Nano Electrochemical Polishing of Titanium Alloy using Ionic Liquid

By

Ashraf Yaaqob Omar Bayagoob
(14625)

Dissertation submitted in partial fulfilment of
the requirements for the
Bachelor of Engineering (Hons)
(Mechanical)

May 2015

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CERTIFICATION OF APPROVAL

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Approved by,

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(Dr. Chandan Kumar Biswas)

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UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK

May 2015

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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ASHRAF YAAQOB OMAR BAYAGOOB

ABSTRACT

The titanium-aluminium-vanadium alloy (Ti-6Al-4V) is the most commonly used in bones, dental and articular prosthetics where high final surface quality is required. Electrochemical polishing is one of the method used to achieve a very low surface roughness. The classical electrochemical polishing process is done by using standard acidic solutions which associated with toxic and expensive disposal. The effect of voltage, rotation speed and polishing time on the surface roughness were investigated for electro-chemical mechanical polishing of Ti-6Al-4V using1-ethyl-3-methylimidazolium acetate. The experiment is conducted on a normal polishing machine which has been modified to suit the process. The ionic liquid acting as an electrolyte, so once the potential applied electrochemical dissolution and mechanical polishing occur at the same time. Then surface morphology is measured using AFM and SEM. The results shows that the surface roughness indexes gradually decreases with time of the process and it's recommended to be in the range of 6-7 minutes. In addition, high voltages are not recommended for the process. Based on SEM and AFM analysis high rotation speed (150-200 rpm) and average voltages (35- 45 V) are the best input parameters for the process which result smooth surface with uniform scale formation.

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CHAPTER 1

INTRODUCTION

1.1 Project Background:

In the recent years, the demand of new products that required complicated shapes and less tolerance has increased rapidly. As a result, many new materials have been introduced to the market. For example, Titanium alloys is one of the complicated materials in the industrial market. In fact, the usage of Titanium alloys increased wildly in several industrial sectors and various applications. Titanium alloys possess excellent properties such as high strength, light weight, corrosion resistance and weld ability that are suitable for fabrication of the aerospace frame, production of engine components and biomedical applications. Titanium alloys are commonly used as an implant material in the human body and Ti-6Al-4V is the most famous alloy used among other several types. Titanium alloys are chemically reactive with most cutting tools and it has low thermal conductivity which made the traditional machining techniques economically expansive.

1.2 Problem statement:

Less surface roughness is very important property for aerospace airframe fabrication and biomedical applications and it can be obtained using Electrochemical Polishing (ECP). The classical practice of electro chemical polishing for this type of alloy is using aqueous solutions. However, there are some problems in using electro chemical polishing process because of a strong passive film on the surface of the titanium. So, perchloric acid used for the polishing process with applying of high voltage current about 30-60 V. In addition, such a process has many hazards related problems because the acids used are potentially explosive. Moreover, the disposal of the concentrated acids is very costly and time consuming. In the recent years, ionic liquids (liquid salts in room temperature) has shown a promising result in the electrochemical polishing process with a safer and more economical approach. An extensive research work still required to determine the suitable machining input parameters of Titanium ECP using ionic liquids.

1.3 Objectives and scope of study:

This project aims to:

1) To study the effect of machining input parameters variation on the surface roughness on electrochemical polishing of Ti-6Al-4V using1-ethyl-3-methylimidazolium acetate (ionic liquid).
2) To make recommendations on input parameters for achieving nano-polishing scale on titanium alloy using ionic liquid.

The scope of the study is limited to instigate the variation of only three of the input machining parameters which are voltage, rotation speed, and polishing time with an experimental method. Then it is going to study the effects of the input parameters on the surface roughness, scale formation thickness and grain formation after the electro chemical mechanical polishing of the titanium alloys. On the other hand, the study is focused on using one type of ionic liquids as electrolyte during the polishing process which is 1-ethyl-3-methylimidazolium acetate.

CHAPTER 2

LITERATURE REVIEW

2 Project background and citation:

Titanium and its alloys are advanced metallic materials possessing many interesting features and properties with outstanding corrosion resistance in a wide variety of environments. Because of numerous advantageous properties, they are used in aeronautics, the automotive industry, in jewellery and in biomedical engineering.

Although many types of titanium alloys are available, the titanium-aluminium-vanadium alloy (Ti-6Al-4V) is the most commonly used, since its physical and mechanical properties are superior to those of commercially pure titanium (CP Ti, 99.8% titanium). Compared to CP Ti, Ti-6Al-4V has greater flexural strength (890 MPa vs390 MPa), superior hardness (350 VHN vs 160 VHN), and a slightly higher thermal expansion coefficient (11.8 3 1026 _C vs 11.4 3 1026 _C) consequently, the mechanical properties of Ti-6Al-4Vare similar to those of Ni-Cr and Co-Cr alloys.

Titanium alloys are commonly used as an implant material in the human body.Ti-6Al-4V alloy is the most common used due to its special properties. Topography and surface chemistry have a profound effect on the way in which the human cells interact with an implant. In addition, it has been shown that surface topography can influence surface behaviour and growth. The corrosion of the implant material is governed by its surface characteristics. Hence, the surface modification techniques has attracted many researches and several studies was established and reported.

Titanium alloys has been successfully polished using various method. According to Chou *et al*. (2010) several techniques can be used to clean and smoothen the titanium substrate surface, namely chemical, mechanical and thermal polishing. Chemical polishing requires HF-containing acid solutions and it does not provide as smooth a surface as the other methods; mechanical polishing can provide a very smooth and flat surface topography, but it is work-intensive and time consuming, furthermore, polishing media can become embedded in the surface. Thermal polishing requires heat-treatment in a high vacuum and specimens must be very clean to start with.

