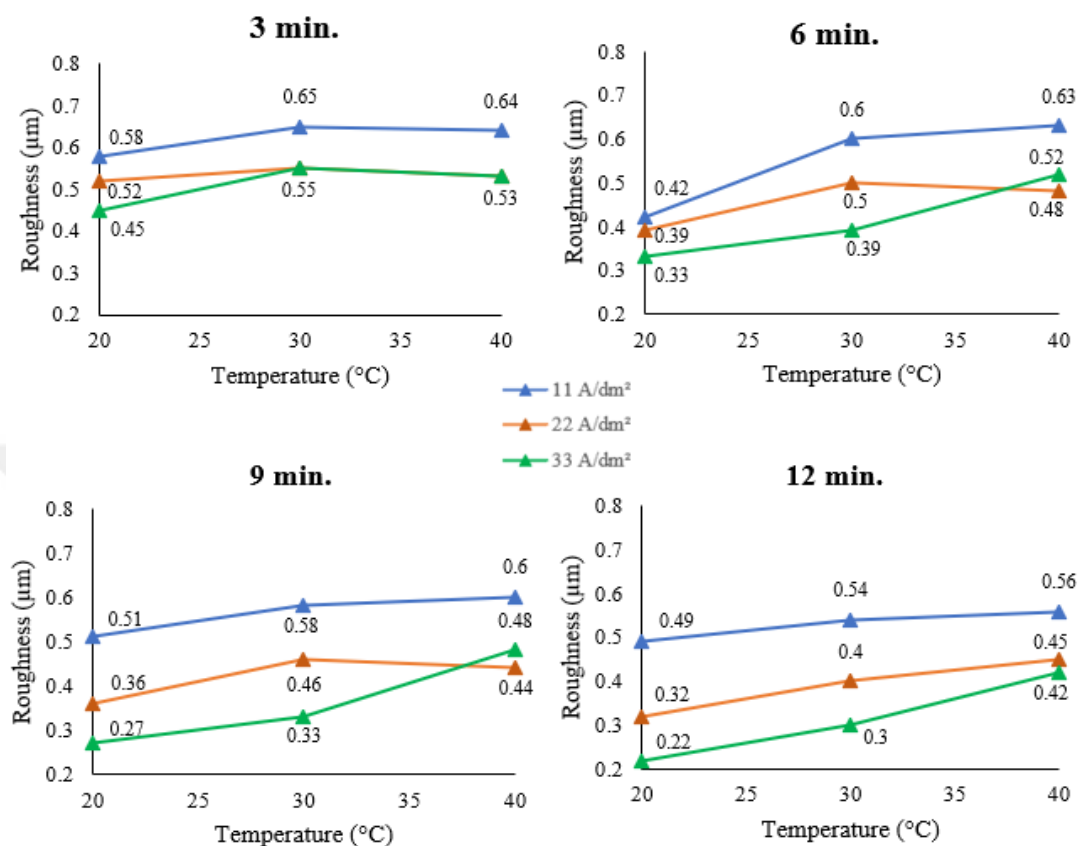


Figure 3. 3 Roughness-temperature graphs of mechanically non-polished TG-2 sample at different polishing times and current densities.

3.4 Roughness vs. Time Relationships

Time is the one of the best important parameters. As the time increases, there may not always be a linear improvement on the surface. The amount of roughness removed from the surface increases and the surface gradually improves with increasing time. However, this improvement continues up to a certain point and as the time increases further, it starts to create the opposite effect on the surface. The surface, which has reached a certain level of smoothness, continues to give metal ions from the surface when exposed to further processing and roughness begins to form again. As seen in

Figure 3.4, at 11 A/dm² current density, the surface roughness increased after 6 min. However, for the other cases, roughness values of mechanically non-polished parts increased with the increasing time.

