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Article

Surface assessment of CNC laser treated commercially pure Titanium and Ti 13 Zr 13 Nb alloy

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Abstract
This study aimed to evaluate the surface changes of commercial pure Titanium disks (CP Ti) and the Ti 13Nb 13 Zr (Alloy) with a zigzag pattern of laser surface treatment. *In vitro*, experimental study of CNC Laser treatment on the CP Ti and Alloy disks. Texturing the surfaces of CP Ti and Alloy disks via CNC laser, the sample disks were analyzed using surface roughness, wettability and FESEM. The FESEM revealed a proper increase in the surface texturing and roughness on macro and micro measures without crack formation or dramatic change of the core substance of the CP Ti and Alloy disks. The CNC laser is an effective and suitable method for surface texturing CP Ti and Alloy for dental implantology.

Keywords: Commercial pure Titanium; Ti 13 Zr 13 Nb alloy; CNC Laser; Laser surface texturing; Dental implant and surface roughness

Introduction

Titanium and its alloys can be considered the “gold standard” material for endosseous dental implants. Among all the available dental implant materials, they are distinguished due to their many desirable properties and long-term clinical survival rates for several decades. Titanium and its alloys can interact closely with the bone tissues besides its highly biocompatible (spontaneous build-up of an inert and stable oxide surface layer [1]. The usage of Ti and its alloys as dental implants may be correlated with some disadvantages despite the excellent evidence of its usage, like the elastic moduli difference between Titanium implant and the surrounding bone, which led to stress in the bone-implant interface and peri-implant bone loss [2].

The dental implant surface modification, specifically the topographical, is considered an effective method for improving the bioactivity of dental implants [3]. A laser that improves the osseointegration could provide an implant rough surface. Several studies showed that implant surface modification by the laser technique could reduce dental implant contamination, with implant torque removal increasing after implantation in rabbit tibia and femur [4,5,6]. The laser surface modification techniques could offer better osseointegration due to the formation of surface microstructures with significant hardness enhancement, corrosion resistance, standard roughness, a high degree of purity and an increase of the oxide layer [7]. Berezani et al. stated that the oxide layer increases more than doubles after implant surface laser treatment [8].

Ti-13Nb-13Zr is a high-strength, modulus and biocompatible alloy. Implants of this alloy would have a modulus of elasticity closer to that of bone than other typically used metal alloys and do not include any elements shown or suggested as having short-term potential adverse effects [9].

Ti–13Nb–13Zr is a near β alloy formulated at the beginning of the 1990s to be used in orthopedic applications due to its low Young's modulus (40–80 GPa) and its non-toxic composition. It presents tensile values of approximately 1,300 MPa and a superior corrosion resistance compared to Ti–6Al–4V and Ti–6Al–7Nb alloys [10]. The first-generation orthopedic α+β Titanium alloys such as Ti–6Al–4V ELI (extra low interstitial), Ti–6Al–7Nb and Ti–5Al–2.5Fe are already in use. In recent years, second-generation low-modulus near β and type Titanium alloys have been developed for orthopedic applications to avoid the "stress shielding" effect caused by the modulus mismatch between the implant and the bone [11].
The Ti–13Nb–13Zr has Niobium as a beta-phase stabilizer. The other alloying element, Zirconium, is isomorphous with Titanium's alpha and beta phases. Combining these two alloying elements has made it possible to develop a structure that is a "near" beta phase, supposedly possessing a superior corrosion resistance over the alpha–beta phase alloys, with enough alpha phase present in the final structure to provide the necessary mechanical strength. It has been proposed that Ti–13Nb–13Zr alloy is more favorable for orthopedic implants than Ti–6Al–4V alloy because of its superior corrosion resistance and biocompatibility [11]. Reasons for this superiority have included the fact that less metal ion release is likely to occur during spontaneous passivation of Ti–13Nb–13Zr alloy because the corrosion products of the minor alloying elements, Niobium and Zirconium, are less soluble than those of aluminum and vanadium. Also, the passive oxide layer on the surface of the alloy is more inert, consisting of a dense rutile structure providing more excellent protection to the underlying alloy [12]. Due to the complete dissolution of the alloy elements in the Titanium matrix, a good combination of microstructure, mechanical properties and densification could be reached [11]. Hence, This study aimed to evaluate the surface changes of commercial pure Titanium disks (CP Ti) and the Ti 13Nb 13 Zr (Alloy) with a zigzag pattern of laser surface treatment.