Compared to the other polishing processes, electro polishing is an effective method to clean, smoothen and polish, the titanium surface. It removes impurities from the metal surface.

Electrochemical polishing has shown a better surface finish as reported by many researchers. Guilherme *et al* (2005) they reported that for cast titanium frameworks the electrochemical polishing is more effective than the manufacturer's polishing instructions with abrasives and rotary instruments. Also, Birch *et al*. (2012), they conducted electrochemical polishing of titanium alloy as an implant material (Ti-6Al-4V) using H_2SO_4/ methanol. The surface chemistry, however was dependent on the applied electrochemical potential. It was found that, the chemical composition of the surface was modified during the process at high potential (9.0V) and a pure TiO_2 of at least 10 nm was created on the top of the alloy surface. Furthermore, Pyka et al (2012), were able to make surface modifications of 3D titanium alloy-based open porous structure, in which a combination of chemical etching and electrochemical polishing using Hydro Fluoride-based solution was applied. This process achieved significant and controllable roughness of additive manufactured 3D titanium alloy open porous structures.

As it was shown in the previous studies that using acidic solutions for the electrochemical polishing process is required applying a high voltage current about 10-30V. In addition, such a process has many hazards related problems because the acids used are potentially explosive and the disposal of the concentrated acids is very costly. However, many researches shows that using ionic liquids for electrochemical polishing process provides better surface finish with more economical and safer method. Ionic liquids are salts typically composed of organic captions and organic or inorganic anions, which exist as liquids at temperatures below 100°C, often at room temperature. They are non-volatile, non-flammable, and present high chemical and thermal stabilities.

As Abbott *el al*. (2012) used anodic electrolytic etching with a novel choline chloride based ionic liquid to remove a surface oxide scale from single crystal aerospace casting on nickel based super-alloy. The removal of scale from cast component provides critical quality checks and assessment and it helps to detect other defects in the single crystal castings. It is shown that the oxide scale and residual casting mould shell can be effectively removed using ionic liquids with low toxic, environment friendly and low cost method.

Electrochemical polishing using ionic liquids is an effective way on other metals than titanium. Abdel-Fattah *el al*. (2009) mentioned that electrochemical polishing of aluminium and copper can be conducted in acid-free ionic liquid electrolyte prepared from ammonium slat. Based on the result of AFM, surface morphology, it has been concluded that using ionic liquid produce more smoother surface finish than standard acidic solutions for copper and aluminium. In addition, Abdel-Fattah also reported as successful electro polished stainless steel (AISI 316L) using ionic liquid medium based Vitamin B4 (NB4) and resulting in the nano scale level. Energy dispersive X-ray and scanning electron microscopy revealed a smoother surface than using the standard methods. Moreover, Wixtrom *et al* (2012) reported an alternative method for electrochemical polishing of Niobium that used on superconducting radio frequency cavities using a novel ionic liquid contains choline chloride. The final surface roughness of the NB was found to be comparable to that of the acid-polishing method as it was measured by atomic force microscopy.

Uda *et al* (2011) investigated the electrochemical dissolution of titanium in TMHA-Tf2N ionic liquid. It has been observed that a shiny metallic silver surface appears with high grade of surface roughness as indicated by fluorescence X-ray. It has been reported that a potential between -0.95 V to +1.6 V was necessary to successfully dissolve the titanium.

Finally, based on Tiley *et al* (2010) a low-stress automated polishing device was developed for preparing titanium and nickel alloys for SEM with both mechanical and electrochemical polishing. The samples were subjected to varying potential cycles and polishing times. The result indicated that applied cycles potentials removed material faster than typical removal techniques. It also showed that $\pm$ 0.8V for 201s provides the highest removal rate for Ti-6A-4V (6.25 nm/min) and by processing the samples for longer time it improved the image quality of the SEM.As the researches indicated that electrochemical polishing of titanium alloy using ionic liquids is a good alternative to achieve nano finishing and it needed to more investigated.

CHAPTER 3

METHODOLOGY

3 Methodology

3.1 Project activities

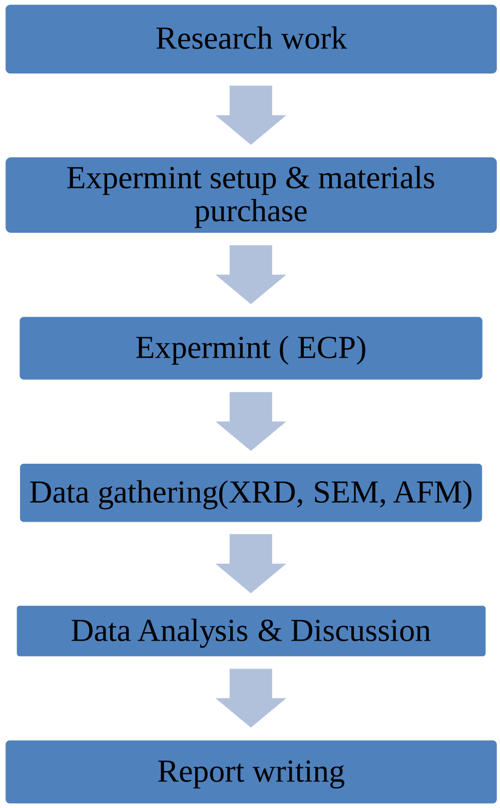


Figure 1: Project methodology flow chart

3.2 1-ethyl-3-methylimidazolium acetate

The ionic liquid used for the electrochemical polishing process for titanium alloys was 1-ethyl-3-methylimidazolium acetate which have chemical structure as shown in Figure 2. As it was proven that it has a wide electrochemical window of about (-2.07-2.12) V at 0.5 mA (Hayyan *et al*., 2012). Additionally, also shows useful enzyme7 and environmentally friendly characteristics and stable to be used.