Roughness – Time Graphs

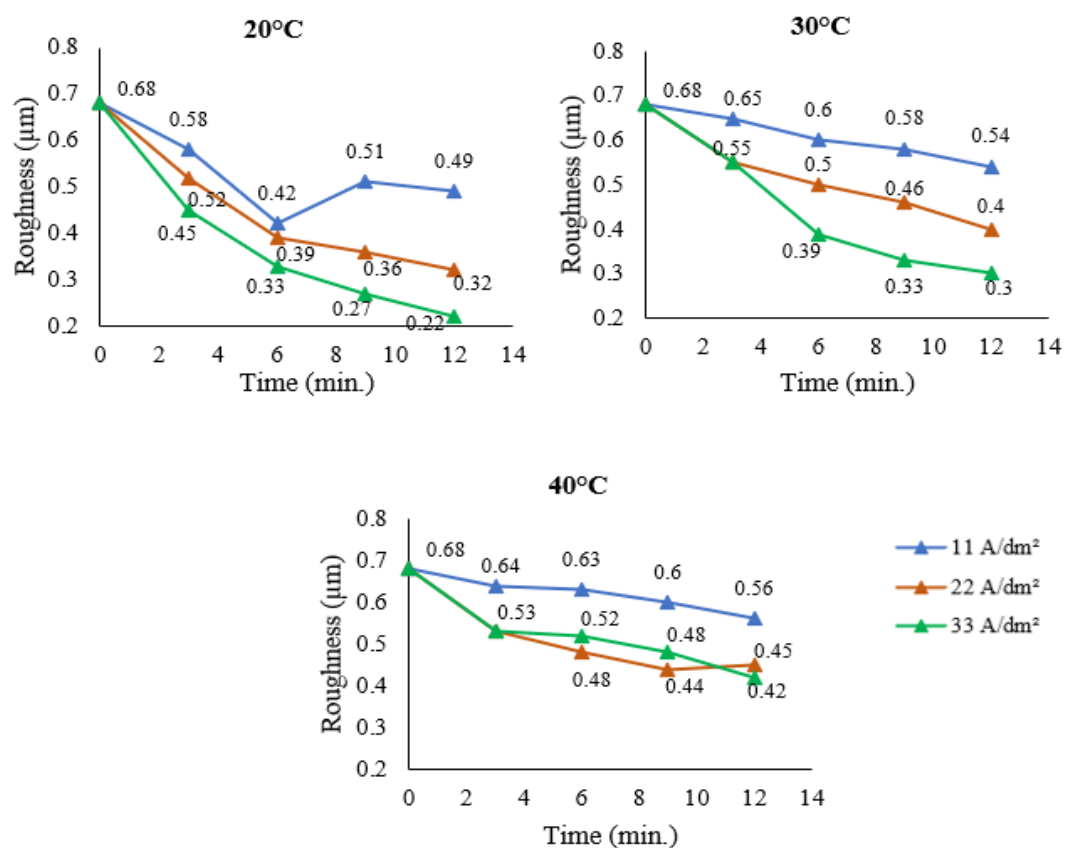


Figure 3. 4 Roughness-polishing time graphs at different temperatures and current densities of mechanically non-polished TG-2 sample.

3.5 Initial Roughness Effect

The initial roughness value of the part affects the result of the electropolishing process. The results of the electropolishing study on the mechanically sanded part clearly show this. The initial roughness value of the part was reduced from 0.68 to 0.15 μm by mechanical sanding. Again, the same temperature, current density and time parameters were used for this part. When Figure 3.5 and Figure 3.6 are examined, it is seen that the mechanically polished part shows a general improvement. In the electropolishing process performed on the rough TG-2 rod, the surface roughness was reduced to a minimum of 0.22 μm . With the mechanical sanding process, the initial roughness value was reduced to 0.15 μm and this value was reduced further with the electropolishing process. This finding was in parallel with the study where a mirror-like, shiny, smooth finish was achieved when sanding was performed as a pre-treatment [63].

In the studies with the sanded part, the surface healing was very slow because of the low amount of rough metal layer that needs to be dissolved on the surface. In the experiments at 30°C and 33 A/dm², as the electropolishing time increased, the surface deteriorated and the roughness value increased. The same situation was observed at 40°C, 11 A/dm² experiments.

