Surface topography and biocompatibility of Ti-6Al-4V after Wire Electro-Discharge Machining (WEDM)

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ABSTRACT

Ti-6Al-4V is the difficult-to-machine material commonly used for biomedical applications. The surface characteristics of the Ti-6Al-4V plays an important role in the biocompatibility and the industries. The machining of titanium is complex due to its characteristics and have limitations such as variations of chip thickness, high heat stress, high-pressure loads, and residual stress during the conventional machining process. The research presents the experimental of wire electro-discharge machining (WEDM) on Ti-6Al-4V. The research aims to characterize the surface topography of Ti-6Al-4V after WEDM process and the biocompatibility of the machined surface. The machined surface analyzed for the surface topography, elementary analysis and cell viability to observe the biocompatibility of WEDM process based on the variation of peak current, pulse-off time and wire tension. The white layer thickness of high input parameters produced a thicker layer on the machined surface. As the effect of the machining process, there is the existence of Ti, Cu, V, Zn, C, O, and Al on Ti-6Al-4V surface. The results show that the WEDM process on Ti-6Al-4V was biocompatible for medical implants as L929 cells positively active in the culture medium. The sets of input parameters for good surface characterization are 6A peak current, 2µs pulse-off time and 8N wire tension.

Keywords: *Ti-6Al-4V; Surface Topography; WEDM; Biocompatibility.*

Introduction

Due to excessively used in medical industries, titanium undergoing surface chemistry and topography so that it can contribute to the implantation

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devices [1]. N. Vu et. al mentioned that in recent years, the biomedical field is shown rapid growth in implantations [2]. The materials that can have good integration into the bone and other tissues is titanium due to good osseointegration between them. The human body accepted titanium as the characteristics of titanium attractive for medical and dental application. Titanium is high corrosive resistance, biocompatible material, non-magnetic property, low specific gravity and high specific strength [3].

The machining of titanium is complex and different compare to other metals such as aluminum, steel and more. Titanium becomes difficult-to-machine materials because of its characteristics that are low thermal conductivity during machining, capacity to maintain strength at high temperature and severe work hardening ability which lead to high machining forces. Besides, the ability to change the phase of titanium alloys makes the deformation process becomes more complex [4]. Furthermore, most of the harder tools in the market not suitable to machine titanium because of chemical affinity that leads to chemical wear in the cutting tool and chips weld easily on the tool to form built-up edge (BUE).

There are several problems that titanium alloy experienced in machining; variations of chip thickness, high heat stress, high-pressure loads, springback and residual stress [5]. Different material removal process produced different types of the cyclic chip due to the different formation at the shear zone and can be classified as; discontinuous chips, continuous chips, continuous chips with built-up edge and segmented chips. Segmented chips are also known as saw-tooth or serrated chips [6]. Machining of the titanium mostly used cutting tool that is from cemented carbides, nitrides, oxides, borides, diamond, cubic boron nitride, etc. and contributes to high tool wear. The material removal process increased the strain rate and decreased thermal softening which increase the tendency for saw-tooth chip formation [7].

The 3D-microstructures of new implant generations are hard to machine by the conventional machining. Hence, the alternative manufacturing that has the capabilities is wire electro-discharge machining process [8]. Wire electro-discharge machining (WEDM) is a non-conventional machining process that removed materials by heat generated between the workpiece and wire electrode. There is short electrical discharge between the workpiece and wire electrode in the discharge gap. The molten material melted and removed by the deionized water. The failed removed material stays on the machined surface creating craters.

Theoretically, the form of craters is spherical but can exist in different shapes because of many factors such as the electrodes material, the pulse duration, the discharge frequency and the discharge current intensity [9]. The WEDM also applicable manufacturing process that allows machine implants products because no negative surface conditions occur after the machining and can be well sterilized [10].

