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Surface texturing of non-toxic, biocompatible titanium alloys via electro-discharge

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ABSTRACT

The developments of Al and V free, biocompatible Ti-alloy have been the subject of researcher in the orthopedic joints replacement domain. The current article addresses the methodology chosen for Ti-alloy design, machinability, and low-cost surface texturing process for a high degree of biocompatibility. It is evident that an astonishing increase in biocompatibility can be achieved by synchronizing electro-discharge spark energy within Ti alloy and tool material coupled with the selection of dielectric medium for surface modification. The finding of this research may benefit a wide range of researchers to design sustainable implants. The positive polarity tool electrode at 10 A is the most desirable process parameter developing the bioactive surface.

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1. Introduction

Titanium alloy demonstrates an extraordinary spectrum of properties due to the combination of outstanding mechanical, chemical responses, non-toxic and biocompatibility. Titanium exhibits allotropic forms i.e., α-Ti (hexagonal close pack structure) and β-Ti (body centred cubic structure) crystalline structure with incomplete d-shell. This incomplete d-shell of Titanium has capacitates to form substitution solid solutions. The solute/ alloying elements have a strong effect on the allotropic properties of titanium. The alloying elements that increase the microstructure transformation temperature range is indicated as α-stabilizer, while alloying elements that reduces the transformation temperature is referred to as β stabilizer. Titanium and its alloy have been widely accepted to design joint prosthesis and skeletal repair structures due to its good biocompatibility and mechanical properties [1]. Unfortunately, it is reported that early introduced Ti implants materials such as α pure-Ti and α+β (Ti-6Al-4V) alloy leads to stress shielding effects as well as the release of Al and V ions in human blood created severe health issues such as Alzheimer's disease, osteomalacia and neuropathy (Sidhu, 2020). Thus, the application of Ta, Mo, Zr elements Ti alloy is the subject of great attention among researchers. This paper presents design concepts, machinability and surface modification of Ti alloys.

1.1. Design concepts of Ti alloys

The ultimate goal of designing Ti- alloys is to obtain the resultant properties of the alloy matchable with the characteristics of biological tissue properties and which further restore and perform sustainably. Some of the most popular alloy concepts in designing the Ti-alloys system are represented in Figure 1.

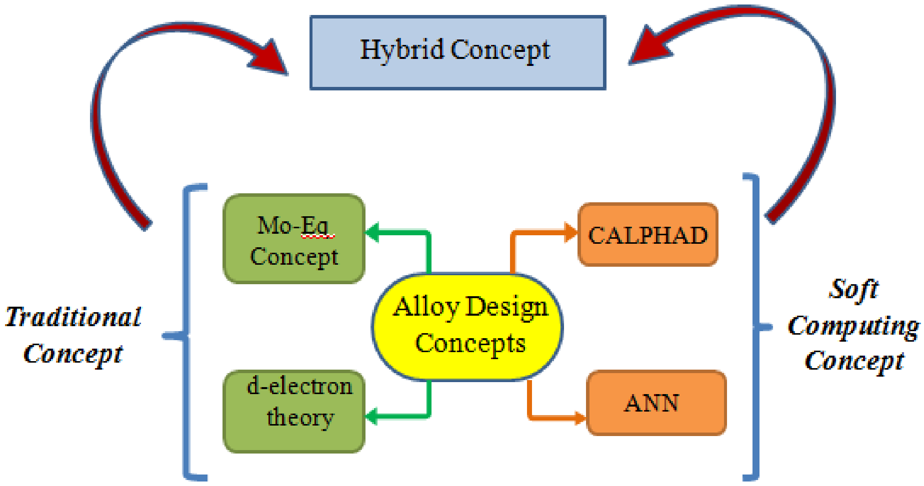

Figure 1. Alloy design concepts

1.2. Machinability of Ti alloys for precise joints developments

The process of materials removal or cutting operations is defined as the term machining. The machining methods may be conventional (i.e. workpiece-tool contact occurs) such as turning, milling drilling, etc. or non-conventional method (i.e. no contact between workpiece and tool) which may include EDM, electrochemical machining (ECM) or laser beam machining (LBM). The cutting forces in conventional machining of titanium alloys are much higher as compared to the steel with equivalent hardness. This is due to the metallurgical characteristics of Ti-alloy that makes it high-cost machinability. To alleviate the challenges related to biocompatibilty and surface integrity, the non-traditional machining process also manifests promising results. Among these, processes such as Electrical discharge machining (EDM), electrochemical machining (ECM) and laser beam machining (LBM) are widely used.