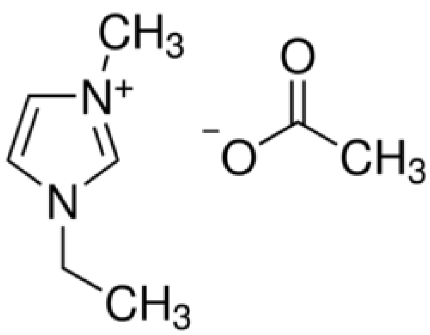


Figure 2 Ionic liquid chemical structure

3.3 Experiment Methodology:

The experiment was conducted in several stages as shown in Figure 3 below.

1. Modify the existing polishing machine.
2. Prepare the sample (cut into 10x10 mm).
3. Apply mechanical polishing & measure the surface roughness.
4. Conduct electrochemical polishing to the samples.
5. Measure the surface roughness and continue the experiment
6. Measurements using SEM, AFM
7. Compare and suggest the best parameters for this process.

Figure 3: Experiment methodology process

Electro chemical polishing consist of two process that done at the same time; polishing and chemical dissolution. As it is shown in the diagram in figure 4 the work piece (Ti-6Al- 4V) will be fed from the top against a rotating polishing dick. The disk is connected to a negative potential while the working piece is attached to an anode then a DC potential is applied. The ionic liquid will be dropped over the polishing dick. However, a gap of 200-500 µm will be maintained between the polishing dick and the work piece.

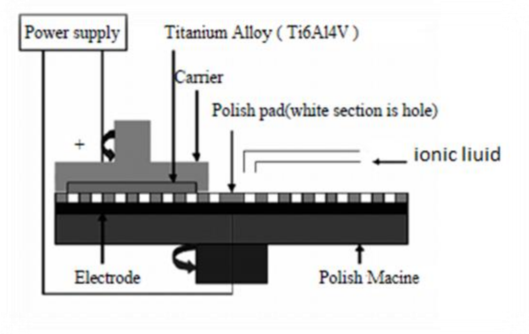


Figure 4 ECP modified machine

The standard polishing machines will be modified to be an electrochemical mechanical polishing machine. A work piece holder will be attached to the polishing machine. In addition, the polishing disk is isolated and a piece of polishing cloth that was placed over it. It's also connected to a negative terminal and positive connected to the working piece as shown in Figure 4.

The work pieces were cut and sized into square shapes with specific dimensions (10 x10 x 4 mm). After that, they were attached to molded plastic which insure correct mounting on the quill. The work pieces then were mechanically grounded using

1200/600 p abrasive papers (SiC) for about 6-7 minutes to eliminate surface irregularities. Then the mean roughness factor R_a of the samples was measured with surface roughness scaler (profilograph) at certain sittings (6 mm measurement length, and 0. 5 mm/s measurement speed).Then, the samples were polished electrochemically. When, the potential applied to the samples, electrochemical dissolution occurred (redox reaction) and the ionic liquid acted as electrolyte in the process. At the same time, a low stress mechanical polishing were applied to the sample as the disk rotated for a certain time. Then, the experiment was repeated using various parameter settings for eight samples as shown in Table1 (four different voltages with two speeds for each).

