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Studies on Laser Surface Texturing of Titanium Alloy (Ti-6Al-4V)

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Abstract

In the present study, a detailed characterization of laser-assisted surface textured titanium alloy (Ti–6Al–4V) has been undertaken. Laser surface texturing with line and dimple geometry has been carried out using ArF excimer laser operating at a wavelength of 193 nm with a pulse length of 5 ns. Following surface texturing, an extensive characterization of the textured surface has been carried out by scanning electron microscopy, electron back scattered diffraction (EBSD) and X-ray diffraction technique. There is refinement of microstructure along with a higher mass fraction of $\mathbb P$ -titanium phase and oxides of titanium (rutile, anatase and few Ti_2O_3 phase) in the textured surface as compared to as-received one. Furthermore, in order to investigate the impact of laser surface texturing on surface energy; wettability studies have been carried out before and after laser modification. The area fractions of linear texture and dimple texture measured by image analysis software were 45 % and 20 %, respectively. The surface energy (and hence, wettability) was increased due to linear (45.6 mN/m) and dimple (39.4 mN/m) texturing as compared to as-received Ti-6Al-4V (37 mN/m).

Key words: Laser surface texturing, Ti-6Al-4V, nanoindentation.

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1. Introduction

Titanium and its alloy are promising materials for bio-implant application due to its high strength to weight ratio, good corrosion resistance, low density and relatively low young's modulus [1]. However, its poor wear resistance and bio-inertness create problems in long term use of Ti-6Al-4V as bio-implant [2]. Wear resistance and biocompatibility may be tailored by modification of surface microstructure and composition [3]. In the past, several studies have been undertaken to improve bio-compatibily of titanium and its alloys by physical, chemical or electro-chemical routes [4]. Laser surface engineering is a promising route for improving the surface dependent engineering properties of metallic component [5]. Laser surface melting and laser gas alloying with nitrogen have been successfully attempted to tailor hardness, corrosion resistance and biocompatibility of titanium and its alloy by laser surface engineering [6,7]. Surface topography plays an important role in influencing the bio-compatibility of any component and may be preferentially introduced by physical, chemical and mechanical means [8-10]. Laser surface texturing is an environmental friendly technique, where, a high energy density pulsed laser beam is applied on the surface to ablate materials from the surface and thereby, introducing the surface roughness with preferred orientation [10]. In the past, studies have been undertaken to understand the effect of texture dimension and orientation on cell attachments on to Ti-6Al-4V surfaces [11-13]. In this regard, it is relevant to mention that though the effect of laser surface texturing of the cell adherence has been studied, however, an extensive effort on the effect of laser surface texturing on the microstructure and mechanical properties has not been undertaken in details. In the present study, a detailed investigation of the microstructures and mechanical properties of laser surface textured Ti-6Al-4V with linear and dimple geometries has been undertaken.

2. Experimental

In the present investigation Ti-6Al-4V of dimension 10 mm \times 10 mm \times 5 mm was mechanically polished up to 3 µm and subjected laser irradiation by using a ArF laser with wavelength 193 nm by masking the surface for achieving textures of linear and dimple geometry. Linear texturing was developed at a laser power density of 2.4 J/cm², frequency of 200 Hz and 50 numbers of pulses. On the other hand, dimple texture was developed using a laser power density of 3.2 J/cm², frequency of 200 Hz and 100 numbers of pulse. Followed by texturing, the surface topography of texturing was measure by laser scanning profilometer using 10 nW He-Ne laser. The microstructures of the top surface of the textured surface were characterized by a field emission scanning electron microscopic (SUPRA 40, Zeiss SMT AG, Germany) coupled with energy dispersive X-ray (EDX) microanalyser and compositional analysis was carried out using energy dispersive spectroscopic analysis. The phases present on the treated surface were analyzed by glancing incidence angle X-ray diffraction (GIXD) (Philips X'Pert PRO Diffractometer, PANalytical, Almelo, The Netherlands) technique operated at accelerating voltage of 40 kV and current equal to 30 mA and at an incidence angle of 1° using Cu-Kα radiation. Average lattice strain developed in the textured surface was measured from the analysis of peak broadening using Scherrer's formula[14]. Residual stress developed on the surface was carefully measured by X-ray diffraction technique using a stress Goniometer for macro-stress (PW 3040/60, Panalytical X'pert Pro, Netherland) by using sin² w method [14]. Quantitative analysis of phases was done from the

integrated intensity of X-ray peaks using normalized relative (integrated) intensity ratio (RIR) method [15]. The hardness and young modulus inside the textured zone and outer area were evaluated by using nano-indentation (Model-Tl950 Tribo-indenter _{TM}) using triangular pyramid (Berkovich) diamond indenter at an applied load 6 mN. The value of hardness and young modulus was calculated from load – displacement graph by using Oliver and Pharr method [16]. The wettability of textured and as-received Ti-6Al-4V against simulated body fluid was evaluated by measuring the contact angle using sessile drop technique [17].

