Electrochemical Polishing of Titanium Alloys with Ionic Liquids

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Keywords- *electrochemical polishing; SEM; AFM; profilograph*

Abstract: The purpose of this study is to propose the best input parameters for electrochemical polishing of Ti-6Al-4V with 1-ethyl-3-methylimidazolium acetate (ionic liquid) with an experimental method. The effect of voltage, rotation speed and polishing time on the surface roughness were investigated. A Polishing time of 6-7 minutes is recommended for good surface finish. In addition, high voltages are not recommended for the process. The surface morphology was measured using AFM and SEM. SEM analysis show that the input parameter settings of 45V and 200 rpm are recommended for electrochemical polishing.

Introduction

In the recent years, the demand of new products that required complicated shapes and tighter tolerance has increased rapidly. Titanium alloys are such material those 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. Ti-6Al-4V alloy is the most commonly used implant material in the human. These implants are Electrochemical Polishing (ECP) to get the desired surface roughness. Topography and surface chemistry have a profound effect on the way in which the human cells interact with an implant. It has been seen 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 have attracted many researches and several studies was established and reported.

ECP of Ti-6Al-4V alloy is performed with perchloric acid applying high voltage. Such process poses many hazards because the acids used are potentially explosive. Moreover, the disposal of the concentrated acids is very costly and time consuming. However, there are some problems in using ECP process as a result of a strong passive film on the surface of the titanium. In the recent years, ionic liquids (liquid salts in room temperature) has shown a promising result in ECP process with a safer and more economical approach. They are non-volatile, non-flammable, and present high chemical and thermal stabilities.

Abbott, *et. al.* [1] 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. According to Chou, *et. al.* [2] there are several techniques to clean and smoothening the titanium substrate, namely chemical, mechanical and thermal polishing. Among these methods, electro polishing was the most effective method to clean, smoothen and polish the titanium surface. Birch, *et. al.* [3] conducted ECP of titanium alloy as an implant material (Ti-6Al-4V) using H_2SO_4/ methanol. It was reported 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. Uda, *et. al.* [4] investigated the electrochemical dissolution of titanium in TMHA-Tf_2N ionic liquid. They observed that a shiny metallic silver surface appears with high grade of surface roughness as indicated by fluorescence X-ray. As reported them, a potential between -0.95 V to +1.6 V was necessary to successfully dissolve the titanium. Tiley, *et. al.* [5] developed a low-stress automated polishing device for preparing titanium and nickel alloys with both mechanical and electrochemical polishing. The result indicated that applied cycles potentials removed material faster than typical removal techniques.

Ionic liquids have potential to be used as electrolyte in polishing process. An extensive research work is still required to determine the suitable machining input parameters of Titanium ECP. The objective of this work is to study the effect of machining input parameters, voltage, rotation speed, and polishing time, on the surface roughness on ECP of Ti-6Al-4V alloy using 1-ethyl-3-methylimidazolium acetate (ionic liquid) as electrolyte.

Experiment Methodology

Electro chemical polishing consist of two process that done at the same time; polishing and chemical dissolution. The work piece (Ti-6Al-4V) was fed from the top against a rotating polishing disk which was connected to a negative potential, as shown in Fig. 1. The working piece was attached to an anode. The ionic liquid was dropped over the polishing disk which was covered with a polishing pad. A gap of 200-500 μm was maintained between the polishing disk and the work piece. A DC potential was applied between anode and cathode and the ionic liquid acted as electrolyte that lead to electrochemical dissolution (redox reaction). At the same time, as the disk rotated, low stress mechanical polishing was done for a specific time.

The work pieces were cut to square shape with specific dimension (10 x10 x 4 mm) and mounted on plastic mold and then samples were polished electrochemically. The experiment was conducted with four levels of voltage and two levels of speed as shown in Table 1 using Taguchi design. The work pieces were electrochemically polished for eight minutes and the Centre Line Average (CLA) roughness, *Ra* on each of the sample was measured at interval of time. A surface roughness scaler (profilograph) was used with settings evaluation length = 0.6 mm, and measurement speed = 0.5 mm/s, filter = Gaussian. The measurements are presented in Table 1.