Roughness – Temperature Graphs

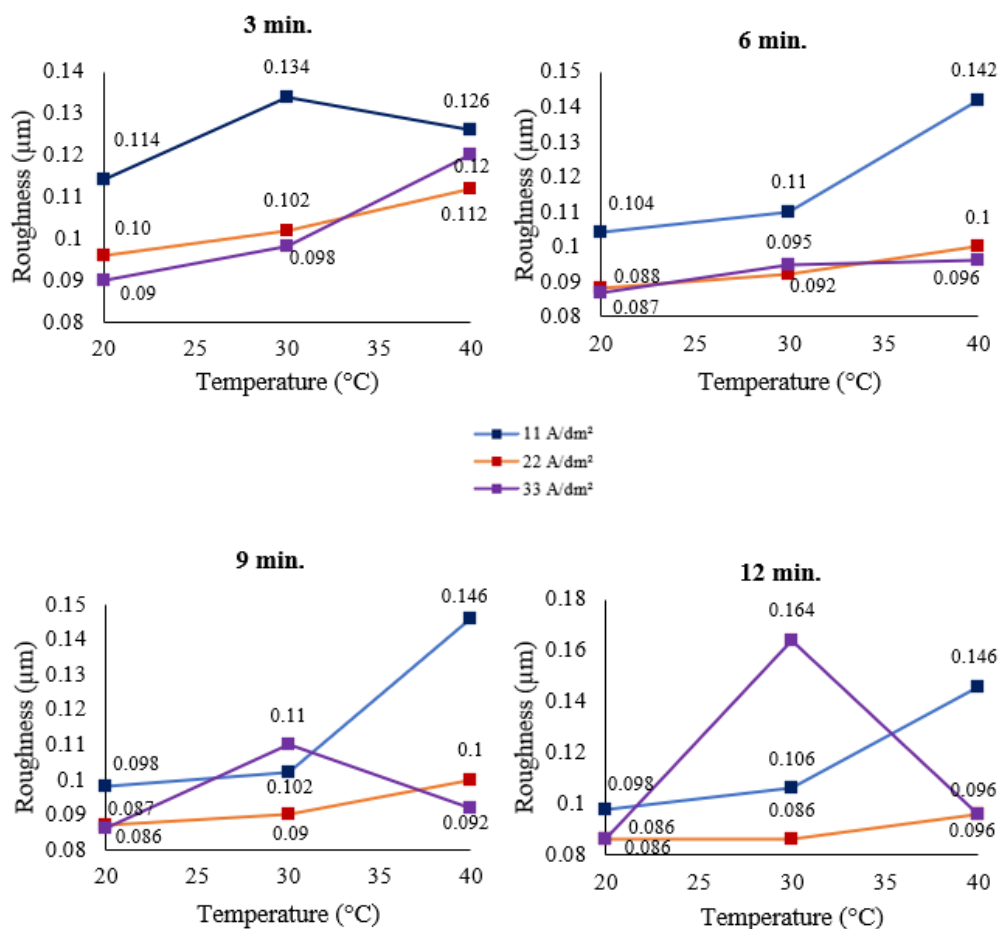


Figure 3. 5 Roughness-temperature graphs of mechanically polished TG-2 sample at different polishing times and current densities.

As seen in Figure 3.6, the lowest roughness value was reached in 12 minutes for the 22 and 33 A/dm² experiments at 30°C and in the 33 A/dm² experiment at 40°C. At all current densities, the best surface results seem to be obtained in the 20°C experiments. 22 A/dm² and 33 A/dm² current densities almost gave the same results. For parts with a larger surface area, the high current density makes the process difficult to control. Since it is difficult to work at high current and voltage values, it can be assumed that a current density of 22 A/dm² gives the best results.

Roughness – Time Graphs

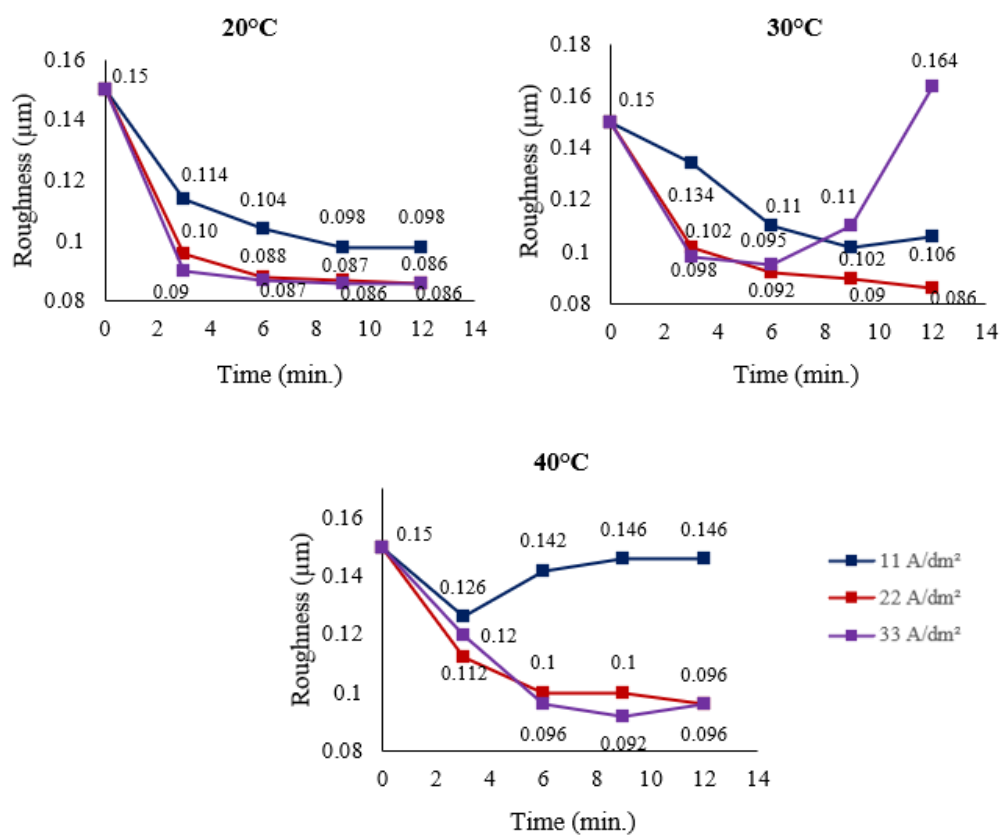


Figure 3. 6 Roughness-polishing time graphs at different temperatures and current densities of mechanically polished TG-2 sample.

Table 3.1 shows the best roughness values of the mechanically non-polished surface, mechanically sanded surface, electropolished surface before mechanical sanding and electropolished surface after mechanical sanding.

Table 3.1 Average roughness values of the different Ti surfaces.