Various methods have been utilized to obtain the biocompatible substrates for the impalnts, whereas, amongst all the Electro Discharge Machining or in other term this process is also termed as Electro discharge treatment (EDT) is the novel technique in the field of surface alterations (Devgan & Sidhu, 2019). This technique utilized thermo-electrical energy source that has the potential to offers desired morphology with precise dimensional accuracy (Ahmed, 2019). In this process, high-frequency electric sparks produced between the tool electrode and workpiece which produces high-temperature heating zone. Thus, results in alteration of surface morphology and phase transformation within the spark area (Mansor, 2020). This thermo-electric process transforms the surface into an oxide/carbide enriched layer which has competences to enhance cellular functionality of human osteoblastic cell (Mughal, 2020).

In EDM, the material is removed by using spark energy generated between the workpiece and tool electrodes. In this process current, voltage, pulse duration, conductivity, polarity and dielectric medium are the significant factors effecting material removal rate (MRR) or machinability (Kumar. S, 2020, Kumar. D, 2020). However, low MRR is a serious restriction in adopting this process for mass production. A lot of research is reported to optimize the process parameters for high MRR [Sidhu, 2020, 2014a, Ablyaz, 2020). In this study, our aim is to obtain Ti alloy substrate favourable for high cell proliferation. Thus, to obtain bio-favourable substrate the non toxic chemical composition with high rough surface promotes cell anchoring.

2. Material and methods

In this experimentation, the work piece material was fabricated by using vacuum arc melting technique (Facility available at DRDO-DMRL, India). The Ti alloy design by using non- toxic β-stabilizer such as Nb, Zr, Ta in appropriate portion. The composition of β-stabilizer was decided on the basis of d-electron concept [Weng, 2021 Gepreel,2013). The composition of the alloy was examined with optical spectroscopy (Arun technology metals limited, England) having 53% Ti, 32% Nb, 8% Zr and 4%Ta. For experimentation, the workpiece was sectioned into flat plates (Size: 50mm x 50mm x10mm) which is further cleaned with ethanol solution. The experimental trials were performed on EDM (model S645 OXCARMAX) at assorted values of current and pulse on/off with both polarities (-/+) at predetermined working gap voltage between electrodes i.e. 140 V. Herein, deionised water is used as dielectric medium and fine grained graphite is selected as tool electrode. From literature and pilot experiments levels of the process parameters were selected and listed in Table 1. Table 2 represents the selected L8 Taguchi's orthogonal array (Ross, 1996) to study the effect of process variables on surface roughness (SR).

Table 1: Process parameters and levels

Factors	Levels	
	Level 1	Level 2
Polarities	Negative	Positive
Current (I)(A)	2	15
Pulse-on (Pon)(μs)	30	60
Pulse-off(Poff)(μs)	30	60

Table 2: L8 Experimental layout and responses

Trail No.	Polarity (-/+)	Peak current (IP)(A)	Pulse-on (Pon)(μs)	Pulse-off (Poff)(μs)	SR1*	SR 2*
1	+ve	2	30	30	0.592	0.596
2	-ve	2	30	60	0.378	0.364
3	+ve	10	60	30	1.146	1.188
4	-ve	10	60	60	0.790	0.646
5	-ve	2	60	30	0.486	0.497
6	+ve	2	60	60	0.507	0.419
7	-ve	10	30	30	0.546	0.672
8	+ve	10	30	60	0.957	0.973

* SR 1, SR2 repetition of responses (surface roughness)

3. Results and discussion

From Table 2 the results of responses are assessed by using MINITAB software. The ANOVA analysis is shown in Table 3. The main effect plot for the elected input parameters is presented in Fig. 3.

The ANOVA results show that the factors exhibited a larger value of F-test affirms its significance in controlling the surface roughness. Similarly, the P-value for each factor indicates its level of significance. The factors considered more significant when $P \leq 0.10$ (90% confidence level). From the results (Fig 3), it is clear that the electrode polarity and current are considered as the significant factors, whereas, pulse on/off time are insignificant as listed in Table 3. It was due to the fact that while machining in positive polarity at high current resulted in accommodation of heat thus results in rough surface.

