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TECHNICAL NOTE

Mechanical Surface Treatments of Ti-6Al-4V Miniplate Implant Manufactured by Electrical Discharge Machining

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ABSTRACT

Present work aims at multi-mechanical surface treatment of Ti-6Al-4V based-miniplate implant manufactured by electrical discharge machining (EDM) for biomedical use. Mechanical surface treatment consists of consequent use of ultrasonic cleaning, rotary tumbler polishing, and brushing. Surface layers are analyzed employing scanning electron microscopy and energy dispersive X-ray spectroscopy. All methods employed are capable of removing surface layer of transformed material created by EDM. These mechanical methods can provide the surface roughness of the miniplate in moderately rough category. Ultrasonic cleaning and rotary tumbler polishing took the significant increase of surface roughness with 90 and 67%, respectively. Furthermore, the brushing technique became the best benchmark for reducing the contamination of Cupper (Cu) on the surface of Ti-6Al-4V implants compared to ultrasonic cleaning and rotary tumbler polishing which hardly gave any impact and took the toxicity effect on both MTT Assay and direct toxicity tests.

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1. INTRODUCTION

Titanium and its alloys (Ti–6Al–4V, Ti–6Al–7Nb, Ni–Ti and others) have a long record of applications in implantation. The excellent combination of biocompatibility, mechanical properties, chemical stability and high corrosive resistance is the main reason for the selection of titanium and its alloys as implant material [1-3].

There are several methods employed in the production of titanium based-miniplate implant. Electrical discharge machining (EDM) is one of the most common ways employed in mass-production of the miniplate implant. EDM has its advantages compared to machining in that cavities with thin walls, and excellent features can be produced, the use of EDM is not affected by the hardness of the work material, and the process is burr-free [4, 5].

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The rate and quality of osseointegration in titanium implants are related to their surface properties. Surface composition and roughness are parameters that may play a role in implant-tissue interaction and osseointegration [6].

Recently, many works have been carried out on surface treated commercial titanium implants to enhance the osseointegration function. By increasing the surface roughness, an increase in the osseointegration rate and the biomechanical fixation of titanium implants have been observed [7]. A huge number of the experimental investigations [6, 8-12] have demonstrated that the implant surface topography influenced the tissue response; smooth (Ra <0.5 μm) and minimally rough (Ra 0.5–1 μm) surfaces showed less strong responses than rougher surfaces. Moderately rough (Ra 1–2 μm) surfaces showed stronger responses than rough (Ra >2 μm).

Furthermore, the chemical composition of implant surface has a crucial role in determining stability and

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reactivity of implants, especially implants made from titanium and its alloy. To date, research on the effects of impurities on implant surface has not been widely conducted. The prior studies have not produced an inevitable conclusion about it anyway. However, the experts of implantation believe that the level of implant surface contamination should be as low as possible.

This research aims to observe the increase of surface roughness of Ti-6Al-4V implants manufactured by EDM (see Figure 1) using several techniques such as ultrasonic cleaning, rotary tumbler polishing, and brushing. This is due to the final surface roughness values that can be achieved by EDM are in the range of 0.25-0.8 µm [13]. Afterwards, through EDS analysis, also observed the effects of these techniques on the decrease of copper (Cu) content on the implant surface and the formation of the oxide layer.

2. METHODS

2. 1. Material Preparation In this research, all investigations were using Ti-6Al-4V titanium alloy. The design of the miniplate implants made in this research referred to the geometry of the existing products that have been widely used [14]. Miniplate implant specimens were manufactured using EDM method with a high peak current of 30 A.

Afterwards, the surface treatment was employed to determine the surface roughness of specimens using several techniques. The first method was the polishing by a steel brush. The specimens were mechanically polished for 30 minutes. The second method was ultrasonic cleaning process. Al_2O_3 was added to take effect on specimen surface roughness. Specimens were cleaned on Digital Ultrasonic Cleaner for 4 hours. The last treatment was by using rotary tumbler polisher, Kyngty KT 6808. Sintered Al_2O_3 was used as polisher balls. This process was done for 8 hours. These methods were chosen because of their low costs as techniques of implant surface treatment that can provide significant results.

2. 2. Surface Roughness The surface roughness of specimens was evaluated by using Surfcom 2900SD3.

Figure 1. Ti-6Al-4V implant miniplate manufactured by EDM [15]

There were two essential parameters of surface roughness observed. Rmax is the difference between the highest and lowest point of the profile in the evaluated region. Ra is average deviation of the roughness profile [16].