Surface	Ra (μm)
Mechanically Non-Polished	0.68 $\pm$ 0.003
Mechanically Sanded	0.15 $\pm$ 0.003
Electropolished Before Mechanical Sanding (20°C, 33 A/dm ² , 12 min.)	0.22 $\pm$ 0.003
Electropolished After Mechanical Sanding (20°C, 22 A/dm ² , 12 min.)	0.086 $\pm$ 0.003

3.6 Optical Microscopy (OM) Results

Figure 3.7 shows the TG-2 sample with mechanically non-polished surface and electropolished surface at 20°C, 33 A/dm², 12 min. conditions, which was the best result for the mechanically non-polished surface. The difference between the matte sample surface before electropolishing and shiny appearance after electropolishing can be seen in Figure 3.7. Even though a high amount of material was dissolved, it is also visible that the surface healing is limited and roughness is present on the surface.

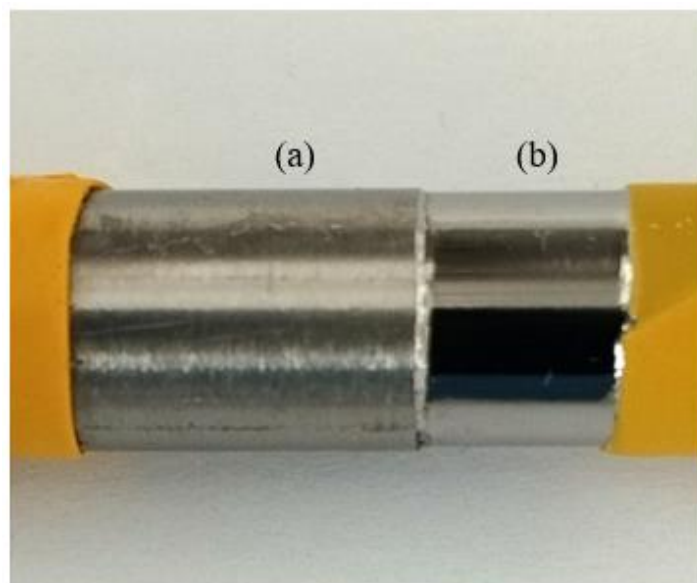


Figure 3.7 Image of TG-2 sample (a) mechanically non-polished surface (b) electropolished surface at 20°C, 33 A/dm², 12 min. conditions.

Surface images before and after electropolishing were examined using an optical microscope. The surface morphology was seen in detail at different magnifications. Figure 3.8, Figure 3.9 and Figure 3.10 show the difference at different magnifications between mechanically non-polished rough surface and electropolished surface at 20°C, 33 A/dm², 12 min. conditions. Images show the surface healing with electropolishing process. However, although the electropolishing process improved the surface, it is clear from the optical microscope images that there are still defects on the surface (Figure 3.10). In the study carried out on this surface, the roughness value could be reduced to a minimum value of 0.22 μm . There is also a noticeable color change on the surface. In Figure 3.7, this discoloration is not visible, but in the optical microscope images, the surface appears brownish. This color change is thought to be due to the high current density.

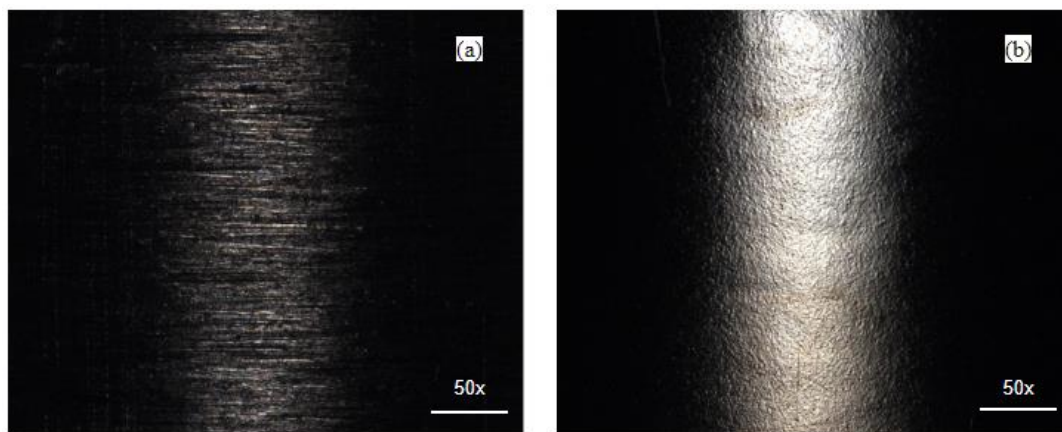


Figure 3.8 Optical micrograph of TG-2 sample at 50x magnification (a) mechanically non-polished rough surface (b) electropolished surface at 20°C, 33 A/dm², 12 min. conditions.

