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Laser Surface Nitriding of Ti-6Al-4V for Bio-implant Application

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The present study aims at enhancing the biocompatibility of Ti-6Al-4V by laser surface nitriding. Laser surface nitriding has been carried out by melting of sand blasted Ti-6Al-4V substrate using a high power continuous wave DIODE laser with nitrogen as shrouding environment (at a pressure of 5 l/min). Following laser treatment, a detailed characterization of the surface has been conducted. Microhardness and biocompatibility have been evaluated. Laser surface nitriding led to formation of dendrites of TiN on the surface. The microhardness is improved to 900-950 VHN (in laser surface nitriding) as compared to 260 VHN of as-received substrate. Biocompatibility behavior showed a better cell viability in laser surface nitrided Ti-6Al-4V sample as compared to as-received one.

Introduction

Titanium and its alloys are widely used as surgical implants because of their good corrosion resistance, high specific strength and biocompatibility [1-3]. However, the applications of pure titanium are sometimes limited due to its low surface hardness and poor wear resistance. Furthermore, the loss of adhesion at the interface was found to be caused by the existence of a layer of porous titanium oxide [4]. Various types of surface modification techniques have been developed aiming at improving the interfacial bonding of the alloy and the bone [5-8]. Laser as a source of monochromatic and coherent radiation has a wide ranging applications in materials processing [9]. Laser surface remelting of titanium in nitrogen containing environment is popularly known as laser gas nitriding (LGN) [10]. The advantages of laser assisted surface treatment over conventional diffusion aided surface treatment include ability to deliver a large power/ energy density (103 to 105 W/cm^2), high heating/ cooling rate (103 to 105 K/s) and solidification velocities (1-30 m/s) [9]. Laser gas nitriding of Ti and Ti-6Al-4V (Ti64) alloys has been extensively investigated with the aim of improving the tribological properties [11,12]. However, presence of microcracks was a commonly encountered problem associated with laser gas nitriding of titanium alloys [13]. In this regard, it is relevant to mention that it is essential to optimize the laser and process parameters to avoid the above mentioned limitations and form a defect free and continuous surface layer [9].

In the present study, an attempt has been made to do laser surface nitriding of Ti-6Al-4V substrate using a laserline DIODE laser. Laser parameters have been selectively chosen to avoid formation of micro-cracks in the laser nitrided surface. Effect of laser parameters on the microstructure, phases and microhardness has been studied in details. Finally, the biocompatibility of the nitrided surfaces has been compared with that of as-received Ti-6Al-4V.

Experimental

In the present investigation, Ti-6Al-4V (Ti64) of dimension: 20 mm x 20 mm x 5 mm was chosen as substrate material. The samples were sand blasted prior to laser processing in order to remove oxide scale from the surface. Laser surface nitriding was carried out by irradiating the substrate using a 2 kW continuous wave (CW) DIODE laser with a beam diameter of 2 mm with nitrogen as shrouding gas. The specimens were mounted on a CNC controlled X-Y stage which was moved at a speed of 1-5 mm/s. To achieve microstructural and compositional homogeneity of the laser treated surface, a 25 % overlap between the successive melt tracks was followed. A large number of trials were undertaken using a wide range of laser power and scan speed combination to see the effect of laser parameters on the characteristics of composite layer. A defect free nitride layer was found to form when lased with a power ranging from 600 - 800 W, scan speed of 6 mm/s at a gas flow rate of 5 l/min. Following laser surface nitriding, the microstructure of the nitrided layer (both the top surface and the cross section) was characterized by optical and scanning electron microscopy. A detailed analysis of the phase was carried out by X-ray diffractometer. The microhardness of the composite layer (both at the top surface and along the cross sectional plane) was measured by a Vickers microhardness tester using a 30 g applied load. For Biocompatibility study, as-received and laser surface nitrided (lased with a power of 700 W) samples were carefully polished and autoclaved. 4 ml of 105 cells of L-929 (mouse fibroblast cell line) were transferred in a 20 mm petri plate containing the specimen and incubated in carbon-dioxide chamber containing 5% CO_2 at 37^0C in a humidified chamber. The cells were left to adhere for 72 hours with proper control (polystyrene petriplate). MTT assay (reduction of 3-(4,5-dimethylthiazol-2-yl) -2,5-diphenyl-tetrazolium-bromide to a purple Formazan product) was used to estimate cell viability and proliferation by using plate reader at 595 nm.