2. 2. SEM-EDS Observations Topography observation was performed by scanning electron microscope (SEM) FEI Quanta 650 at the accelerating voltage of 10 kV. This observation was followed by energy dispersive spectroscopy (EDS) observation by using Oxford Instruments. This was to observe the reduction of Cu content on the surface of the implants. In the EDM process, Cu contamination was generated from both the wire-cutting process when forming the outside profile of miniplate and the slicing process using Cu-wire.

2. 3. Biological Evaluation Several techniques used as implant surface treatment are expected to reduce contamination of Cu elements on the surface of the specimen. The MTT assay and direct toxicity tests were performed to evaluate the biological effects of each sample. In the MTT assay test, the sample was observed for three days to observe the impact on the viability and proliferation of mesenchymal stem cells (MSC). In the direct toxicity test, each sample was observed for seven days to evaluate cell morphological changes and the percentage of proliferation ability.

3. RESULTS AND DISCUSSION

3. 1. Surface Roughness Surface roughness was measured for different types of specimens, and the results are summarized in Table 1. Benchmark EDM plus ultrasonic cleaning specimens proved to have the highest Ra. This method significantly increased the original roughness achieved by previous EDM process. Further, it also took the highest percentage of roughness increase about 90%.

Benchmark rotary tumbler polishing specimens also proved an excellent increase enough of surface roughness.

TABLE 1. Surface Roughness: EDM – electrical discharge machining, UC – ultrasonic cleaning, RTP – rotary tumbler polishing

Condition	R_{max}(µm)		R_a(µm)	
	Mean	**Std. dev.**	**Mean**	**Std. dev.**
EDM	6,72	1,01	1,01	0,11
EDM+UC	12,05	2,05	1,92	0,18
EDM+RTP	11,15	2,34	1,69	0,19
EDM+Brushing	7,83	0,87	1,25	0,05

On the other hand, benchmark brushing specimens could not increase the surface roughness of specimens. Even there were found some specimens that experienced a decrease in surface roughness than before.

As the easiest and fast method used to acquire the good surface roughness required in implantation, ultrasonic cleaning is recommended as a better method for surface characterization. In addition, this method can clean debris and contaminants on the implant surface.

3. 2. SEM-EDS Observations All benchmarks of surface treatments were subjected to SEM observations. Micrographs are depicted in magnification of 1000x. Figure 2 shows the comparison of the surface micrographs after several processes. Figure 2a depicts the surface of the original EDM specimens. Material is seriously damaged by EDM. Drops of resolidified metal, debris, and craters are well visible.

Figures 2b and 2c show the specimen surface after rotary tumbler polishing and ultrasonic cleaning processes. Both SEM results show somewhat identical results. They are clear that substantial part of the macro-roughness is removed. Sintered-Al_2O_3 used in these methods successfully increased the roughness of the specimen surface to moderately rough. Visually, we can conclude that these surfaces have higher surface roughness than the original EDM and brushing process. Micrograph of the specimen surface after brushing depicted in Figure 2d. It appears that the specimen surface is much smoother compared to ultrasonic cleaning and rotary tumbler polishing. This process was able to scrape debris on the surface of the specimen.

As shown in Figure 3a, the contaminant of Cu element on the original surface of EDM is very high. This is due to Cu-wire used during the wire-cutting process to form the outer profile of miniplate, as well as during the slicing process to divide into several pieces of the specimen. The EDS results for the specimen surface after rotary tumbler polishing (Figure 3b) shows that there is no significant decrease in the contamination of Cu element on the specimen surface. It is clear that this treatment could not remove Cu element on the specimen surface. Figure 3c shows the results of the EDS analysis for benchmark ultrasonic cleaning. The result is almost identical to the benchmark rotary tumbler polishing. There is no significant decrease in the contamination on the surface of the specimen, but the percentage reduction is slightly less than the rotary tumbler polishing process. EDS results of benchmark brushing, as shown in Figure 3d, shows that the contamination of Cu element on the surface of the specimen is almost 0%. With longer brushing time, this process is more suitable as roughness smoothing technique to obtain the minimally rough (<0.1 μm) roughness category.

3. 3. Biological Analysis The results of EDS analysis indicate that both rotary tumbler polishing and ultrasonic cleaning methods could not take any effect on the decreasing contamination of Cu elements on the sample surface. Therefore, in this biological analysis test, the performance of the sample would be compared to the sample-brushing which was dramatically capable of removing contamination of Cu elements. The results of the MTT assay test are shown in Table 2.