The combination of electrical parameters produces different process performance on the machined surface. Some important input process parameters influence WEDM processes such as pulse-on time, pulse-off time, gap voltage, flushing pressure, wire-speed, wire tension, peak current, and taper angle [11]–[13]. Investigation of WEDM done by A. Kumar et. al. [14], was commercially pure titanium grade 2 on different input parameters; pulse-on time, pulse-off time, peak current, spark gap voltage, wire feed, and wire tension. Machined workpiece observed on the cutting rate, gap current, and surface roughness. Moreover, Ramanuj Kumar et al. [15] investigated the analysis of material removal rate and surface roughness in machining of titanium alloy using the EDM process. Other than that, the process performance of WEDM can be observed based on the cutting rate, wire rupture, dimensional deviation, white layer thickness and kerf width [16].

Biocompatibility refers to the ability of the biomaterial to have good interaction between medical devices, tissues, and physiological systems without eliciting any undesirable local or systemic effects in the recipient [10]. Besides, the biocompatibility of the devices generating the appropriate beneficial cellular and tissue response in a specific situation. Biocompatibility is a part of overall safety assessment of the devices and depending on several factors, which are the chemical and physical nature of its material, the types of tissue that exposed to the device and the duration of the exposed.

In this research, the surface topography of wire electro-discharge machining of Ti-6Al-4V on the biocompatibility is studied. The research began with the machining of the workpiece and followed by the characterization of the surface topography. The WEDM Ti-6Al-4V then was cultured with L929 cells in order to observe the cell viability of the medium.

Materials and Method

Wire Electro-Discharge Machining Process

Grade 5 titanium alloy (Ti-6Al-4V) used as the workpiece in this experimental work. The composition of the Ti-6Al-4V stated in Table 1. The Ti-6Al-4V cut by using WEDM Mitsubishi FX Series Machine. The workpiece was cut to 60mm x 30mm x 20mm for the surface characterization while 220mm x 39mm x 60mm for the biocompatibility testing. The machine used plain brass wire with a diameter of 0.05mm consists of 60% of Cu and 40% of Zn. Three different input parameters with minimum and maximum values used as in Table 2.

Table 1: The chemical composition of Ti-6Al-4V

Element	Ti	Al	V	Fe
Weight Percentage (%)	0.90	0.06	0.04	0.0025

Table 2: The input parameters of WEDM

Parameter	Test I	Test II	Test III
Peak current (A)	6.00	8.00	10.00
Pulse-off time (μs)	2.00	3.00	4.00
Wire tension (N)	8.00	10.00	12.00

Surface Characterization

The samples prepared by cold mounting using the epoxy and its hardener. Following the preparation, the mounted Ti-6Al-4V has proceeded with the grinding and polishing process. The grinding process used silicon carbide sandpaper; 240 grit, 320 grit, 600 grit, 1200 grit. The polishing process used Twin Variable Speed Grinder-Polisher from 6 um, 3 um and 1 um of polycrystalline diamond.

The mounted Ti-6Al-4V then etched with Kroll's reagent (100ml of distilled water, 2 ml of HNO_3 and 2 ml of HF) for 3 minutes. The surface characterization observed using a Hitachi Scanning Electron Microscope (SEM) Model SU3500. The elementary analysis to identify the foreign materials and chemical composition conducted using EDX.

Cell Culture

The preparation of cell growth evaluated by placing the culture vessels in a laminar flow hood with culture medium components which have been pre-warmed to 37°C. The medium for L929 cells composed of 500 ml of the minimum essential medium Eagle (MEM 1X), 57 ml of bovine calf serum (BCS) iron supplemented, 5.7 ml L-Glutamine (200 mm), 5.7 ml non-essential amino acid (100X) and 5.7 ml antibiotic/antimycotic solution (100X). The medium was aspirate while L929 cells were washed with 1X PBS before trypsinization.

