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The Change of Surface Alloy Compositions and Corrosion Behavior after WEDM Machining of Commercially Pure Titanium (grade 4) and Ti-6Al-4V (grade 5)

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Abstract

Nowadays, titanium and its alloys are widely used in medical applications because of excellent corrosion resistance and a high strength-to-weight ratio. Nevertheless, titanium and its alloys are classified as difficult-to-machine metals by conventional machining methods. Therefore, unconventional machining processes are recommended. Wire-electrical discharge machining (WEDM) has become an important non-traditional machining process, as it provides an effective solution for machining components made from difficult-to-machine metals.

However, WEDM could damage the surface layer and have an effect on the biocompatibility properties. This research was focused on the effect of WEDM on grade 4 commercially pure titanium and Ti-6Al-4V (Grade 5) after WEDM cutting. The diffusion of wire substance was studied, and corrosion behavior in Hank's solution was examined.

The experimental result showed that the depth of damaged layer on WEDMed specimens was around 5-12 μm, and the surface compositional analysis showed a very limited depth of contamination from wire electrode. The corrosion rate of the WEDMed specimens are 300 - 400 higher than the rate found in the as-received specimens.

Key word: Corrosion, Diffusion, WEDM, Titanium, Ti-6Al-4V

Introduction

Titanium is widely used in medical applications due to its high strength to weight ratio, good corrosion resistant and biocompatibility [1, 2]. Titanium derives its corrosion resistance by forming a stable, and highly adhesive, protective oxide film on its surface. This surface oxide film limits the rate of corrosion and metal release to a level that is non-toxic to the human body [3]. Most biomedical materials are manufactured by machining process, in which the machining effectiveness is related to materials properties. Titanium and its alloy are classified as difficult-to-machine by traditional method due to their mechanical properties [4, 5]. Hence, there are situations where traditional methods are not satisfactory and non-economical for difficult-to-machine materials.

In this research, the Wire-Electrical Discharge Machining (WEDM), an electrical machining method, has been employed. The problems associated with the traditional machining processes are eliminated since WEDM used the electrical spark without making direct contact with the material being removed. This process also provides a precise and effective solution for machining of difficult-to-machine materials [6-8].

However, several studies [9-12] showed that tremendous heat generated during WEDM caused changes in the microstructure and alloy composition [13, 14] and produced a non-uniform re-growth of oxide on the surface of machined materials. When these components are used as implants in the human body, this damage may cause problems, for example an increase in the rate of corrosion to a level that is harmful to humans.

The present work involves measuring the depth of damaged layer, investigating the changes in the surface alloy composition and the resulting changes in corrosion rate under machining conditions after WEDM machining. The corrosion studies were carried out in replicated human body fluid [15].

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Materials and Experimental Procedures

The experiment started with raw materials examination. Metallographic examination was performed according to the procedure as follows; section of specimens by abrasive machining and metallurgical preparation were performed. The JEOL-JSM 5410LV SEM equipments were used for photographing of specimen microstructure, examined at 1000x and 2000x magnitude (see also Figures 1 and 2). The microstructure of CP-Ti consists of large α phase with amounts of 1-2% β phase was observed at α grain boundary. The average grain sizes of α and β are 28 µm and 2-3 µm, respectively. The microstructure of Ti-6Al-4V consists of α phase and approximately a proportion of 23% β phase. The average grain sizes of α and β are 7-8 µm and 1 µm, respectively [16].

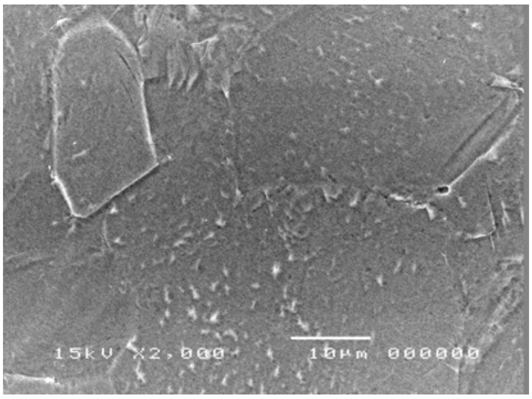

Figure 1. Microstructure of commercially pure titanium (grade 4) at 2000x.