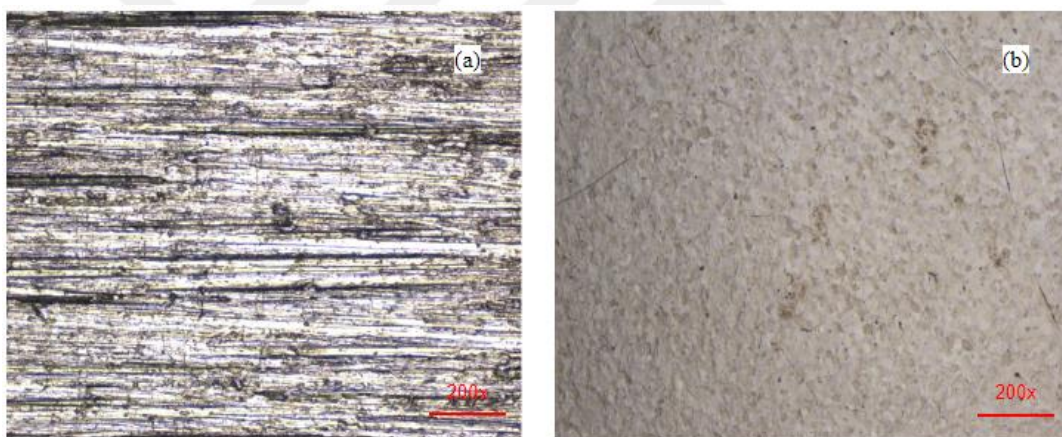


Figure 3.9 Optical micrograph of TG-2 sample at 200x magnification (a) mechanically non-polished rough surface (b) electropolished surface at 20°C, 33 A/dm², 12 min. conditions.

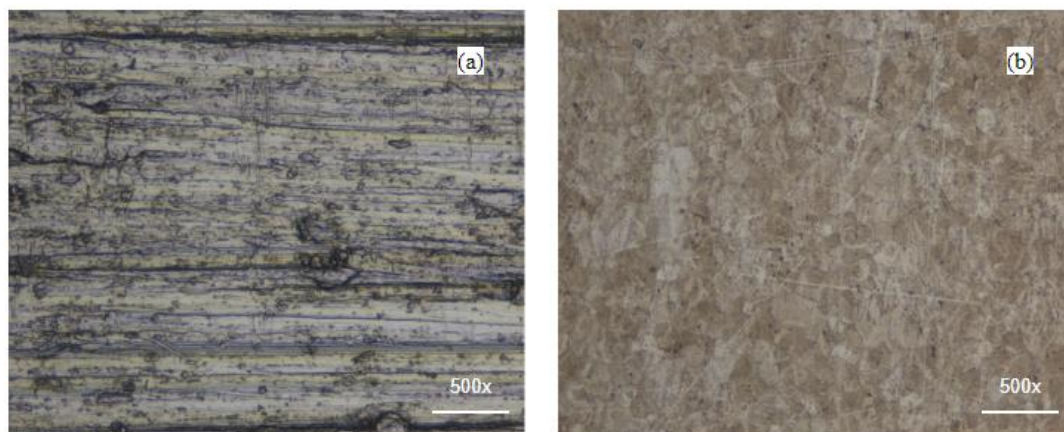


Figure 3.10 Optical micrograph of TG-2 sample at 500x magnification (a) mechanically non-polished rough surface (b) electropolished surface at 20°C, 33 A/dm², 12 min. conditions.

Figure 3.11 shows images of mechanically sanded and electropolished surfaces. The difference of the surfaces is clearly visible. It was found that the experiment performed at 20°C, 22 A/dm², 12 min. gave the best result on the TG-2 sample. The roughness value of this visually bright and smooth surface is 0.086 μm , the lowest value reached in the study.

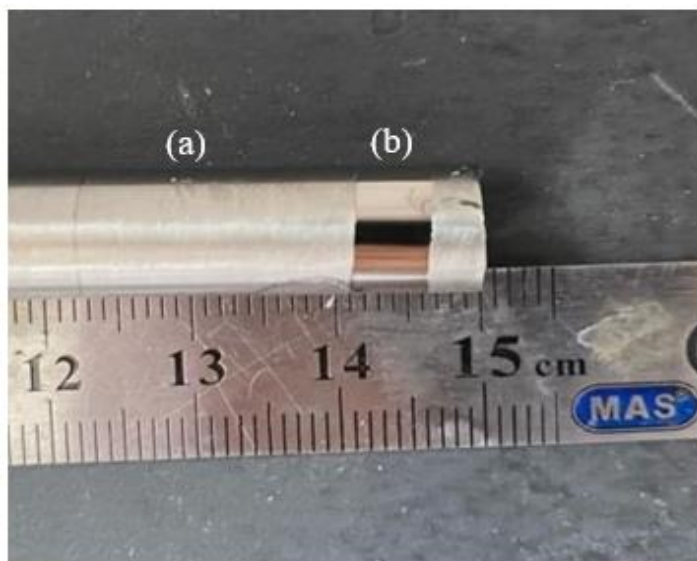


Figure 3.11 Image of TG-2 sample (a) mechanically polished surface, (b) electropolished surface at 20°C, 22 A/dm², 12 min. conditions.