The disaggregate of cells done by addition of trypsin solution (1X). The culture vessels then incubate at 37°C for 5 minutes. The medium is added to stop trypsinization. The cells incubated in 37°C incubator with 5% CO_2 for 14 days before proceeding with determination number of cells. The trypsin added into the complete media and cell in the universal tube, centrifuged for five minutes in Eppendorf Centrifuged 5720. The 50μl cell was put into a universal tube with 50 μl of trypan blue stain, which then placed on a hemocytometer to observe the activation of cells.

Cell Viability

The cell viability was evaluated using Alamar Blue Assay which is known as cell viability indicator. The first treatment for the extraction of cell viability was by immersed Ti-6Al-4V in complete media for 24 hours at 37°C without agitation with the weight-volume ratio of 200 mg/ml. The negative control was represented by complete media with no material to ensure the activation of the cells during the treatment. The pure extract diluted with complete media to have a weight-volume ratio of 100, 50 and 20 mg/ml dilutions in each 24 well plate which contained a monolayer of L929 cells.

The L929 cells already seeded with $1x10^5$ cells/ml in the plate for 24 hours. The mixture was incubated in carbon dioxide incubator at 37°C/5% CO_2 for another 24 hours. After 24 hours, the cell viability was tested using Alamar Ble Assay with ration 1:10 and incubated for 4 hours at 37°C in CO_2 indicator. The stained culture was detected by absorbance at 570 nm using Universal Microplate Reader.

Results and Discussion

Surface Topography

The surface topography of the WEDM machined surface shows in Figure 1. From the SEM micrograph observation, there was a formation of the white layer on the machined surface. The white layer produced due to the deionized water failed to remove some of the molten materials during the machining process. The excess molten materials cooled rapidly and heat conduction takes place at the workpiece, triggered the excess materials to stay hard on the machined surface. Figure 1(a) shows the white layer thickness (WLT) for low parameters (Test I) while Figure 1(b) shows the white layer thickness (WLT) for high parameters (Test III). The WLT of Test III (12.7µm) higher than the WLT of Test I (9.72µm). As the peak current increase, larger pulse duration allows high temperature to penetrate deeper into the subsurface. This lead increment in production of molten material which resulting thicker white layer. The thickness of white layer increase with the discharge energy.

The thickness of the white layer affected by combinations of the input parameters during the machining process. The white layer surface appears with many globules of debris, craters, and cracks as shown in Figure 2. The peak current for Test III is 10A while Test I is 6A. Higher peak current producing larger pores, hence, more resolidified molten material observed on the machined surface. Figure 2 shows the surface topography on the white layer surface. The higher the peak current values lead in the increment of energy, hence, the more material eroded from the machine surface.

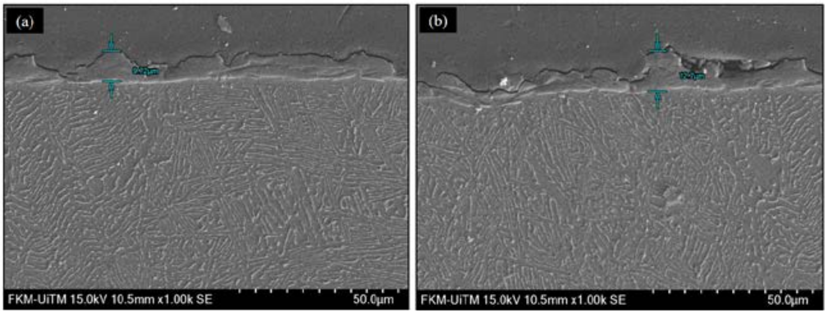

Figure 1: Formation of a white layer on the machined surface (a) Test I (b) Test III