3. Results and Discussions

Figures 1(a-d) show the scanning electron micrographs of the top surface of laser surface textured Ti-6Al-4V alloy lased with ArF laser (wave length 193 nm) in air with (a) linear geometry (lased at an applied energy density 2.4 J/cm², frequency of 200 Hz with 50 numbers of passes) (b) the same at high magnification, (c) with a dimple geometry (lased with an applied energy density 3.2 J/cm², frequency of 200Hz with 100 numbers of passes) (d) the same at high magnification. Figure 1(a) reveals that presence of periodic linear texture with a width of 25 2m and depth of 8 2m at an interval of 20 2m. Figure 1(c) reveals the presence of dimple texture of average diameter 60 2m and depth 8 2m. Both the Figures show the presence of the texture in a uniform fashion. The high magnification views of the textured surface (cf. Figure 1(b,d)) show similar features with the presence of fine grained 2-Ti (labeled as 1), 2-Ti (labeled as 2) and oxides (labeled as 3). There was no signature of 2' Ti in the microstructure, which is usually observed due to laser melting of titanium [6]. Hence, it may be confirmed that there was not melting of the surface due to laser texturing. Presence of oxides is due to reaction of ablated titanium with oxygen during/after laser processing. From the microstructure of textured region it is also evident that there are presence of very fine ablated particles on the grain boundaries, attreibuted to ablation assisted deposition process. The area fractions of linear texture and dimple texture measured by image analysis software were 47 % and 16 %, respectively. The average roughness of the polished substrate was 0.4 2m. The average surface roughness due to linear and dimple texturing were increased to 2.722m and 3.5 2m, respectively. The increased surface roughness due to texturing is attributed to ablation assisted roughening. Hence, it may be concluded that in the present study there is also roughening of surface in addition to modification of surface topography due to laser surface texturing. Furthermore, due to introduction of linear grooves and dimples there is also increase in actual surface area, which is beneficial in enhancing important properties like wettability and biocompatibility and reported elsewhere.

Figure 2 shows the X-ray diffraction profiles of (a) as-received, (b) linear textured and (c) dimple textured surface determined by point X-ray diffraction study. From Figure 2(a) it is relevant that as received Ti-6Al-4V contains both $\mbox{\ensuremath{\mathbb P}-Ti}$ and $\mbox{\ensuremath{\mathbb P}-Ti}$ phase. Surface texturing with both linear and dimple morphology caused formation of $\mbox{\ensuremath{\mathbb P}-Ti}$, $\mbox{\ensuremath{\mathbb P}-Ti}$ and presence of oxide phases (TiO $_{\mbox{\ensuremath{\mathbb P}}}$ (predominantly rutile with one anatase peak) and few Ti₂O₃) due to surface oxidation as texturing was carried out in air. Presence of oxides is beneficial in enhancing both the wear resistance and bio-compatibility [18]. In addition to the presence few oxides peaks, there was a significant broadening of the peaks in both line and dimple textured surface which is attributed to introduction of lattice strain and refinement of the grain size. It may be noted that lattice strain was marginally increased in linear textured zone (from 0.27 for as-received to 0.33 in textured surfaces). However, in dimple textured geometry the change in lattice strain was insignificant. The increase in lattice strain in linear textured surface is possibly attributed to introduction of lattice defect during texturing which

is predominately ablation based. However for sample with dimple morphology change in lattice strain is insignificant possibly due to reheating phenomenon during formation of textured morphology.

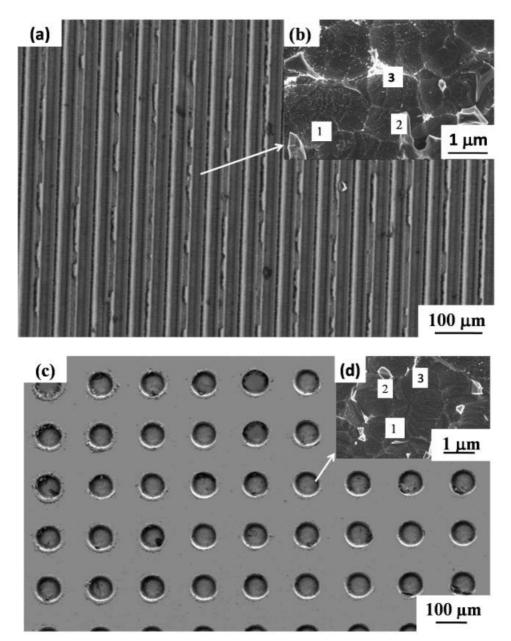


Fig. 1 Scanning electron micrograph of top surface of Ti-6Al-4V with (a)linear geometry (b) high magnification view of textured zone, (c) dimple geometry and (d) high magnification view of textured zone.