Figure 3.12, Figure 3.13 and Figure 3.14 show a clear difference between the surface morphology of the mechanically sanded part with reduced surface roughness and the electropolished part. It is clear that the images of the sanded surface show the roughness on the surface but show a much-improved morphology compared to the unsanded surface. As a result of 22 A/dm² current density, it is clearly evident in the optical microscope images that there is no color change on the surface, this current density does not damage the part and leaves a clean surface. It is seen that the roughness value was reduced, but despite this, a completely flat surface could not be obtained, and there are slight defects on the surface.

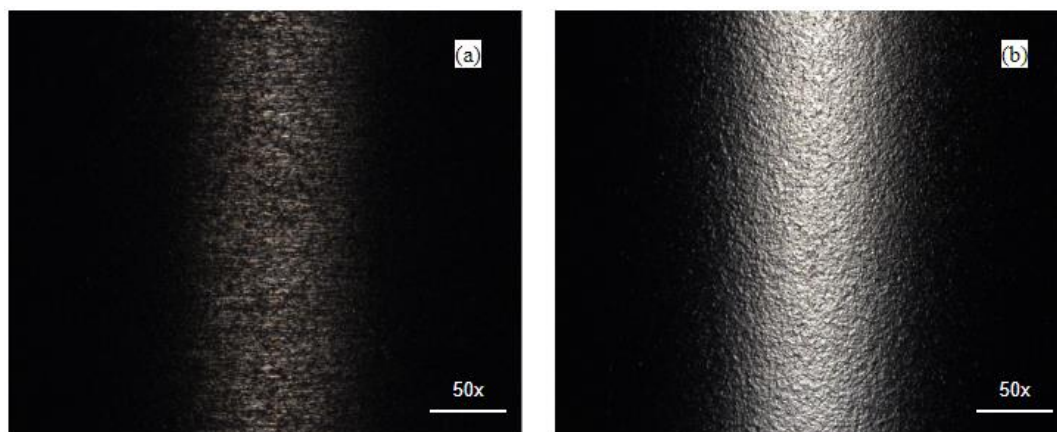


Figure 3.12 Optical micrograph of TG-2 sample at 50x magnification (a) mechanically polished surface (b) electropolished surface at 20°C, 22 A/dm², 12 min. conditions.

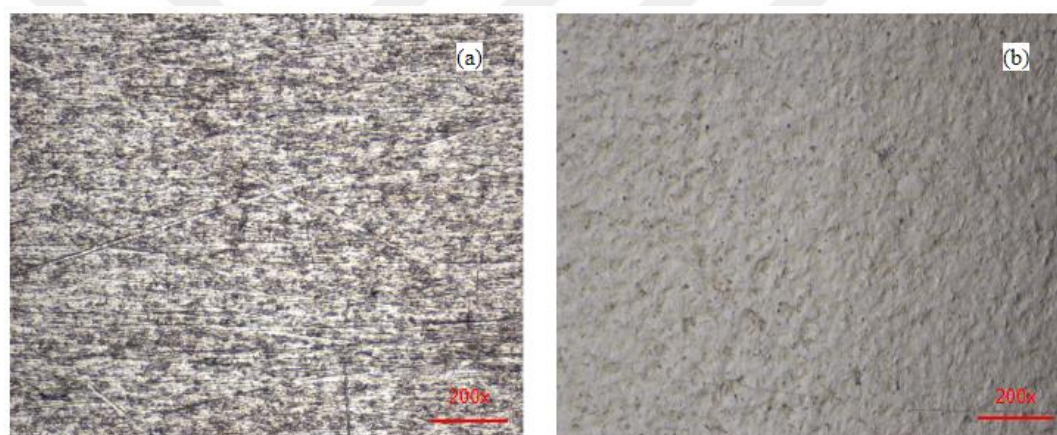


Figure 3.13 Optical micrograph of TG-2 sample at 200x magnification (a) mechanically polished surface (b) electropolished surface at 20°C, 22 A/dm², 12 min. conditions.

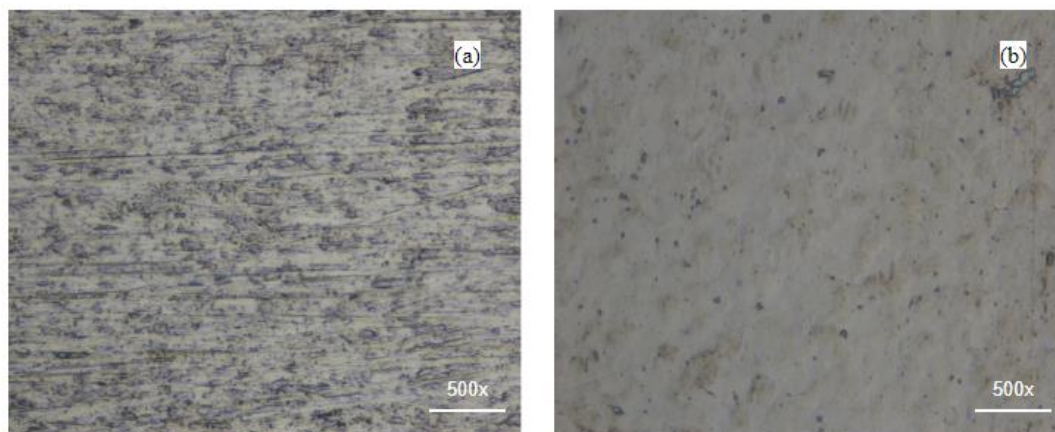


Figure 3.14 Optical micrograph of TG-2 sample at 500x magnification (a) mechanically polished surface (b) electropolished surface at 20°C, 22 A/dm², 12 min. conditions.