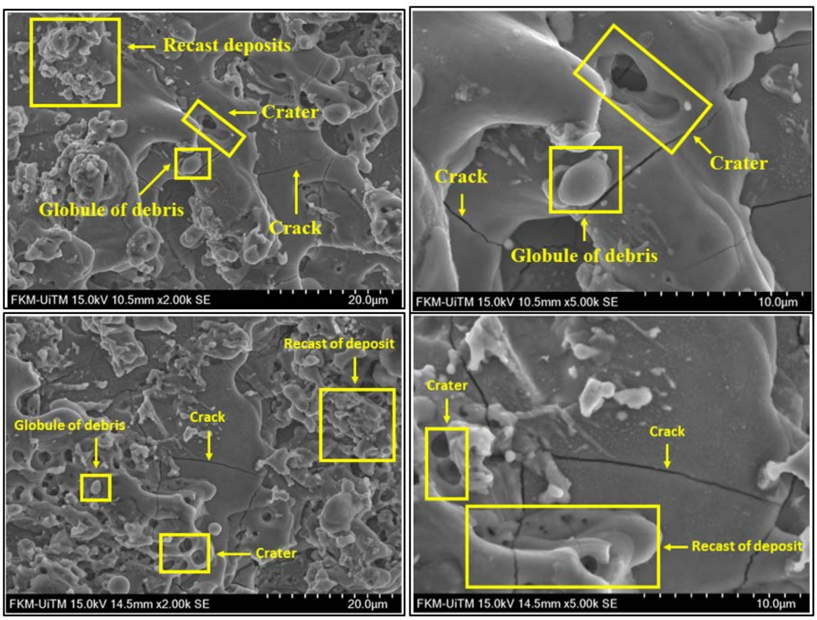

Figure 2: Surface topography on Ti-6Al-4V machined surface

The microcracks formed because of thermal stresses during the WEDM process. The microcracks thickness for low parameters (Test I) is 0.143 um, while for high parameters (Test III) is 0.411 um as shown in Figure 3. The microcracks thickness increases as the white layer thickness increase. The thickness of the white layer increases proportionately with the spark energy. The increment of peak current increasing the discharge energies and impulsive force during the machining process. Hence, more material eroded from the machined surface. Therefore, larger microcracks formed same finding as A. Kumar et al. [17].

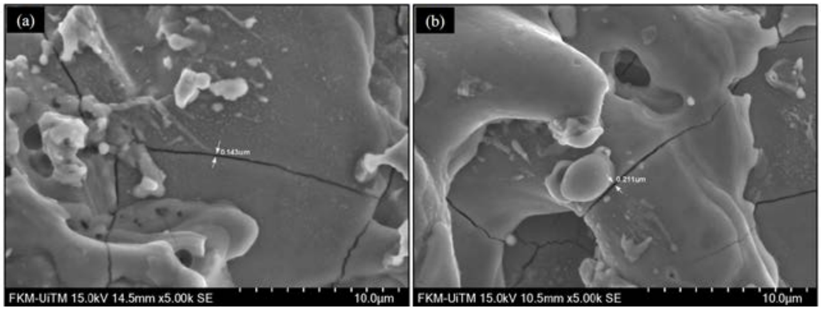

Figure 3: Microcracks on the machined surface (a) Test I; 0.143 um (b) Test III; 0.211 um

There is the presence of zinc and copper on the elementary analysis in Figure 4 because of the migration of the element from the migration from wire electrode material, which is the brass wire that comprises of 64% of copper and 36% of zinc. The elementary analysis in Figure 5 on the machined surface show the existence of oxygen due to the deionized water flow in the discharge gap. The other elements of Ti-6Al-4V remained to stay after the machining process.