Materials and Methods

Sample grouping

The CP Ti and Alloy disks were divided into four groups, as follows:

- (Ti) CP Ti control group, without surface treatment or structuring.
- (TiL) CP Ti with laser surface structuring.
- (Al) Alloy control group, without any surface treatment or structuring.
- (Al L) Alloy with laser surface structuring.

Samples preparation

Circular disks, 9 mm diameter and 2 mm thickness of commercial pure Titanium disks (CP Ti) and the Ti 13Nb 13 Zr (Alloy) were cut with a wire cut machine (Knuth Smart DEM-Germany). Then, these disks were polished to a mirror-smooth uniform appearance via rotation machine with sandpapers proceeded from 500 to 2400 grit. To remove contamination, the samples were placed in the ultrasonic cleaning device for 15 minutes with ethanol and then for 10 minutes with distilled water, respectively. Finally, the samples were dried at room temperature for 15 minutes [13].

Pilot study

Five scanning speeds were tested: 2000, 1200, 500, 300 and 150 mm/sec. Speeds 2000, 1200 and 500 mm/sec. This resulted in creating scattered dots that did not reveal the pattern required. Speed of 150 mm/sec. Made pattern lines overlap, and the surface turns Blackish grey, which may strongly suggest scorching the surface.

The 300 mm/sec speed was selected for it created the distinct pattern required without signs of burning the surface or scattered dots. Three designs were tested before settling for this one. The other ones had right and sharp angles in the zigzag lines instead of the obtuse ones used in this study. However, the other designs were eliminated due to increased wettability angles (reduced surface wettability) since most studies have found that hydrophilic surfaces tend to enhance the early stages of cell adhesion, proliferation, differentiation and bone mineralization compared to hydrophobic surfaces [14]. The contact angle values for the different designs are described in Figure 1, Table 1.

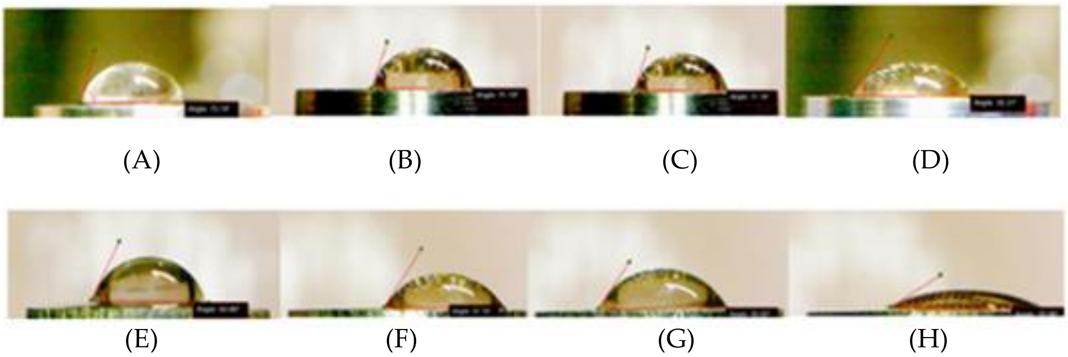


Figure 1. Contact angles of multiple designs; (A) CP Ti disc without laser treatment; (B) CP Ti disc with right angle zigzag pattern; (C) CP ti disc with sharp angle zigzag pattern; (D) CP Ti disc with obtuse angle zigzag pattern; (E) Alloy disc without laser treatment; (F) Alloy disc with right angle zigzag pattern; (G) Alloy disc with sharp angle zigzag pattern; (H) Alloy disc with obtuse angle zigzag pattern.

CP Ti			Alloy		
Sharp	**Right**	**Obtuse**	**Sharp**	**Right**	**Obtuse**
59	59	55	57	50	36
60	60	54	60	57	38
64	58	53	58	55	35
63	61	49	55	55	32
65	62	50	60	49	28
62.2	60	52.2	58	53.2	33.8

Table 1. Wettability contact angle values.

The laser system performed the desired profile on the CP Ti and Alloy disk surfaces. The surfaces of the CP Ti and Alloy were structured under standard atmosphere by using pulse mode CNC fiber laser machine (Raycus 50 Watts-China) with laser power 30-Watts, wavelength 1064 nm, Frequency of 200 pulses per second and scanning speed up to 300 mm/sec. Corel Draw software (version XII) was used for drawing the zigzag design shapes. The samples-laser source disk distance was 22 cm. When the system was triggered, the laser beam started shooting at the sample with a continuous series of laser pulses in an ablation process to form the zigzag lines design. The lines were made 416.38 µm with 116.1 µm spaces in between the lines. The zigzag lines were created with an angle of 134.88°.