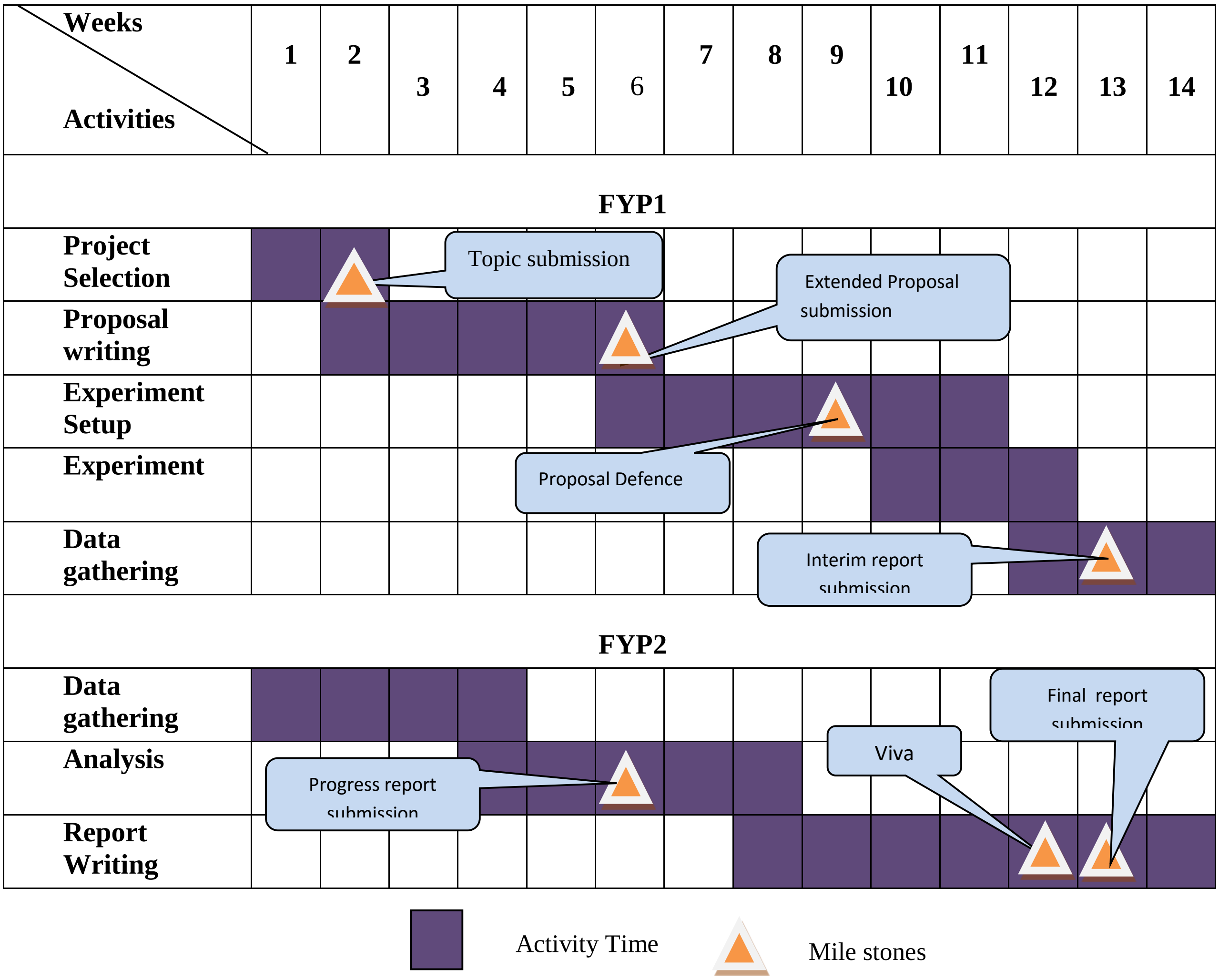
Table 1 Experiment input parameters

Sample	Voltage(V)	Speed (rpm)
1	15	50
2	15	200
3	30	50
4	30	200
5	45	50
6	45	200
7	60	50
8	60	200

The work pieces were electrochemically polished for certain minutes with the same sitting, then the mean roughness factor was measured with profilograph. The experiments continued until the samples reached targeted surface smoothness. Finally, the morphology of the sample surfaces was investigated with Atomic Force Microscopy Nano Scope (AFM) and high resolution Scanning Electron Microscopy (SEM) at 15Vk and magnification of 3000x. The chemical composition of the selected samples were determined with EDX (Oxford Instrument attached to SEM). Besides that, AFM provides a clear topographical image of the targeted surface with a qualitative visual analysis before and after the electro chemical polishing process. SEM also will be used to measure the scale formation of the work piece after the polishing process as specified by the scope of the study.

3.4 Gantt chart and key milestone

Table 2 Gantt chart and key milestone FYP1 &FYP2



CHAPTER 4

RESULT AND DISCUSSION

4 Result and discussion

4.1 Surface Roughness Indexes

The objective of this study was to optimize the input parameters of the electrochemical polishing for Ti-6Al-4V with ionic liquid to achieve a smooth and uniform surface at the nano scale. To optimize the parameters the ECP technique eight samples were subjected to the experiment at four input voltages (15-60V) at either high or low rotation speed for each. The titanium samples were mechanically grounded and their surface roughness indexes were measured using profilograph at minute zero on Table 3.The result obtained was collected and tabulated as shown in Table 3&4.

Table 3: Average surface roughness (μ m) for different input parameters

Sample	Voltage (V)	Rotation Speed (rpm)	Polishing Time (minutes)					
			0	3	4	5	6	8
			R_a (μm)					
1	15	50	0.986	0.722	0.558	0.540	0.535	0.525
2	15	200	0.999	0.884	0.872	0.866	0.776	0.611
3	30	50	0.403	0.380	0.368	0.354	0.327	0.323
4	30	200	0.783	0.713	0.689	0.648	0.542	0.520
5	45	50	1.038	0.793	0.768	0.742	0.619	0.606
6	45	200	0.752	0.684	0.649	0.566	0.561	0.547
7	60	50	0.261	0.258	0.235	0.213	0.195	0.164
8	60	200	0.690	0.684	0.612	0.524	0.549	0.719

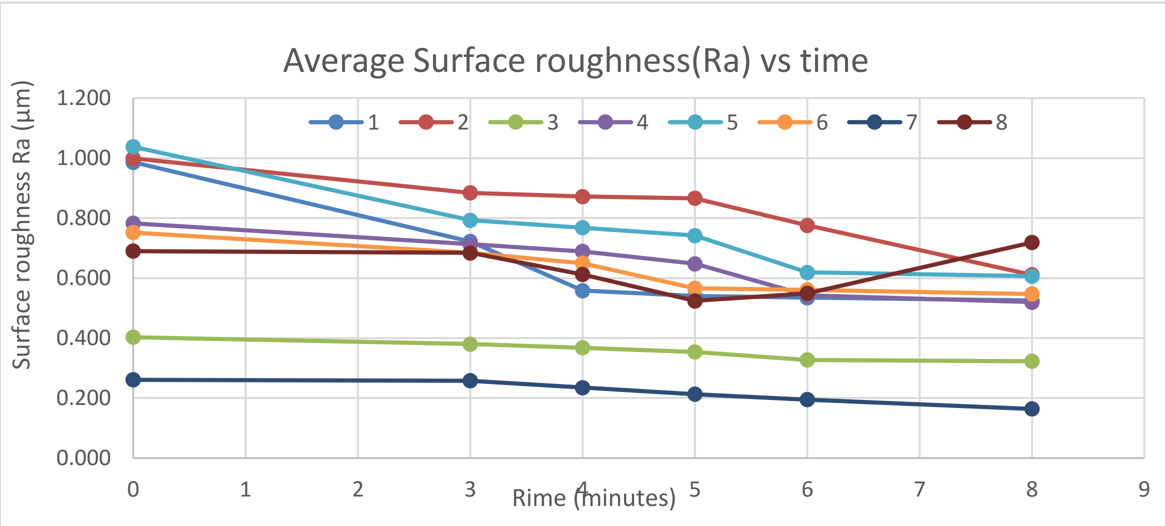


Figure 5: Average Surface Roughness (Ra) for different input parameters vs time(minutes)