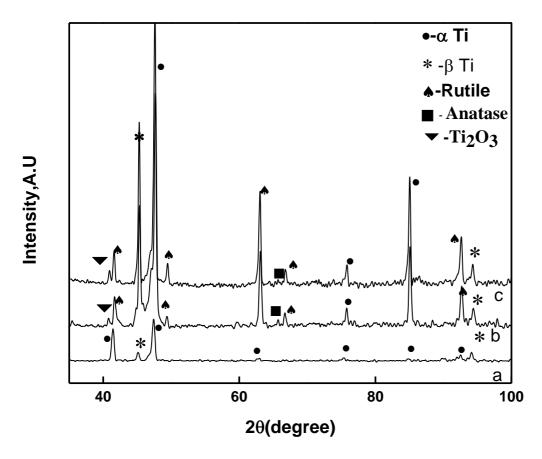


Fig. 2 X-ray diffraction profiles of the top surface of Ti-6Al-4V in (a) as received condition and the same with (b) linear texture (c) dimple texture.

The residual stress introduced during texturing was evaluated by stress goniometer. It is observed that as received Ti-6Al-4V sample showed a high amount of residual compressive stress (-130 MPa) which is possibly due to cold rolling. On the other hand, on the surface of linear texture, magnitude of compressive stress decreases which is mainly due to annealing effect during laser irradiation (-78 MPa). Furthermore, for the sample with dimple texture, the residual stress is very low (36 MPa) and tensile in nature. The presence of tensile residual stress is attributed to a very high rate of cooling followed by laser irradiation of the ablated surface. Robinson et al. [19] found the generation of tensile residual stress in laser melting process.

Hardness developed in texture zone along with its Young's modulous were evaluated using nano-indentation technique by application of triangular pyramid berkovich indenter. The displacement as function of load was derived from the depth sensing indenter system which reflects loading unloading history of the sample. Figure 3 illustrate typical load vs displacement curve of substrate (plot-1), linear textured surface (plot-2) and dimple (plot 3) indented with a maximum load of 6000 \mathbb{R} N. The area inside the loading unloading curve loop of the loading and unloading curve represents the energy dissipated from the surface due to plastic deformation. The corresponding hardness and young modulus values were derived from load displacement curve using Oliver Pharr method [16]. From Figure 3 it may be noted that there are shifting of the load

displacement curve to the left for the textured surface as compared to the as received Ti-6Al-4V. Shifting of the curve to the left indicates about compressive residual stress arrested due to texturing. The degree of shifting was not however, varied with the morphology of texturing. It was observed that there is a significant improvement in hardness in the dimple and linear textured zone to a value of 6 GPA and 4 GPa, respectively as compared to 2 GPa of as-received Ti-6Al-4V. The significant improvement in hardness along the textured zone is attributed to microstructural refinement and the presence of oxide phases in the textured zone. The improved hardnness of the textured zone is beneficial for improving the wear resistance property of the surface. However, no significant change in Young's modulus was observed due to texturing.

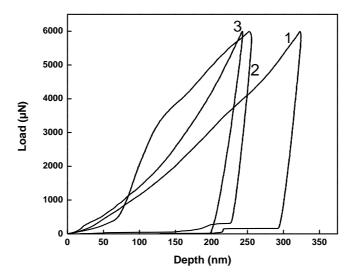


Fig. 3 Load vs. Displacement curve of as received (plot 1) and textured region with (plot 2) linear texture and (plot 3) dimple texture Ti-6Al-4V determined by nano-indentation technique by application of triangular pyramid berkovich indenter with a maximum load of 6000 $\[mathbb{D}$ m.

A detailed study of the contact angle of the simulated body fluid on textured surface showed that there is a decrease in contact angle to 55°-57° as compared to 60° in as-received surface. The decrease in contact angle on textured surface is attributed to change in surface composition and degree of roughness due to laser texturing and is advantageous in enhancing the cell attachment on the surface.

4. Summary and Conclusion

From the above mentioned investigation it may be concluded that laser surface texturing of Ti-6Al-4V with a linear and dimple morphology was conducted with UV laser of wavelength (193 nm) to form a defect free and periodic textured patterns. The microstructure of textured region consist refined grains of a-Ti with presence of \mathbb{Z} -Ti particles on the grain boundary. X-ray diffraction analysis showed the presence of \mathbb{Z} -Ti, \mathbb{Z} -Ti, Rutile, Anatase and Ti₂O₃ phase on the textured region. A significant improvement in nano-hardness was

achieved due to laser texturing with a maximum improvement for dimple texturing (6 GPa) with no significant change in Young's modulus. Hence, it may be concluded that the proposed laser textured of Ti–6Al–4V is successful for enhancing wettability and wear resistance for its application as bio-implant.

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