	Alloy		CP TI	
	Al	**Al L**	**Ti**	**Ti L**
Sq	5.254	113.34	12.29	151.8
Sz	31.86	335.9	98.08	558.16
Sa	4.358	106	9.193	129.44
Sdr	1.044	2.8782	0.8413	1.0648

Table 2. Mean values of surface roughness parameters for CP Ti and Alloy discs.

Field emission scanning electron microscopes (FESEM)

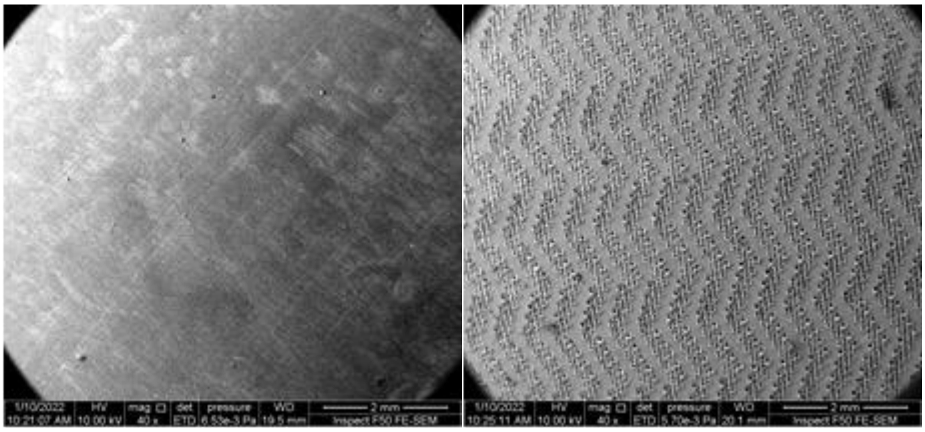
Field Emission Scanning Electron Microscopes (FEFESEM) and EDX sensor(thermos fisher teneo 2021, Holand) was used to examine the surface morphological topography of the four groups.

Surface roughness

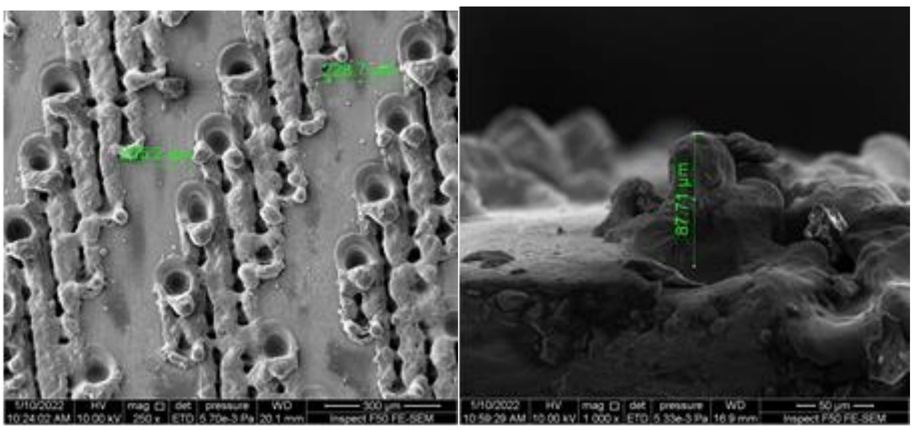
An atomic force microscope (AA3000 Angstrom Advanced Inc., USA) was used to analyze the surface roughness of the Titanium disks produced after laser surface structuring.

Results

The FESEM results of CP Ti and Alloy disk samples are seen in Figures 3 and 4, respectively. The surface morphological analyses of these pictures reveal that the laser's effect on the Ti samples' surface was more intense than that on Alloy surfaces. Which is shown clearly by the higher lines and dots created. Laser created deep trenches and holes marking the lines where the laser beam hit the surface. On the alloy samples, the lines where the laser beam scanned are left with closed lines rather than open trenches. Although this created a surface rougher on Ti, the surface created on alloy was more compact and dense, suggesting remelting layer formation.