Figure 5 shows the average surface roughness factor in (µm) which obtained from the profilograph readings verse time. In general, the figure indicates that the surface roughness index is decreasing gradually with the time of the electrochemical polishing process. As the result in Figure 5 indicates that from 6-8 minutes the change in the surface roughness Ra is significantly small 3%-2% accept for sample 8. So, the optimum time for the process is from 6-7 minutes. The results in Figure 5 show that the voltage has a great influence on the material removal rate of the samples. As the voltage increases the material removal rate increases. The high material removal rate results from the high reaction rate of the anodic reaction. However, high voltage results a bad surface quality as sample 8. This is due to the passivation film on the surface was not able to protect the titanium alloy from anodic oxidation. In addition, under constant applied voltages, samples 1, 2, 5 and 4 were the highest change in the surface roughness indexes comparing to the initial state which indicates lower voltages are more recommended for the process.

Table 4: Total Surface Roughness Rt (µm) for different input parameters

Sample	Voltage (V)	Rotational speed (rpm)	Polishing Time (minutes)					
			0	3	4	5	6	8
			R_t (µm)					
1	15	50	4.807	4.095	2.973	3.112	3.667	3.504
2	15	200	5.060	4.833	4.857	4.770	4.567	3.515
3	30	50	3.993	3.932	2.387	2.298	2.841	2.805
4	30	200	3.593	3.087	2.922	2.825	2.744	2.65
5	45	50	3.585	4.042	3.302	3.869	3.313	2.859
6	45	200	3.179	3.787	3.446	3.968	2.764	2.267
7	60	50	2.246	1.383	1.532	1.573	1.356	2.636
8	60	200	3.383	3.661	3.425	3.158	2.980	7.364

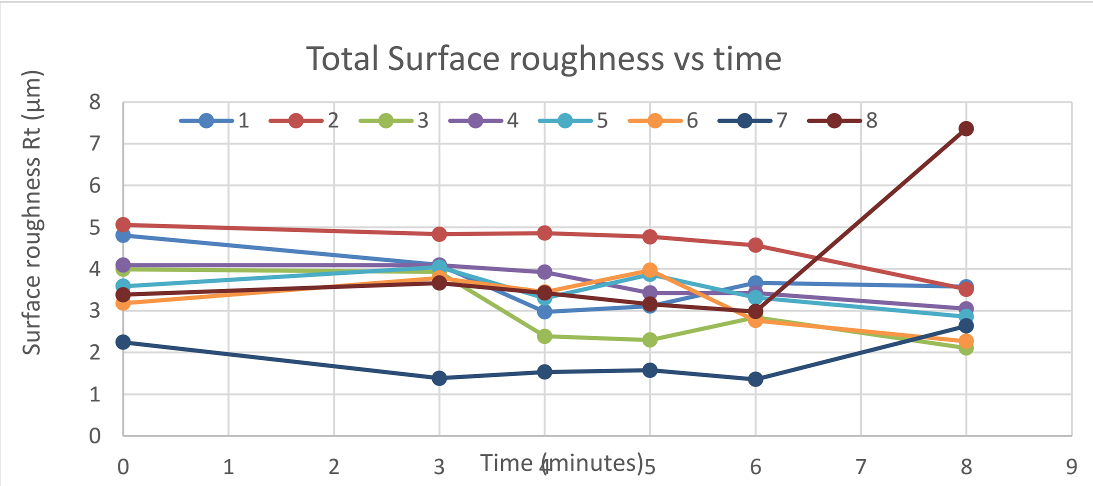


Figure 6: Total Surface Roughness for different input parameters vs time (minutes)

Table 4 represents the total surface roughness factors (peak to valley) values vs polishing time for the samples at their respective parameters. Figure 6 shows that the total surface roughness indexes are gradually decreasing with time accept for the last two samples (7&8) which at high voltage 60 V which could occurs due to the passivation film on the surface was not able to protect the titanium alloy from anodic oxidation so after a few minutes the surface got rougher. It can be shown also, at 30V and 45V the total surface roughness is less at higher rotational speed. Generally as the rotation increases it leads to lower material removal rate. In fact, the amount of the ionic liquid on the surface decreases when the rotation speed is higher and it result smoother surface at average voltages. Based on Figure 6, the result shows inconsistency for some samples between 4 - 6 minutes due to inconsistency on the measuring area. The data confirmed that applying electrochemical polishing of titanium with ionic liquids provided smoother surfaces compared to pure mechanical polishing and similar to ECP with acidic solutions. Furthermore, under constant applied voltages, samples 4, 6, & 7 were the smallest total surface roughness indexes values. In addition, these samples were selected for SEM and AFM analysis due to resource limitations.

4.2 Scanning Electron Microscopy (SEM) and EDX Analysis

4.2.1 SEM analysis

Scanning electron microscopy is one of the famous tools that is used to determine the microstructure of materials and study the surface grain formation. Figure 7 presents SEM of the polished samples (1, 4, 6&7) of Ti-6Al-4V at 3000x magnification scale, of roughness indexes Ra 0.502, 0.520, 0.547 and 0.164 µm as measured with profilograph. That Ra values measured over relatively large macroscopic area of 3 mm length includes scratches formed on the surface before and during the polishing. At a small magnification scale the surface of all samples appears to be very smooth and uniform after the electrochemical polishing. However, in this analysis the images were at large scale 3000x which gives clear identification of the surface grain formation. Figure 7a shows a rough surface with uniform grains (15V, 50rpm). On the other hand, figures 7b and 7c shows smoother surface and less uniform grains at 7b (sample 4). In addition, figure 7d represents sample 7 where the process at higher voltage and low rotation speed, the surface appears to be smooth and less uniform with black grains where more titanium is concentrated and less aluminium and vanadium. As a summary, SEM shows sample 6 input parameters is more recommended (45V, 200 rpm).