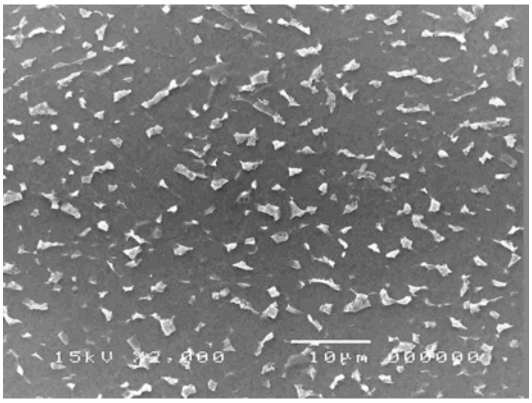

Figure 2. Microstructure of Ti-6Al-4V (Grade 5) at 2000x.

Before the machining experiment, the Charmilles Robofil 300 WEDM machine was prepared. All specimens were WEDM machined into dimension of 20x10 mm (5 mm thickness) by using a 0.25 mm diameter cobra-cut wire (a proprietary zinc coated brass wire).

A series of process parameters used in the experiment were chosen through reviews of literatures and preliminary study. The preliminary study on the process parameter referred to the technology file from the reference technologies manual of the robofil 300 WEDM machine.

The experimental strategy adopted in this experiment was Taguchi's L-18 orthogonal array. In this investigation, the process parameter consisted of V, IAL, A, B, Aj, Wb, INJ. Taguchi's L-18 Orthogonal array can reduce the number of conditions from $2^1(3^7)$ = 4374 to 18 conditions (see also Table 1) [17].

WEDM cutting of specimens referred to a series of process parameters set in the experimental design (see also Table 1). There are 2 or 3 repeated in each condition (see also Figures 3 and 4).

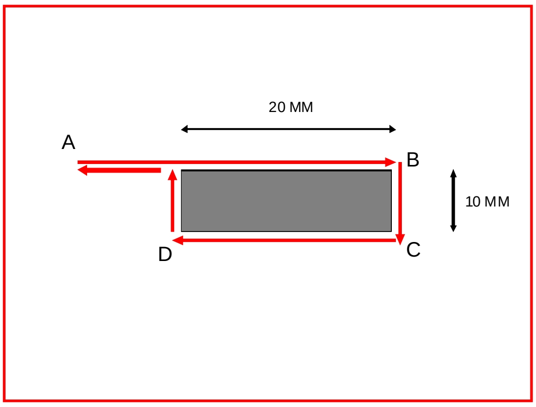

Figure 3. Cutting path.

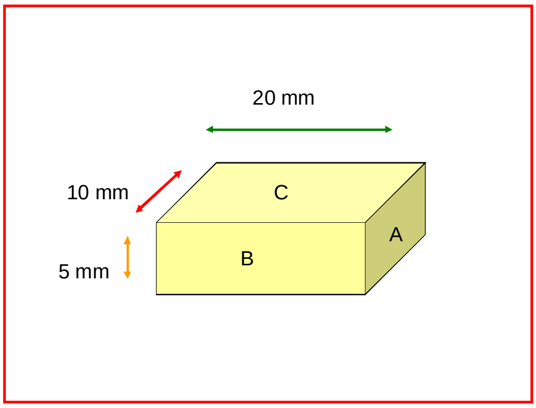

Figure 4. Illustration of machined specimens.

Table 1. Experimental series and their corresponding parametric value (thickness = 5 mm, S = 2.0, Ws = 8.0).

NO.	V	IAL	A	B	Aj	Wb	INJ
1	- 80	2	0.2	20	20	0.6	2
2	- 80	2	0.3	22	24	0.9	4
3	- 80	2	0.4	24	28	1.2	6
4	- 80	3	0.2	20	24	0.9	6
5	- 80	3	0.3	22	28	1.2	2
6	- 80	3	0.4	24	20	0.6	4
7	- 80	4	0.2	22	20	1.2	4
8	- 80	4	0.3	24	24	0.6	6
9	- 80	4	0.4	20	28	0.9	2
10	- 100	2	0.2	24	28	0.9	4
11	- 100	2	0.3	20	20	1.2	6
12	- 100	2	0.4	22	24	0.6	2
13	- 100	3	0.2	22	28	0.6	6
14	- 100	3	0.3	24	20	0.9	2
15	- 100	3	0.4	20	24	1.2	4
16	- 100	4	0.2	24	24	1.2	2
17	- 100	4	0.3	20	28	0.6	4
18	- 100	4	0.4	22	20	0.9	6

After WEDM cutting of specimens, SEM/EDS examinations were performed. The measuring of the rate of corrosion will be performed on the surface A of specimens.