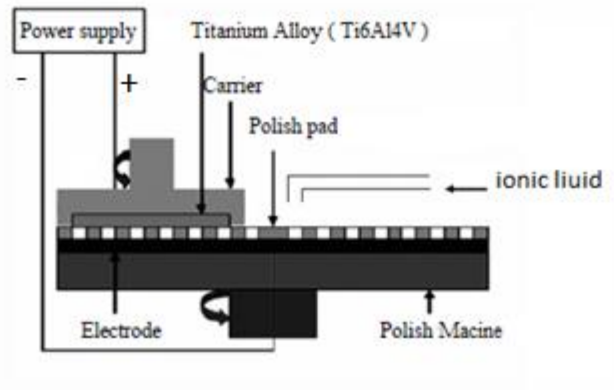


Fig. 1 ECP machine

Finally, the morphology of the sample surfaces were investigated with high resolution Scanning Electron Microscopy (SEM) at 15Vk and magnification of 3000x. Besides that, Atomic Force Microscopy (AFM) was used to study topographical images of the targeted surface with a qualitative visual analysis before and after the electrochemical polishing process.

RESULTS

A. Surface Roughness

The objective of this study was to optimize the input parameters of the electrochemical polishing of Ti-6Al-4V with ionic liquid to achieve a smooth and uniform surface.

Table 1: R_a (µm) for different input parameters

Run	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

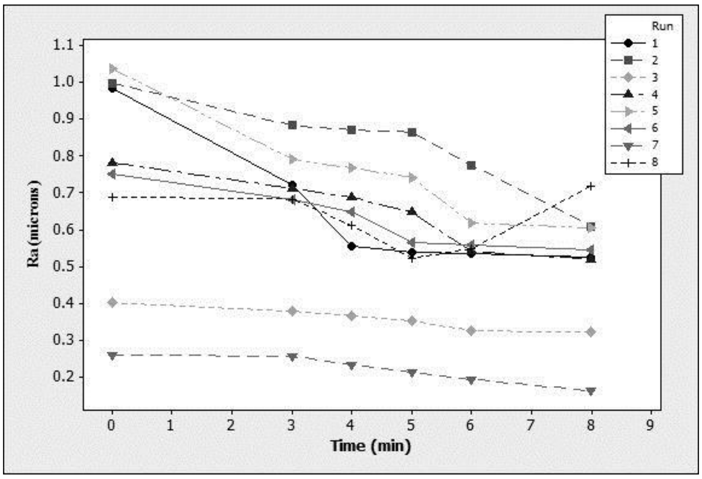


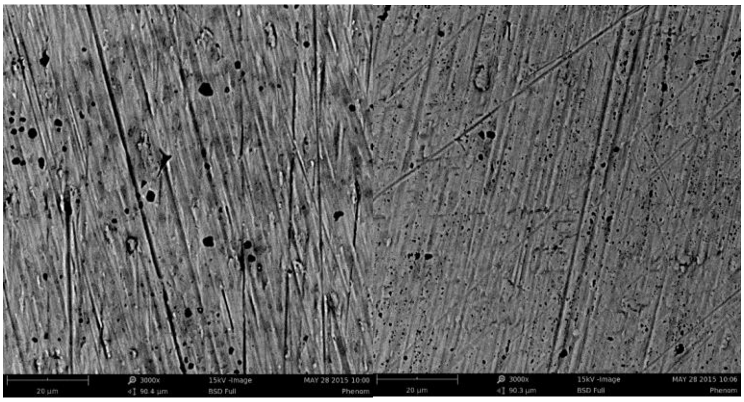
Fig. 2: *Ra* vrs time for various run

Fig. 2 shows the variation of *Ra* values with polishing time. In general, the figure indicates that the surface roughness index decreases gradually with the time of polishing. From results in Table 1, it indicates that between 6-8 minutes the improvement in *Ra* value is significantly small 3-2%, except for run 8. Hence, it can be concluded that the optimum polishing time will be between 6-7 minutes.