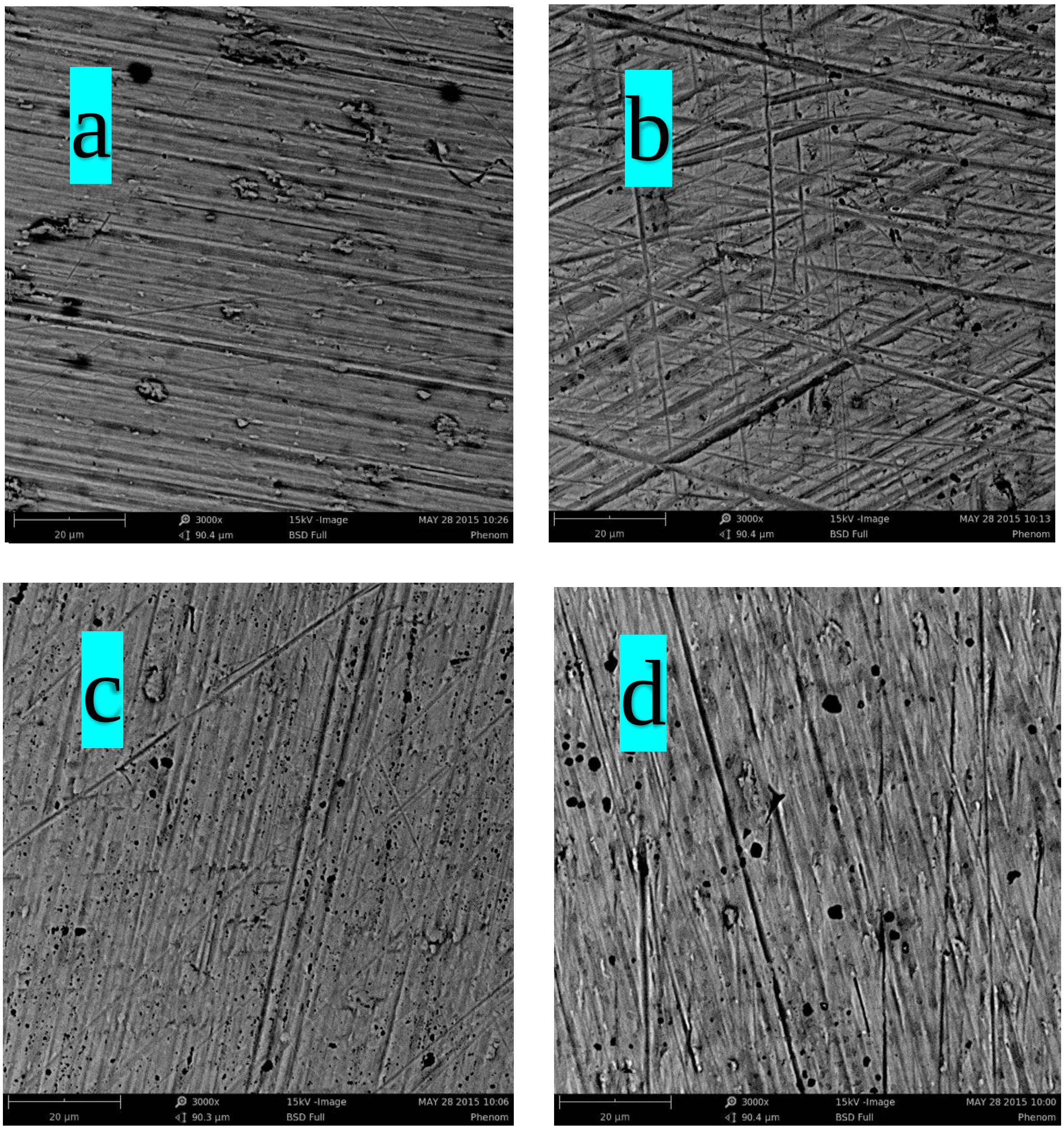


Figure 7 : SEM images of Ti-6Al-4V alloy after electrochemical polishing, sample1 (a), sample 4(b), Sample 6(c), sample 7(d)

4.2.2 EDX analysis

EDX stands for backscattered electron images in the SEM display compositional contrast that results from different atomic number elements and their distribution. Energy Dispersive X-ray Spectroscopy (EDX) allows one to identify what those particular elements are and their relative proportions. Figure 8 shows SEM image of sample 2 after electrochemical polishing with ionic liquid and two spots spectrum analysis. Spot1 was selected in the grey region of the surface while spot 2 was selected as a part of the black region. As table 5 and 6 shows that five elements were presented in the surface (Ti, Al, V, O, and C) and indicates their composition of each spot. Spot

1 contains more Aluminium and Vanadium and less carbon compared to spot 2. Furthermore, the presence of carbon as one of the element on the surface indicates that the alloy is covered with a thin layer mainly composed of titanium, aluminium and vanadium oxides which were formed due to the chemical reaction of the ionic liquid with the sample.