Results and Discussion

Indeed, the damaged layers consisted of 3 layers, namely re-attached layer, recast layer and heat-affected zone. However, the heat-affected zone was found to be limited in its thickness and could not be distinguished from the as-received microstructure. Therefore, the depth of damaged layer reported here was measured from the extreme surface to the end of recast zone.

Measuring of the depth of damaged layer was set in 15 measuring fields in each condition. The Semafore software was used for direct measurement and the statistical method was employed to examine the reliability of the result.

The result of measurement is shown in Table 2. From the result, Condition 15 showed the highest damaged layer thickness for both materials (see also Figure 5). From the result, the thickness of damaged layer varied from 6 to 12 µm.

Table 2. Depth of damaged layer of machined specimens.

Condition	CP-Ti (µm)	Ti-6Al-4V (µm)
1	6.769	6.515
2	7.708	9.689
3	6.925	7.496
4	9.736	8.301
5	8.517	8.928
6	8.046	8.591
7	6.835	9.202
8	7.616	6.713
9	8.818	9.684
10	6.798	9.739
11	10.406	7.72
12	9.614	8.693
13	7.449	7.966
14	8.12	7.092
15	10.64	11.597
16	7.858	9.983
17	8.846	10.4
18	9.799	9.69
Average	8.32566	8.77772

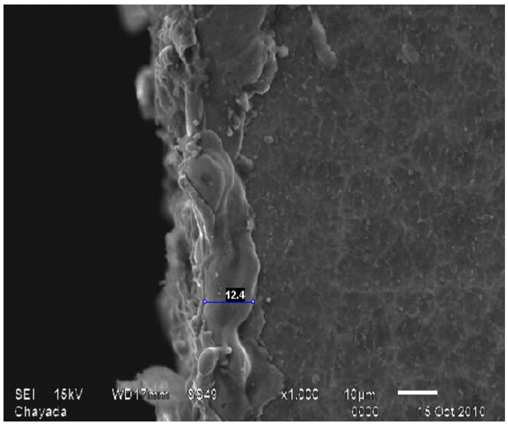

Figure 5. Damage layer of condition 15 WEDMed Ti-6Al-4V (1000x).

The surface compositional change was studied using ISIS 300 Oxford Instruments coupled with EDS equipment. The examination started with line-scanning initials from the extreme surface with the depth of 20-30 µm on Surface C. The result suggests that Cu and Zn contamination did not appear according to line-scanning examination.

The area analysis was performed on Surface B to confirm the experimental result. The result also suggests that the composition at the analyzed area did not consist of Cu and Zn.

The point analysis was applied on some positions of the machined surface (surface B in Figure 4), for example points where there are positions of swell re-attached layer and bed recast layer, respectively (see also Figure 6).

From the series of examination by using EDS, the results showed limit in wire-electrode element contaminations. From the magnitude of line-scanning examination it can be estimated that the depth of damage layer is not more than 2 µm [18, 19].

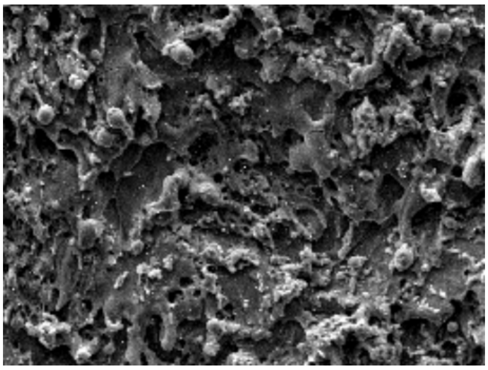

Figure 6. Surface B of condition 7 WEDMed CP-Ti (1000x).