3.7 Scanning Electron Microscopy (SEM) Results

The morphology of electropolished samples were characterized by SEM and the obtained images are presented in Figures 3.15-3.17. The features of polished and unpolished TG-2 surface at the boundary are shown in the SEM micrograph given in Figure 3.15. Figure 3.16 and 3.17 show the surface healing at different magnifications. Qualitatively, clear information about the morphological surface change can be obtained from SEM images. For mechanically non-polished surface, the best electropolishing result was obtained at 20°C temperature, 33 A/dm² current density and 12 minutes conditions. It is seen that the roughness on the surface disappears and a clean smooth surface is obtained. However, it is also seen in the SEM images that there are still scratches on the surface and the roughness cannot be completely eliminated.

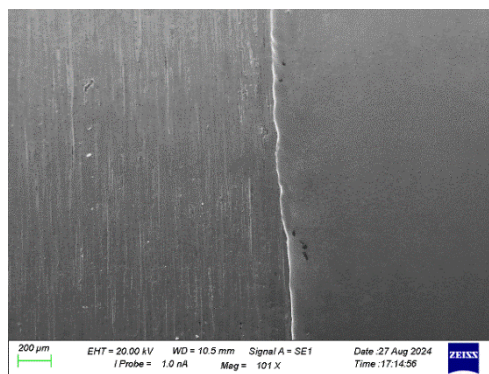


Figure 3.15 Mechanically non-polished and electropolished surface boundary SEM image of TG-2 sample at 100x magnification.

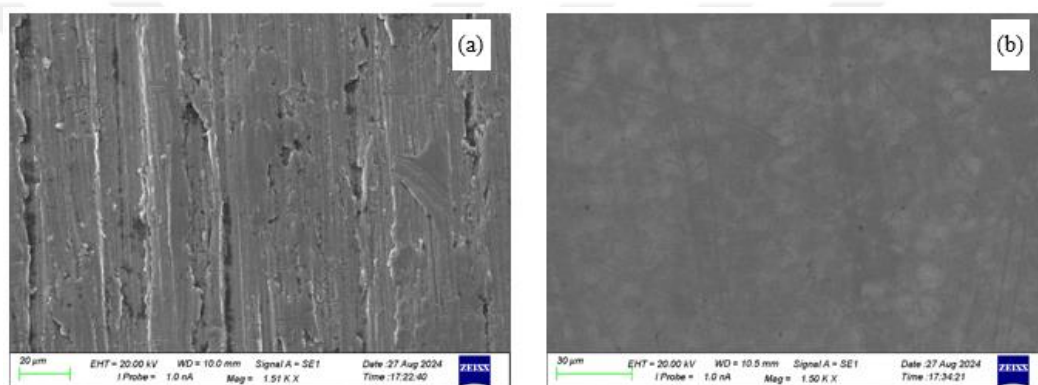


Figure 3.16 SEM images of TG-2 sample at 1500x magnification (a) mechanically non-polished rough surface (b) electropolished surface at 20°C, 33 A/dm², 12 min. conditions.

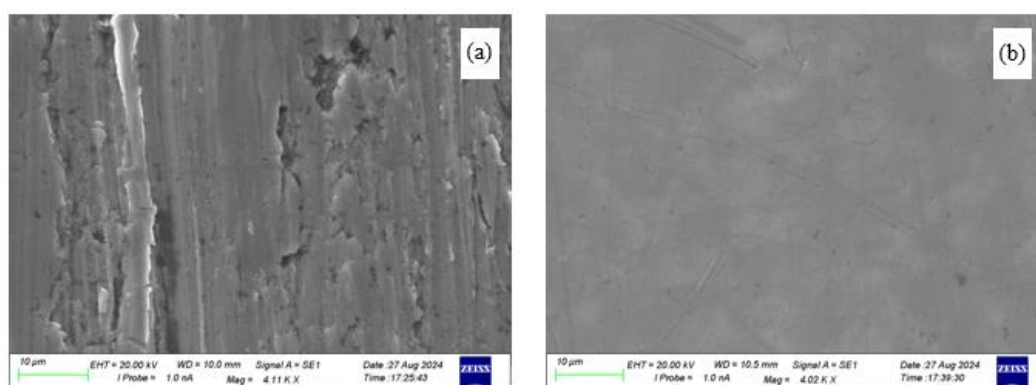


Figure 3.17 SEM images of TG-2 sample at 4000x magnification (a) mechanically non-polished rough surface (b) electropolished surface at 20°C, 33 A/dm², 12 min. conditions.