Results and Discussions

In the present section, a detailed characterization of microstructure and phases of the laser surface nitrided Ti-6Al-4V was undertaken to observe the effect of process parameters on the characteristics of the modified layer. In addition, the mechanical and biocompatibility properties of the surface nitrided layer were evaluated and compared with as-received Ti-6Al-4V. The results of the characteristics and mechanical/electro-chemical properties of the composite layer are discussed in details.

Characteristics of nitrided zone

Figure 1 shows the scanning electron micrograph of the top surface of as received Ti-6Al-4V substrate used in the present study. The microstructure consists of elongated α-Ti and presence of lamellae of β-Ti at the inter-granular region. From the as-received microstructure it is revealed that substrate used in the present study is cold rolled Ti-6Al-4V. The average grain size of the substrate was found to vary from 15μm to 25μm. Figure 2 shows the scanning electron micrograph of laser surface nitrided Ti-6Al-4V lased with a power of 700 W. Laser surface nitriding caused formation of dendrites of titanium nitride in α-Ti matrix. The dendrites were highly refined with an average primary arm spacing of 3-15 μm. The interdendritic arm spacing of the nitrides was found to vary with laser parameters [14].

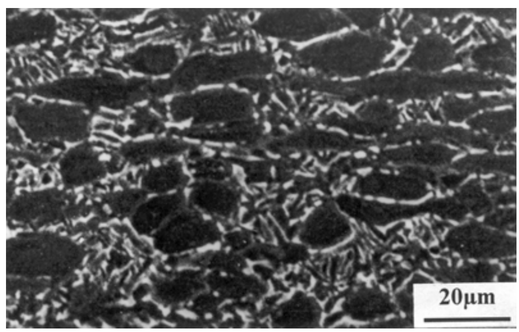

Figure 1: Scanning electron micrograph (SEI) of as received Ti-6Al-4V

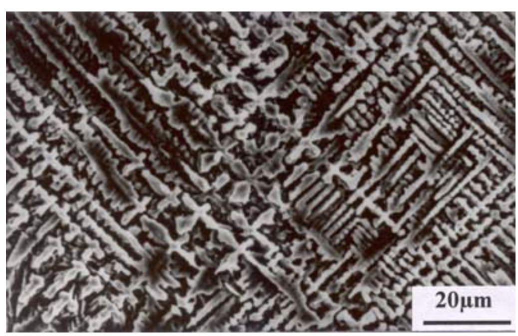

Figure 2: Scanning electron micrograph (SEI) of the top surface of laser nitrided Ti-6Al-4V lased with a power of 700 W.

Figure 3 shows the effect of applied power on the thickness of nitrided layer for laser surface nitrided Ti-6Al-4V. From Figure 3 it is relevant to mention that the depth of nitrided layer varies from 600 to 1200 μm and increases with increasing applied

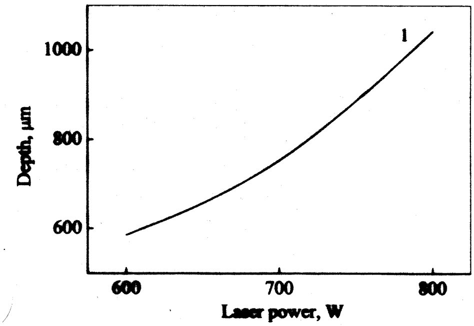

Figure 3: Variation of depth of nitriding with applied laser power.

power. Increased depth of nitriding with increasing applied power is attributed to a higher level of adsorbed energy.

Figures 4 (a,b) show the X-ray diffraction profiles of (a) as received and (b) laser surface nitrided (with a power of 600W) Ti-6Al-4V. From the X-ray diffraction profiles it is relevant that as received Ti-6Al-4V used in the presence study consists of a mixture of a and β-Ti. The relative volume fraction of β-Ti is however lower than that of α-Ti. Laser surface nitriding, on the other hand, leads to formation of mainly TiN though a few α-Ti peaks were also observed in the X-ray diffraction profiles.