The corrosion rates were measured in a standard flush cell. The Autolab Instrument was used for measuring the rate of corrosion. All the measurements were performed in Hank's solution, artificial body fluids. The automated software NOVA 1.5 was used to analyze and plot the data into a form of the polarization curve. The Tafel extrapolation method was used to determine the rate of corrosion. Figure 7 shows the selected polarization curve of specimen. Table 3 lists the corrosion rate measured from 18 conditions of CP-Ti and Ti-6Al-4V, respectively.

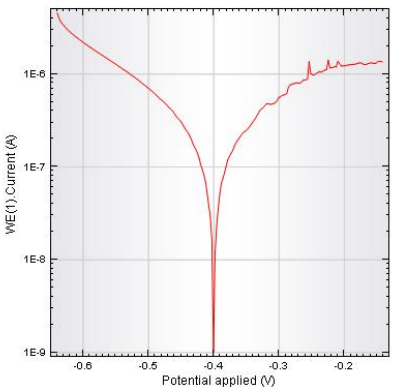

Figure 7. Polarization Curve of conditions 13 WEDMed CP-Ti.

The result of corrosion experiment shows that the WEDM machined specimens provided extremely increased corrosion rates in comparison to those found in the as-received specimens. However, the passivation process can bring the rate of corrosion back to the level possessed by the as-received materials [20].

Table 3. The corrosion rates of WEDMed Specimens.

Condition	CP-Ti ($\times 10^{-3}$ mm/y)	Ti-6Al-4V ($\times 10^{-3}$ mm/y)
1	21.214	194.140
2	4.2184	21.494
3	1.1218	6.878
4	24.163	6.474
5	174.990	21.580
6	453.550	3.340
7	39.130	1.838
8	182.900	37.959
9	3.678	13.090
10	3.564	1.514
11	3.333	5.596
12	31.580	52.094
13	1.825	1.242
14	30.258	3.487
15	83.780	23.410
16	45.480	11.093
17	55.604	31.603
18	7.748	41.564
As-recieved	0.059	0.874
E1 passivation	0.442	0.922
E6 passivation	0.339	-
E3 passivation	-	0.859

Results Analysis

The software SPSS 15.0 was employed to determine the influence of process parameters on the depth of the damaged layer. The analysis of the parametric influence on the damaged depth for various experimental conditions is depicted in Figures 8 and 9. The results showed that B, A and V value were the most significant process parameters to the depth of the damaged layer of CP-Ti and Ti-6Al-4V. This is due to the fact that these parameters were affected by heat energy applied to the specimens' surface and influence to the damaged layer formed.

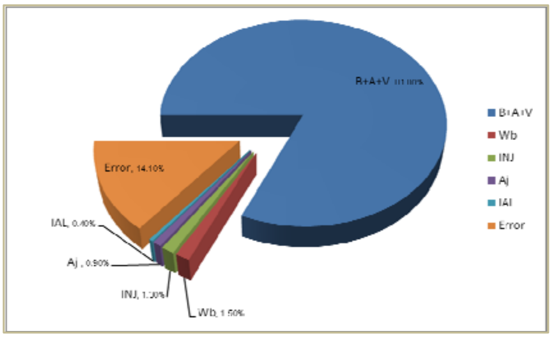

Figure 8. Influence of process parameter on the depth of damaged layer of WEDMed CP-Ti.

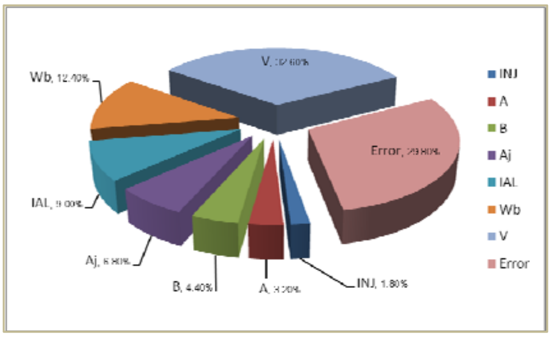

Figure 9. Influence of process parameter on the depth of damaged layer of WEDMed Ti-6Al-4V.