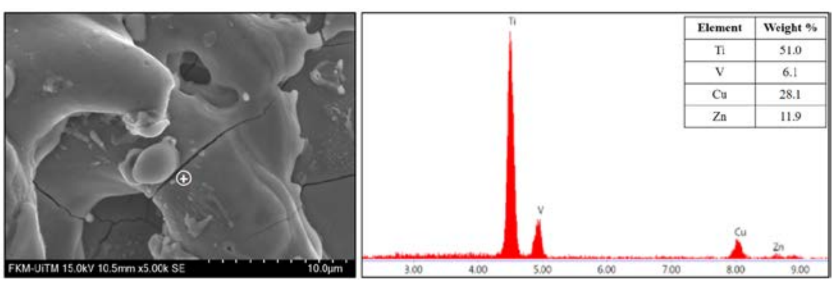

Figure 4: EDX analysis of Ti-6Al-4V; Selected Spot 1

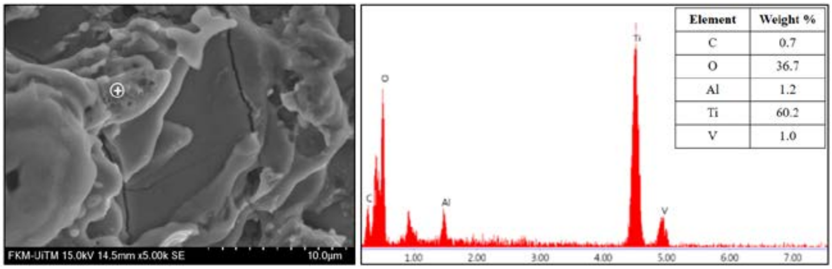

Figure 5: EDX analysis of Ti-6Al-4V; Selected Spot 2

Cell Viability

The cell growth wells and become larger in spindle-shaped. Based on Figure 6, it proved that the Ti-6Al-4V by WEDM process does not effect on the cells. If low cell viability value exists, it revealed that Ti-6Al-4V generates a cytotoxic effect. As the concentration of Ti-6Al-4V medium increases in the culture medium, the viability of the cells cultures was not altered. The concentration in 25 mg/ml to 200 mg/ml observed to have good cell viability as the percentage is more than 80%. The cells grow well in the medium, hence showing no toxic effects released. If low cell viability value exist, it revealed that Ti-6Al-4V of WEDM process generates a cytotoxic effect. The WEDM process is biocompatible and non-toxic.

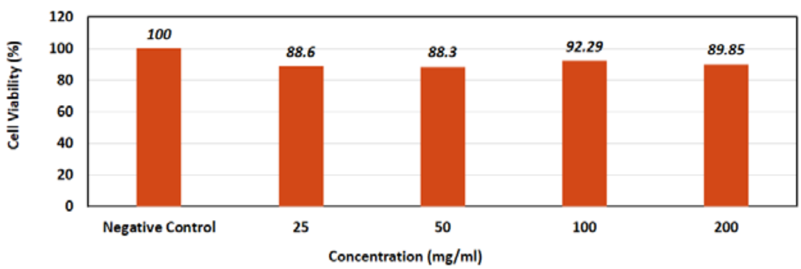

Figure 6: Percentage of L929 cell viability at different concentration

Conclusion

Based on the experimental work, the research can be concluded that the thickness of the white layer of Test III is higher than Test I. The machined surface exists of globules of debris, craters, microcracks and recast of deposits affects from the discharges energies and rapid cooling during the machining process. The microcracks of Test III is thicker than Test I. The higher the thickness of the white layer, the thicker the microcracks on the machined surface. The cell viability showed a positive result with more than 80% for each concentration of Ti-6Al-4V medium that being cultured with L929 cells. The biocompatibility tests is the first consideration of any medical product and should be free from any toxic substances. Hence the better surface characterization in this research is the set of input parameters in Test I; peak current (6A), pulse-off time (2μs), wire tension (8N).

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References

[1] B. E. J. Lee, S. Ho, G. Mestres, M. Karlsson Ott, P. Koshy, and K. Grandfield, “Dual-topography Electrical Discharge Machining of Titanium to Improve Biocompatibility,” *Surf. Coatings Technol.*, vol. 296, pp. 149–156, 2016.