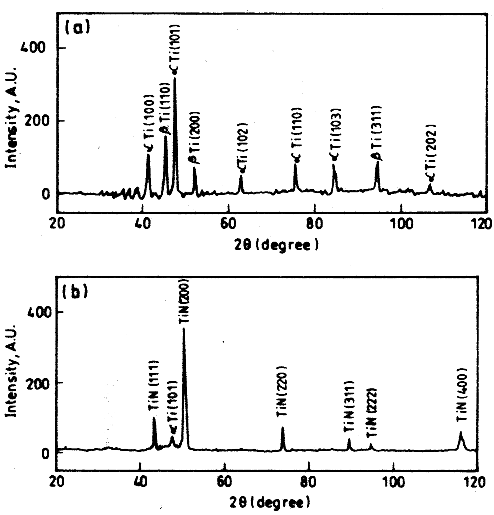

Figure 4: X-ray diffraction profile of (a) as received, (b) laser surface nitrided (with a power 600W) Ti-6Al-4V.

Properties of the nitrided layer

Microhardness of the top surface and cross section of the laser surface nitrided Ti-6Al-4V was carefully measured to observe the influence of laser surface nitriding on microhardness and its distribution.

Figure 5 shows the variation of microhardness with depth from the surface of laser surface nitrided Ti-6Al-4V lased under different processing conditions. Laser surface nitriding is found to improve the microhardness of the melted region from 260 VHN of as received surface to a maximum of 900 VHN in surface nitrided sample. The microhardness is maximum at the surface, remains almost uniform along the nitrided layer and gradually decreases to the substrate microhardness at the nitrided layer-substrate interface. The average microhardness of nitrided layer is found to vary with laser parameters. The maximum microhardness at the top surface is beneficial for improving the wear resistance of the surface.

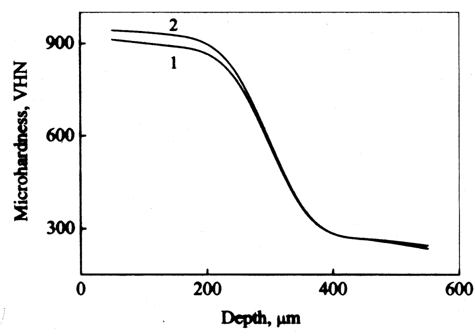

Figure 5: Variation of microhardness with depth from the surface of laser surface nitrided Ti-6Al-4V with a power of 800W (plot 1) and 600W (plot 2) respectively

Biocompatibility study

Cell proliferation on each specimen was measure by MTT assay. Cell attachment is expressed in terms of percentage of adhere cells with respect to the positive control (Petri plate). Figure 6 shows the MTT results of the cell proliferation count after 72 hours of cultivation. The number of cells in as received and laser surface nitrated exceeds the positive control (100 %) as shown in the Table 1. It is observed that cell proliferation/viability is significantly improved by laser surface nitriding of Ti-6Al-4V.

Table 1: Summary of the biocompatibility behaviour of as-received and laser surface nitrided Ti-6Al-4V

Sl.No.	Sample	MTT reading O.D. at 595 nm	Percentage of increase in cell number
1	Control (plastic surface)	0.734	100
2	As received Ti-6Al-4V	1.614	219.8
3	Laser surface nitrided Ti-6Al-4V	2.295	312

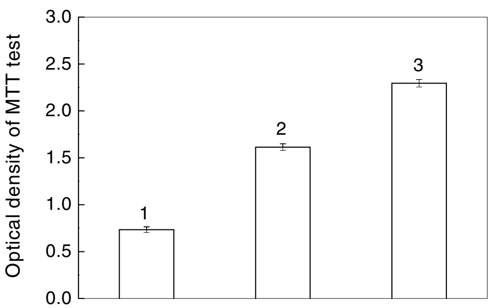

Figure 6: Optical density (at 595 nm) of MTT cell count measured after 72 hours for (1) control (Petri plate), (2) as received and (3) laser surface nitrided Ti-6Al-4V.

Summary And Conclusions

In the present study, an attempt has been made to modify the surface of Ti-6Al-4V substrate for bio-implant application. Laser surface nitriding has been carried out using a laser-line Diode laser. From the results the following conclusions may be drawn:

1. Laser surface nitriding caused formation of TiN dendrites in a-Ti matrix.
2. Microhardness of the surface nitrided layer was improved (to a maximum of 900 VHN) as compared to 260 VHN of as-received substrate. Microhardness was found to be uniform all throughout the nitrided layer.
3. Biocompatibility was found to improve significantly due to laser surface nitriding.

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