Electropolishing process for the mechanically polished surface, gave the best result at 20°C temperature, 22 A/dm² current density, 12 minutes conditions. It is already mentioned that the initial surface roughness affects the result and this effect is quite clear in the SEM images given in Figure 3.18, Figure 3.19 and Figure 3.20. This surface is much smoother and cleaner than the electropolishing result of the mechanically non-polished surface.

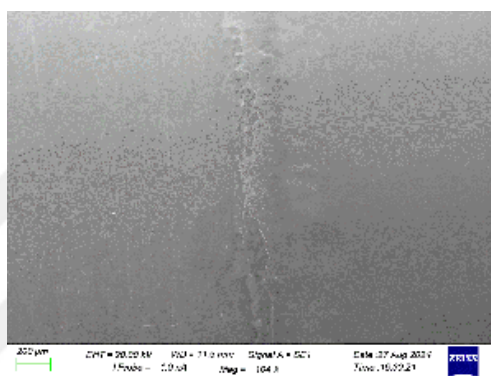


Figure 3.18 Mechanically polished and electropolished surface boundary SEM image of TG-2 sample at 100x magnification.

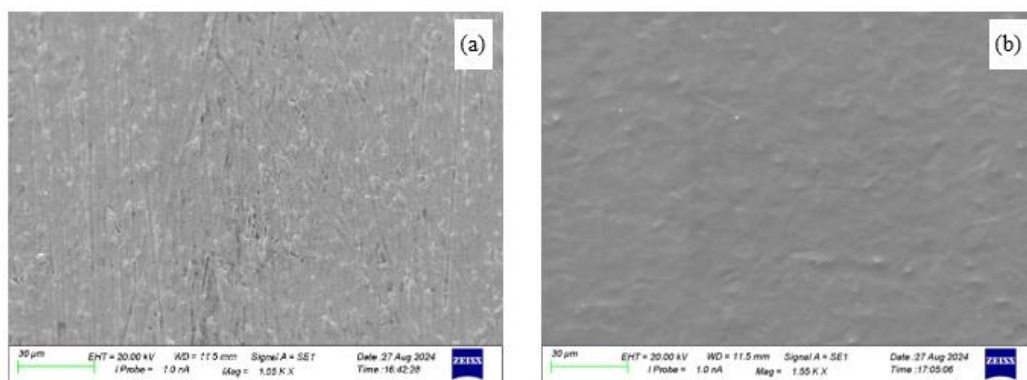


Figure 3.19 SEM images of TG-2 sample at 1500x magnification (a) mechanically polished surface (b) electropolished surface at 20°C, 22 A/dm², 12 min. conditions.

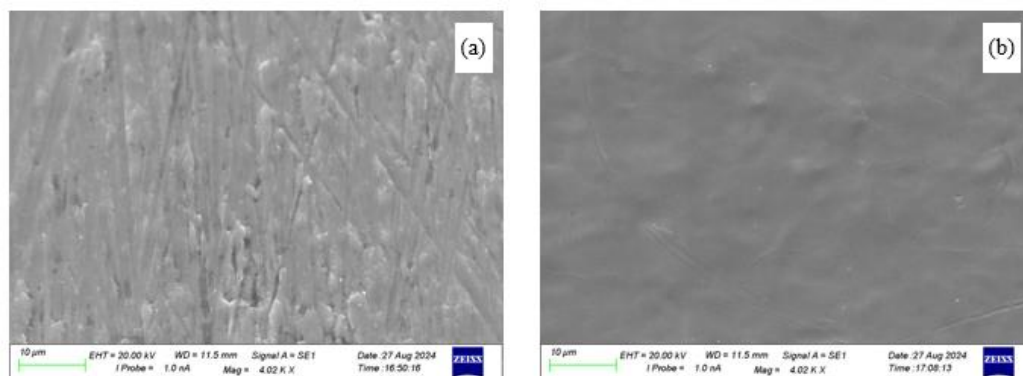


Figure 3. 20 SEM images of TG-2 sample at 4000x magnification (a) mechanically polished surface (b) electropolished surface at 20°C, 22 A/dm², 12 min. conditions.

CHAPTER 4

CONCLUSIONS

This study investigated the electropolishing character of Titanium-grade 2 material in hydrofluoric acid and mono ethylene glycol composition at different temperature, current density and time parameters. The results show the part surface burned due to the high current density in the experiments performed at 44 A/dm².

It was found that the initial roughness value of the part to be electropolished directly affected the surface quality of the final product. In the experiments carried out on the surface of non-mechanically polished part, the best result was obtained as 0.22 µm at 20°C, 33 A/dm², 12 min. Optical microscope images of this surface showed a color change due to high current density. In the electropolishing process performed on the part with mechanical polishing as the pre-treatment, the best result was 0.086 µm in the 20°C, 22 A / dm², 12 min. experiment. Therefore, in metal parts with rough and damaged surfaces, it may be necessary to apply pre-treatment before electropolishing to obtain a smooth, shiny mirror-like surface.

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