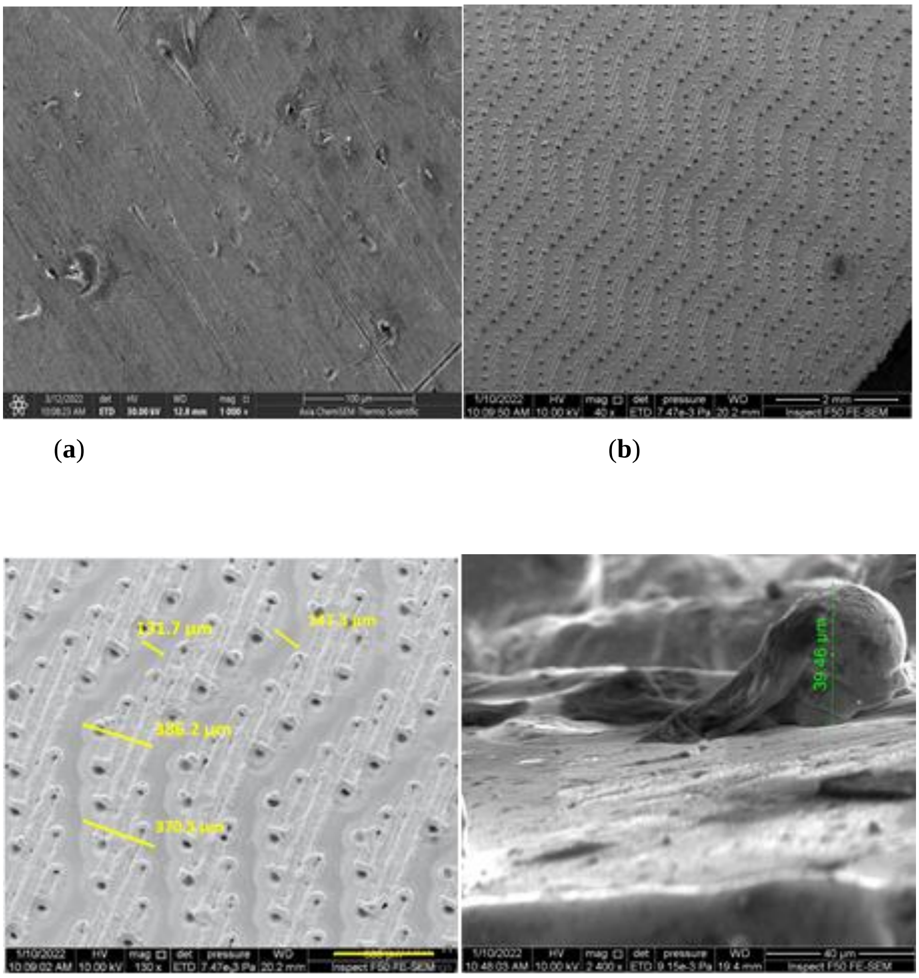


(**a**) (**b**)



(**c**) (**d**)

Figure 2. FESEM analysis of (a) Ti group, (b and c) Ti L group, and (d) Ti L group in cross-section.



(**a**) (**b**) (**c**) (**d**)

Figure 3. FEFESEM analysis of (a) Al group; (b and c) Al L group; (d) Al L group in cross-section.

Discussion

Laser surface hardening and remelting processes rely on heating the surface above the desired temperature with the help of a laser and then allowing the surface to cool down. The laser surface hardening process modifies the surface properties by imparting microstructural changes. In contrast, surface remelting induces changes in the surface topography, roughness, wettability and wear and corrosion resistance, influencing the biocompatibility of the surface. Such changes are brought in essentially because of the characteristic melting, evaporation and rapid solidification during laser surface remelting processes [15].

The Ti surface has a much faster cooling rate, leading to the solidification of the targeted area by laser beam forming the holes (Figure 2C). A slower cooling and solidification rate of the alloy allowed the heated area to flow as a liquid forming the lines (Figure 3C), and the affected area around the target area was more expansive on the alloy, which meant that there were fewer unaffected lines or zones in the Alloy discs. A remelting process on the surface of the alloy creates more profound and more compact protective layers of the much desirable Titanium oxide [16].

Conclusions

Laser surface modification of Cp Ti and Ti alloys has a pronounced positive effect on enhancing desirable qualities for dental implants. This study showed that the Ti

13 Zr 13 Nb alloy already has many mechanical advantages in dental implants over Cp Ti and can better accept laser surface structuring, leading to an intensified surface Ti oxide protective layer. Additionally, adopting a zigzag line pattern can combine many benefits of previously used patterns of dots or straight lines.

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