[2] N. B. Vu *et al.*, “In vitro and in vivo biocompatibility of Ti-6Al-4V titanium alloy and UHMWPE polymer for total hip replacement,” *Biomed. Res. Ther.*, vol. 3 (3), no. March, 2016.

[3] F. H. (Sam) Froes, *Titanium for medical and dental applications—An introduction*. Elsevier Inc., 2018.

[4] A. Pramanik, M. N. Islam, A. Basak, and G. Littlefair, “Machining and Tool Wear Mechanisms during Machining Titanium Alloys,” *Adv. Mater. Res.*, vol. 651, pp. 338–343, 2013.

[5] A. Pramanik, “Problems and Solutions in Machining of Titanium Alloys,” *Int. J. Adv. Manuf. Technol.*, vol. 70, no. 5–8, pp. 919–928, 2014.

[6] M. S. Dargusch, S. Sun, J. W. Kim, T. Li, P. Trimby, and J. Cairney, “Effect of tool wear evolution on chip formation during dry machining of Ti-6Al-4V alloy,” *Int. J. Mach. Tools Manuf.*, vol. 126, pp. 13–17, 2018.

[7] a. Vyas and M. C. Shaw, “Mechanics of sawtooth chip formation in metal cutting,” *J. Manuf. Sci. Eng. - Trans. ASME*, vol. 121, pp. 163–172, 1999.

[8] F. Klocke, M. Schwade, A. Klink, and A. Kopp, “EDM machining capabilities of magnesium (Mg) alloy WE43 for medical applications,” vol. 19, pp. 190–195, 2011.

[9] A. V Linovsky and A. P. Morgunov, “The Influence of Steel 35 Wire

EDM Parameters on the Surface Roughness and Morphology," *J. Mech. Eng.*, vol. 14, no. 2, pp. 36–48, 2017.

[10] A. K. F. Klocke, D. Welling*, J. Dieckmann, "Titanium Parts for Medical Sector Made by Wire-EDM," no. May 2012, 2015.

[11] B. Singh, S. Kumar, and S. Kumar, "Electric Discharge Machining and Its Parameters : A Review," *Int. J. Eng. Technol. Sci. Res.*, vol. 5, no. 5, pp. 457–463, 2016.

[12] I. Maher, A. A. D. Sarhan, and M. Hamdi, "Review of improvements in wire electrode properties for longer working time and utilization in wire EDM machining," *Int. J. Adv. Manuf. Technol.*, vol. 76, no. 1–4, pp. 329–351, 2014.

[13] B. R. Chauhan, P. H. Agarwal, and S. D. Daxini, "A Review on the Conventional and Wire-Electro Discharge Machining of Titanium and Its Alloys," *Int. J. Recent Sci. Res.*, vol. 9, no. 3, pp. 25311–25322, 2017.

[14] A. Kumar, V. Kumar, and J. Kumar, "An Investigation into Machining Characteristics of Commercially Pure Titanium (Grade-2) using CNC WEDM," *Appl. Sci. Mater.*, vol. 159, pp. 56–68, 2012.

[15] S. Roy *et al.*, "ScienceDirect ScienceDirect ScienceDirect Analysis of MRR and Surface Roughness in Machining Ti-6Al-4V 2nd International Conference on Materials Manufacturing and Design Engineering ELI Titanium Alloy Using EDM Process Analysis of MRR and Surface Roughnes," *Procedia Manuf.*, vol. 20, no. 2017, pp. 358–364, 2018.

[16] L. Manufacturing and L. Sigma, "Experimental Study of Process Parameters through Dissimilar Form of Electrodes in EDM Machining Experimental Study of Process Parameters through Dissimilar Form of Electrodes in EDM Machining," no. January, pp. 0–31, 2016.

[17] A. Kumar, V. Kumar, and J. Kumar, "Surface crack density and recast layer thickness analysis in WEDM process through response surface methodology," *Mach. Sci. Technol.*, 2016.