The Fig. 3 revealed that machining at 10A current with positive tool polarity incorporating optimum values for responses.

Table 3: Analysis of Variance for surface roughness

Source	DF	Adj Sum of square	Adj Mean squares	F-Value	P-Value
Polarity	1	0.12488	0.12488	5.98	0.092*
Current	1	0.29626	0.29626	14.19	0.033*
Pulse on	1	0.01129	0.01129	0.54	0.515
Pulse off	1	0.01484	0.01484	0.71	0.461
Error	3	0.06265	0.02088		
Total	7	0.50990			

*Significant factors

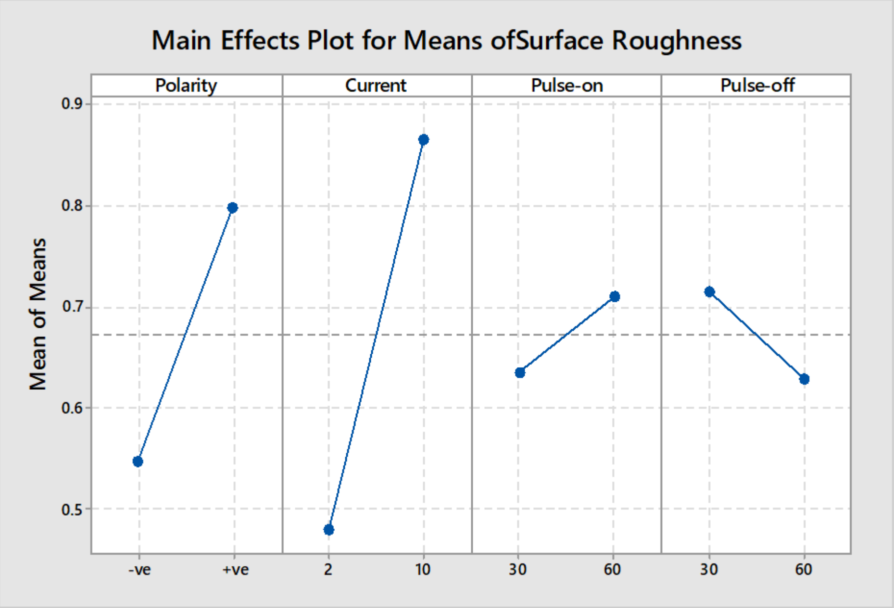

Figure 3. Main effects plot for means of surface roughness

The formation of biocompatible interface developed on Ti alloy (Fig 4a) due to electro-discharge treatment (Fig 4b) was examined for chemical composition. X-ray spectra of treated reveal the formation of bio-favorable oxides and carbide phases (Fig 4c). Furthermore, the scanning electron microscopy shows the rough surface with slight discontinuous porosity, which prompted excellent osseointegration (Fig 4d).