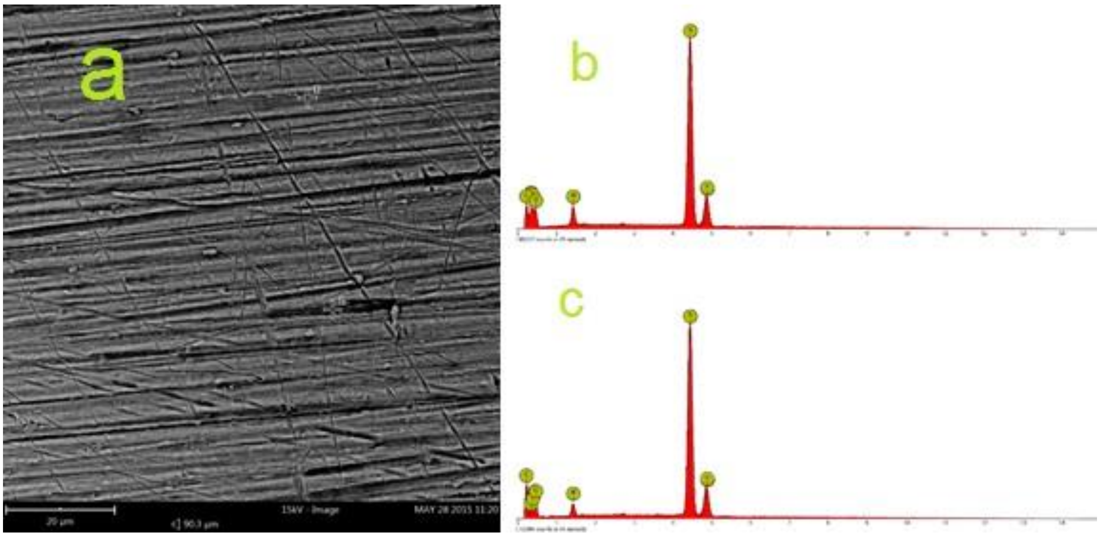


Figure 8 SEM image (a), EDX spectrums spot 1(b), EDX spectrums spot 2(c), of sample 2 after electrochemical polishing

Table 5: EDX spectrum elements composition at spot 1

Element Number	**Element Symbol**	**Element Name**	**Weight Concentration**	**Error**
22	Ti	Titanium	57.6	0.3
23	V	Vanadium	1.8	0.2
13	Al	Aluminium	2.9	0.1
6	C	Carbon	13.1	0.5
8	O	Oxygen	24.7	0.8

Table 6: EDX spectrum elements composition at spot 2

Element Number	**Element Symbol**	**Element Name**	**Weight Concentration**	**Error**
22	Ti	Titanium	57.6	0.4
23	V	Vanadium	1.0	0.3
6	C	Carbon	17.8	0.5
8	O	Oxygen	21.7	0.5
13	Al	Aluminium	1.8	0.1

4.3 Atomic Force Microscopy (AFM) analysis

Atomic force microscopy is a very high-resolution type of scanning probe microscopy which demonstrate resolution in the fraction of nanometre, it is also possible to measure a roughness of a sample surface at a high resolution. Figure 9 presents AFM 3d surface representation of the electrochemically polished samples (1, 4, 6&7) of the Titanium alloy. The Ra values measured using profilograph over relatively large macroscopic area of 3 mm length which includes characteristic scratches formed before and during the polishing. However, The Ra indexes measured for small microscopic, scratch free areas that was calculated based on the AFM results as in Figure 9 is much lower. For image of 2.5X2.5 μm area shown in Figure 9 ECP1 it is equal to 39 nm, which was found to be representative at microscopic scale. The value of the macroscopic roughness indexes Ra, change slightly with the increase of the voltage. With the increase of the voltage the electric charge increases the mean roughness indexes decreases as in Figure 9 ECP4. At 45V the mean surface index increases to 49 µm. As the previous results indicates at higher voltage the mean surface index increases with time. However, in figure9 ECP7 it has the lowest value which related to the initial value at zero time was relatively small.