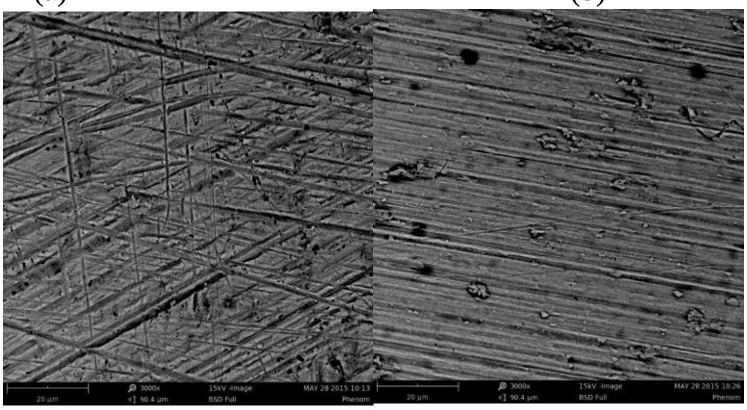
The results in Fig. 2 show that the voltage has a great influence on the material removal rate of the samples. As the voltage increases the material removal rate increases. The high material removal rate results from the high reaction rate of the anodic reaction. However, high voltage results a bad surface quality as run 8. This is maybe occurs because the passivation film on the surface was not able to protect the titanium alloy from anodic oxidation. The samples from runs 1, 4 6, and 7 were selected for SEM and AFM analysis due to resource limitations.

B. Scanning Electron Microscopy (SEM) Analysis:

SEM was used to determine the microstructure of materials and study the surface grain formation. Fig. 3 presents SEM micrographs at 3000x magnification of the runs 1, 4, 6 and 7, having *Ra* values 0.502, 0.520, 0.547 and 0.164 µm, respectively. The surface grains are clearly indicated on the images that include scratches those were formed on the surface before and during the polishing. Fig. 3a shows a rough surface with uniform grains (15V, 50rpm). On the other hand, Fig. 3b and 3c show smoother surface and less uniform grains at Fig. 3b (run 4). Fig. 3d represents run 7 where polishing was done 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 aluminum and vanadium. As a summary, SEM shows run 6 input parameters are recommended (45V, 200 rpm).



(a) (b)



(c) (d)

Fig. 3: SEM images of Ti-6Al-4V alloy after electrochemical polishing run 1 (a), run 4(b), Run 6(c), run 7(d)

C. Atomic Force Microscopy (AFM) analysis

AFM is a very high-resolution type of scanning probe microscopy which has resolution in nano scale. Hence, it is possible to measure a roughness of surface at a high resolution. Fig. 4 presents AFM 3D surface of an area of 500 x 500 nm scanned after the electrochemically polished for runs 1, 4, 6 and 7 of the Titanium alloy. The *Ra* values obtained from AFM measurement were 37, 32, 49, and 10.3 nm for the runs 1, 4, 6 and 7, respectively.

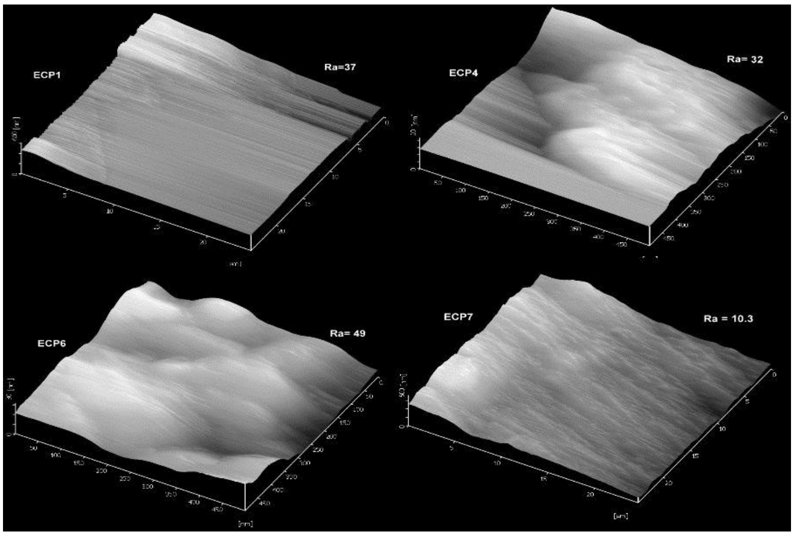


Fig. 4: Atomic Force Microscopy 3D surface representation for run 1, 4, 6 and 7

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 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. The surface roughness gradually decreases with the time of the process and it is recommended that polishing time should be 6-7 minutes. In addition, high voltages are not recommended for the process. From the SEM and AFM analysis, rotation speed of 200 rpm) and voltages 45 V are the best input parameters for the process which result smooth surface.

Acknowledgment

This study was carried out as an URIF project (00153AA-B49) funded by Universiti Teknologi PETRONAS, Malaysia. The authors would like to thank the funding agency.

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