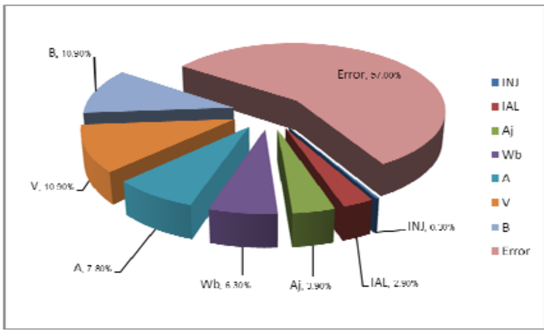

Figure 10. Influence of process parameter on the corrosion rates of WEDMed CP-Ti.

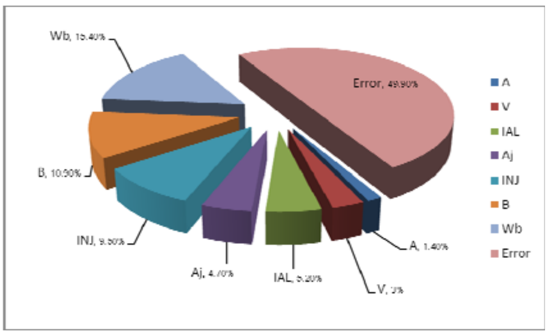

Figure 11. Influence of process parameter on the corrosion rates of WEDMed Ti-6Al-4V.

The corrosion rate measurement results were also analyzed. The analysis of parametric influence to the corrosion rate is shown in Figures 10 and 11.

The analytical results showed that B and V value were most significant parameters to the corrosion rates of CP-Ti. It seems reasonable that they affected to spark duration and energy, respectively, and deteriorated the oxide film layer on the specimen surface, which then resulted in an increase in the corrosion rate, whereas wire mechanical tension (Wb) was the most significant parameter to the corrosion rates of Ti-6Al-4V. However, Wb value is not the parameter directly affected to heat generated on the specimens' surface. Based on these results, more explanations can be given. The wire tension can affect the surface roughness. Certain authors [(21)] suggested that increase of the surface roughness on the electro-erosion specimens can be valuable to corrosion attack such as pitting or crevice corrosion.

In additional, the parameter used in this experiment may set in too narrow; hence, significant results cannot be seen. To confirm this assumption, an additional experiment was performed on Ti-6Al-4V. The additional experiment was performed by an increase of the process parameters out of range (see also Table 4). This result also showed that the increase of process parameters may cause clearly observed Cu contamination. The diffusion profile is shown in Figure 12.

Table 4. Process parameter of additional experiment.

V	IAL	A	B	Aj	Wb	INJ
-100	5	0.5	26	32	1.0	4

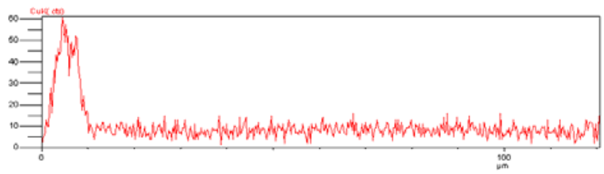

Figure 12. Diffusion profile of additional experiment.

Conclusions

In this research, the effect of WEDM on the changes in damaged layer, surface alloy compositions and corrosions rates of commercially pure titanium grade 4 and Ti-6Al-4V (grade 5) were studied.

From the result of SEM examination, many craters and damaged layer, a typical appearance surface machined by WEDM, were clearly observed in micrograph of machined surface. The surface also consisted of molten metal due to rapid heating and cooling and then re-solidification on the surface. The measured depths are around 6-12 µm. The result of the of the EDS experiment revealed that the contamination was limited, and the depth of diffusion was estimated to be no more than approximately 2 µm.

However, the result of corrosion rate measurement showed that the WEDM specimens are 300 – 400 times higher than the rates found in the as-received specimens.

From the result above, it can be seen that the WEDM process resulted in surface alteration. Although there are limits in depth of damaged layer and wire electrode diffusion, the high corrosion rates reduce the materials performance when it was used in biomedical application. In fact, most biomaterials are subject to prolonged exposure to the biological electrolyte. The high corrosion rates value as reported in this experiment must be considered seriously. Therefore, a further study should be performed on the procedure to reduce the effect from WEDM.

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