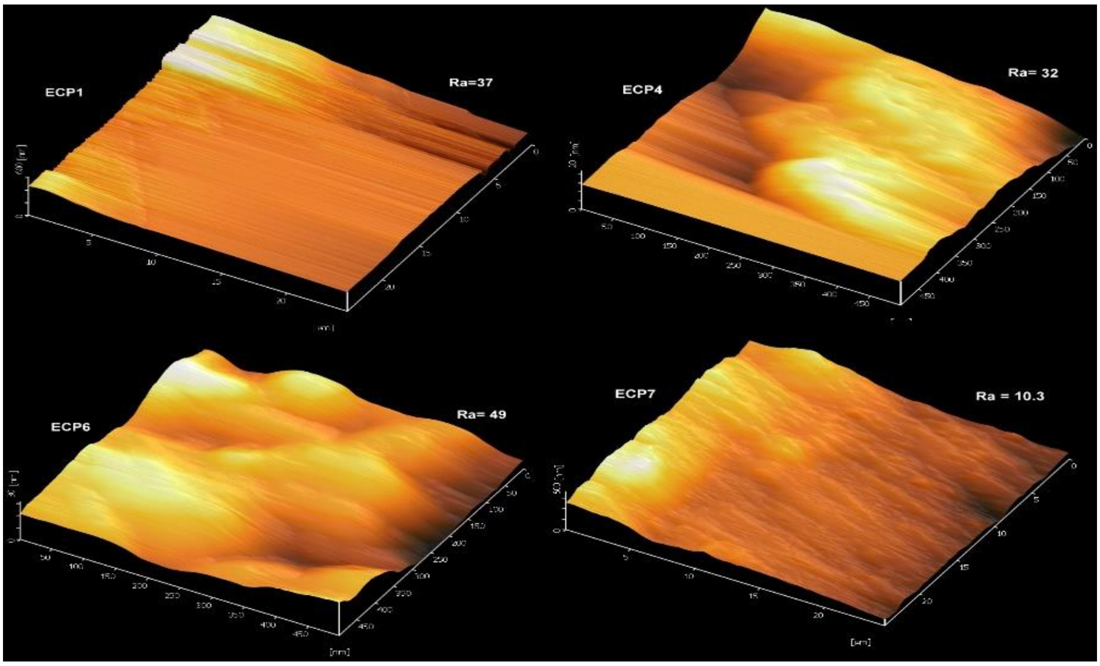


Figure 9: Atomic Force Microscopy 3D surface representation for sample 1,4,6 &7

CHAPTER 5
CONCLUSION AND RECOMMENDATIONS

5 Conclusion and recommendations

5.1 Conclusion

Electrochemical polishing of titanium alloys using ionic liquids is an alternative to the standard methods results a nano scaled surface roughness. The process is very cost-effective and environment friendly that can replace the existing process. The data confirmed that applying ECP of titanium with ionic liquids provided smoother surfaces compared to pure mechanical polishing and similar to ECP with acidic solutions. As the surface roughens were investigated at different input parameters. It can be concluded that, the surface roughness indexes gradually decreases with the time of the process and it's recommended to be in the range of 6-7 minutes. In addition, High voltages is not recommended for the process. Based on SEM and AFM analysis high rotation speed (150-200 rpm) and average voltages (35- 45 V) are the best input parameters for the process which result smooth surface with uniform scale formation. Finally, more investigation is required in this topic since the study successfully reached its objectives and was able to narrow down the parameters into a small range.

5.2 Recommendations

My recommendations for the future work that is related to the same topic. First, it is preferable all samples should have similar surface roughness index, that before conducting the electrochemical polishing. This could leads to an accurate and easy analysis. In addition, it would be better to conduct X-Ray Diffraction (XRD) analysis which provide the researcher with the ability to determine the compound formed on the surface of the alloy after ECP.

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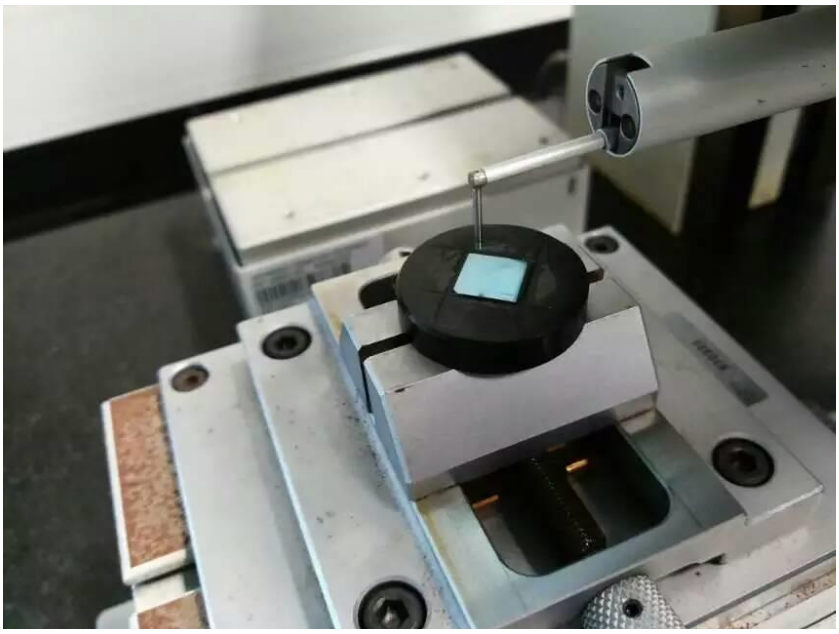
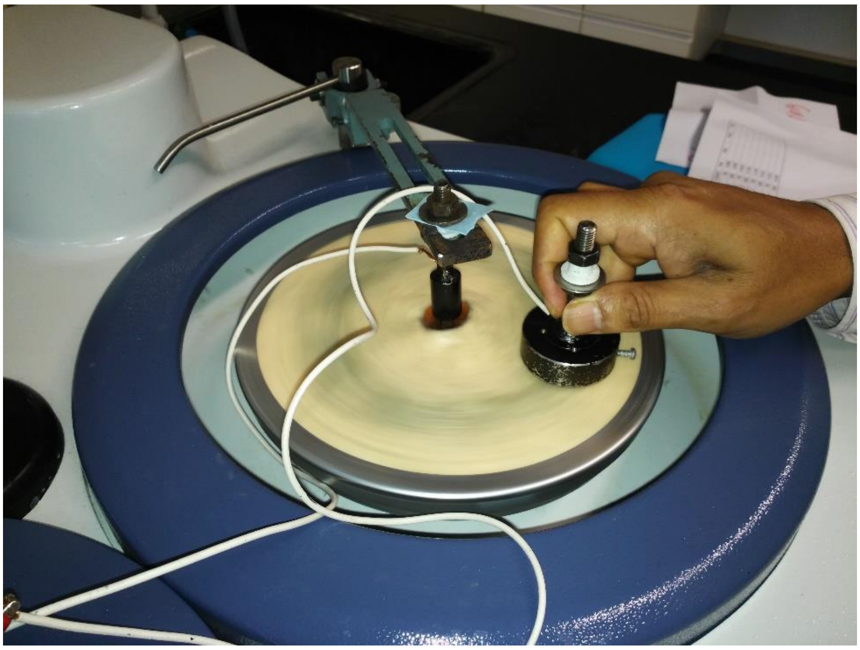
Appendix



Appendix a: Profilograph during sample measurement



Appendix b: Electrochemical polishing of titanium alloys samples