The results of the MTT test, as shown in Table 2, showed that benchmark brushing, with almost 0% of the contamination of Cu element, does not affect the viability and proliferation of mesenchymal stem cells (MSC) with living cell percentage of > 50%.

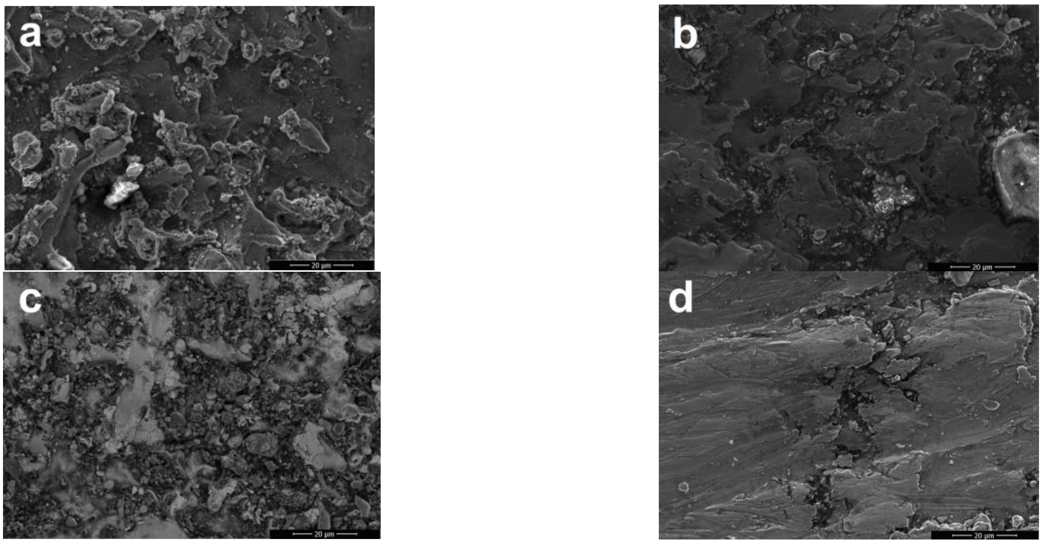

Figure 2. The comparison of micrographs of the surface. (a) original EDM; (b) after rotary tumbler polishing; (c) after ultrasonic cleaning; and (d) after brushing process

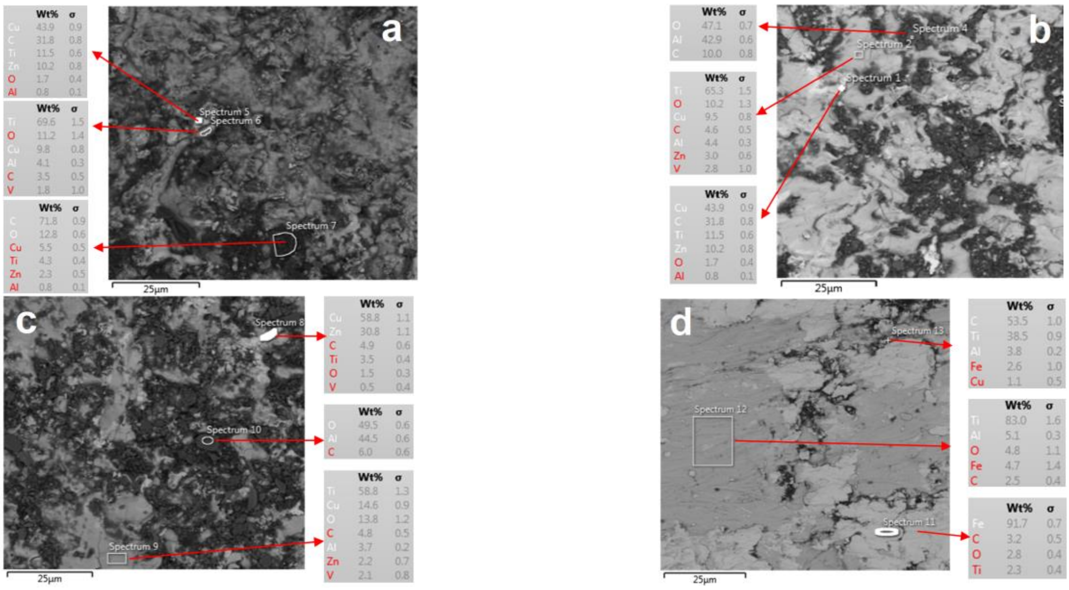

Figure 3. The comparison of EDS results (red arrows indicate the spectrum of areas analyzed): (a) original EDM; (b) after rotary tumbler polishing; (c) after ultrasonic cleaning; and (d) after brushing process