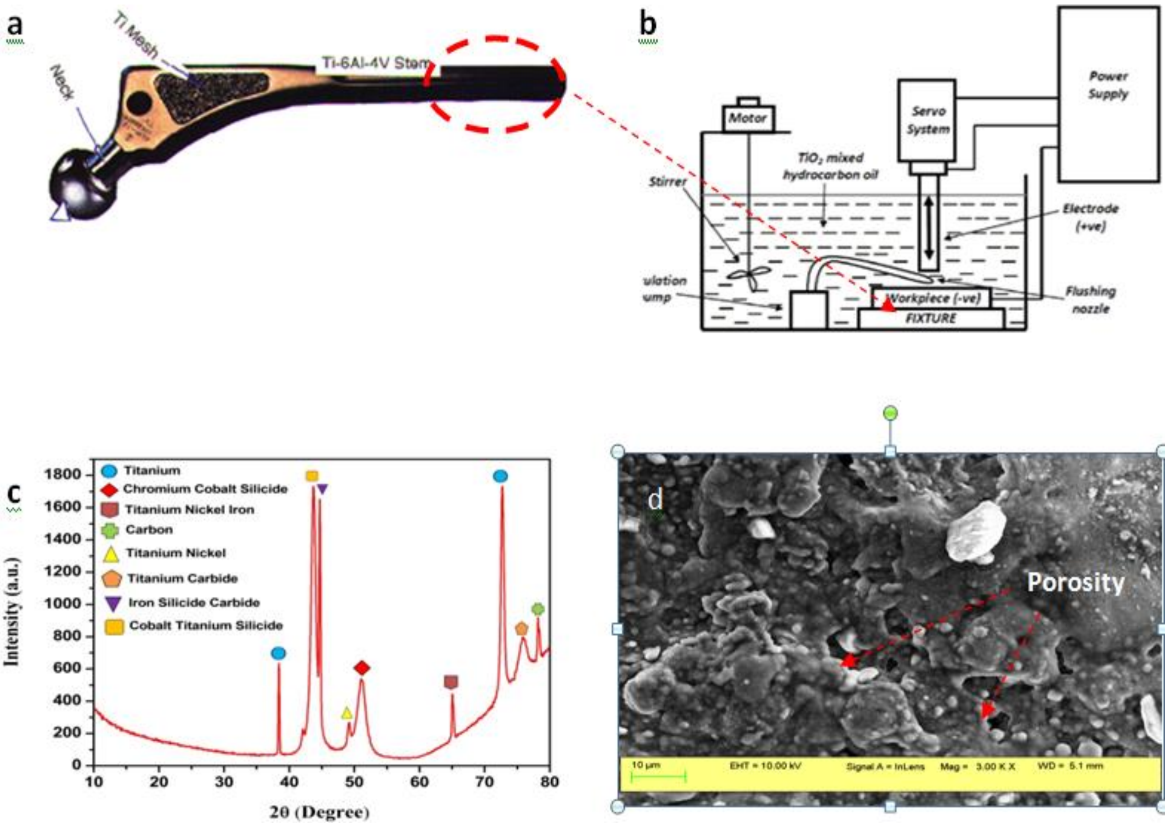

Figure 4. (a) Titanium alloy implant for hip joint (b); Electro discharge treatment of Ti-alloy stem for excellent osseointegration (c) X-ray spectra representing formation of biocompatible phases (d) Porosity for natural cell anchoring

Furthermore the surface obtained at optimum condition (+ve polarity, 10 A) was examined for %hemolysis test. The In-vitro hemolysis test was performed to evaluate the biological response of treated substrate. Initially the surface of specimen was sterilized by autoclave at 1210C for 30 minutes. For the %hemolysis test healthy rabbit blood was collected in micro-tubes and centrifuge for 10 min at 2500 rpm at the temperature of 40C to separate RBCs. Further, leukocytes and blood plasma were removed and remaining RBCs were rinsed twice by an isotonic phosphate-buffered saline (pH-7.4). Two testing reference were selected, Triton Xtm-100 demostrated 100%hemolysis, which was considered as a positive control whereas; isotonic phosphate-buffered saline (pH-7.4) has negligible hemolysis, so it was designated as a negative control. Finally, all specimens were taken in 12 well plates (dish) by ensuring that machined surfaces were directly in contact with a puddle of RBCs suspension. Finally, 1 ml equivalent volume of RBCs suspension were exposed to the treated surfaces for 1hour and incubated at 370C. By following the process, inhabited RBCs suspension from well plates were collected in microcentrifuge tubes. The absorbance of supernatant samples was measured by spectrophotometry at the wavelength of 540nm. The %hemolysis was calculated with the formula:

$$\%hemolysis = \frac{Ab_{sample}}{Ab_{Positive\ C}} x100$$

where $Ab_{positiveC}$ is the absorbance of positive control and Ab_{sample} is the absorbance of testing samples.

Herein Triton XTM-100 (Positive control) was observed with 100% hemolysis, and Isotonic PBS (Negative control) yielded 3.39±0.42%. The absorbance on Triton XTM-100 was calculated thrice as 0.398, 0.391 and 0.462 and the values absorbed on treated samples were 0.059, 0.023 and 0.011. It was also observed that trial 3 showed the least values of %hemolysis was 7.43%. Thus, the treated surface showed excellent chemical interaction with RBCs.

4. Conclusion

This paper presents the surface alteration of designed non toxic β-Ti alloys and presented the optimum process parameter to obtain biocompatible surface. The surface modified after electro-discharge process reduces %hemolysis due to carbide/oxide surface formation. The formation of rough porous surface at high current (10A) improves cell anchoring with implants and, in this manner, it stimulates the patient's post-surgery healing process.

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