TABLE 2. The Result of The MTT Assay Test

Sample		Brushing	RTP/UC	Cell control	Media control
Day 1	OD I	0,41	0,19	0,59	0,15
	OD II	0,41	0,16	0,59	0,15
	OD III	0,44	0,16	0,61	0,14
	OD IV	0,39	0,14	0,64	0,12
Mean		0,41	0,16	0,61	0,14
% of cell		57,9	5,37	100	0
Day 2	OD I	0,43	0,18	0,57	0,15
	OD II	0,59	0,18	0,59	0,15
	OD III	0,52	0,17	0,61	0,14
	OD IV	0,53	0,17	0,64	0,12
Mean		0,52	0,18	0,61	0,14
% of cell		80,58	7,74	100	0
Day 3	OD I	0,5	0,19	0,59	0,15
	OD II	0,53	0,2	0,59	0,15
	OD III	0,54	0,16	0,61	0,14
	OD IV	0,57	0,17	0,64	0,12
Mean		0,53	0,18	0,61	0,14
% of cell		84,26	8,43	100	0

In contrast, the benchmark RTP/UC, the sample showed toxicity effects on viability and the proliferation of MSC living cell percentage of <10%. Afterwards, direct toxicity test results are shown in Table 3.

TABLE 3. The Results of The Direct Toxicity Test

Sample	Number of cells		mean	% proliferation of cells
	Well 1	Well 2		
Control	475.000	484.800	479.900	100,00
brushing	228.000	292.000	260.000	54,18
RTP/UC	0	0	0	0,00

The results of the direct toxicity test showed that, on the 3rd day after direct contact with MSC, no morphological changes in the cell planted with sample-brushing were observed. On the same day, no MSC growth was observed on sample-RTP/UC.

On the 7th day, the harvesting of cells, the data was obtained. The proliferation capability of MSC planted with sample-brushing was 54.18%, whereas for sample-RTP/UC, there was no proliferation of MSC (0%) or sample indicated the toxicity effects.

4. CONCLUSION

This study aims to modify the surface of Ti-6Al-4V miniplate implants manufactured by EDM by using several techniques. Ultrasonic cleaning and rotary tumbler polishing techniques added with sintered-Al_2O_3 provided significant results of surface increase with the percentage of 90 and 67%, respectively from original EDM Ra of 1.01 μm. The brushing technique did not provide significant changes to the increase in surface roughness of the specimen, but on the other hand, it was almost entirely capable of removing contamination of

Cu element on the surface of the implant compared to the other two methods. Furthermore, the contamination of Cu element in the sample- ultrasonic cleaning/rotary tumbler polishing indicated the toxicity effect. Future research is expected to combine some of these techniques to acquire the surface roughness in the moderately rough category (Ra 1-2 μm) which at once can significantly remove the contamination of Cu element from the surface of the implants manufactured by EDM.

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چکیده

هدف از انجام این کار درمان سطحی چند منظوره ایمپلنت مبتنی بر مینیاتوری Ti-6Al-4V است که توسط ماشین کاری الکتریکی (EDM) برای بیومدیکال استفاده می شود. درمان سطح مکانیکی شامل تمیز کردن و استفاده از اولتراسونیک، تمیزکردن روتاری، و مسواک زدن است. لایه های سطحی با استفاده از میکروسکوپ الکترونی اسکن و طیف سنجی اشعه ایکس پراکنده انرژی تجزیه می شوند. تمام روش های استفاده شده قادر به حذف لایه سطحی مواد تبدیل شده توسط EDM می باشد. این روش های مکانیکی می تواند زبری سطح miniplate را در دسته های نسبتا خشن ارائه دهد. تمیز کردن التراسونیک و صیقل دادن دوشش باعث افزایش شدید زبری سطح به ترتیب 90 و 67٪ گردید. علاوه بر این، روش مسواک زدن بهترین معیار برای کاهش آلودگی (Cu) روی سطح ایمپلنت های Ti-6Al-4V در مقایسه با تمیز کردن التراسونیک و صیقل دادن چرخ دنده بود که با سختی هیچ تاثیری نداشت و اثر سمیت را در هر دو روش MTT به دست آورده و آزمایش مستقیم سمیت بررسی گردید.

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