Laser surface modification of Ti implants to improve osseointegration

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Abstract. Commercially Pure Titanium foils, were irradiated using a pulsed Nd:YAG laser under ambient air, in order to produce and characterize a well controlled surface texture (roughness and waviness) that enhances osseointegration. To study the “peri-implant” reparative process response, the laser treated Ti foils were implanted in the tibia of 10 male Wistar rats. At 14 days post-implantation, the histological analysis showed a tendency to more bone formation compared to the untreated control implants. The formation of a layer of TiN on the surface and the obtained roughness, have been demonstrated to improve bone response.

1. Introduction
Titanium dental implants are widely employed due to their good performance and predictable lifetime. The long-term benefits of such implants are caused by the responses of different surrounding tissues. These take place on the bone–implant interface. However, the improvement of this interface is still an open problem. Since the main target is to enhance the development of strong bonds, ceramic and biological coatings have been widely investigated as a means to improve metal osseointegration (intimate apposition of bone to biomaterial). In this regard sandblasting, plasma spraying and acid etching have become the three most common approaches used to alter the surface topography and increase the surface area of implants [1-2 and references therein].

But the main problem of surface treatment is the contamination of the surface during the roughening procedure. Using laser techniques for roughening the implants surface, contamination is avoided, because the laser enables implant surface treatment without direct contact, and an easier control of the micro-topography is achieved [4-5].

The aim of the present study is to produce and characterize a well controlled surface texture (roughness and waviness) on Titanium implants to promote osseointegration. In previous works, a systematic study of the influence of laser parameters on Carbon Steels and Ti was performed, particularly the influence of energy density, number of laser pulses and background atmosphere [6]. For this report, we selected from those studies, those parameters that seemed suitable for the present application but varying the number of laser pulses per site.

2. Experimental details
Commercially Pure Titanium foils (5 x 1 mm², 0.1mm thick), located on a X-Y motorised stage, were irradiated using a pulsed Nd:YAG laser, operating at 355 nm under air atmosphere. Before laser
treatment, all samples were cleaned ultrasonically in acetone, and then rinsed in isopropyl alcohol. The laser pulses hit the surface in a 5 mm$^2$ area with local average fluence of 2.5 J/cm$^2$. The scanning speed of the motorized stage was synchronised to the repetition rate of the laser, resulting in 200 and 500 laser shots per site. Samples with 200 and 500 pulses will be named T01 and T02 respectively.

Scanning electron microscopy (SEM) analyses of the sample surfaces were performed before and after implantation to record morphological details. Surface analysis of the samples after laser treatment was conducted using Atomic Force Microscopy (AFM) to determine micro-roughness profiles and topography. In order to compare the crystalline structures of the laser-treated samples with those of the non-irradiated materials, XRD spectra were recorded at grazing angle, using Cu K$_{\alpha}$ radiation ($\lambda = 0.154$ nm).

To study the “peri-implant” reparative process response, 10 male Wistar rats, 90g body weight, were used. The animals were fed ad libitum. The guidelines of the National Institute of Health (NIH) for the use and care of laboratory animals were observed (NIH Publication No 85-23, Rev. 1985).

Surgical procedure: The animals were anesthetized intraperitoneally with a solution of 8 mg of ketamine chlorhydrate (Fort Dodge® - Argentina) and 1.28 mg of xylazine (Bayer Argentina S.A.) per 100g body weight. The skin of both tibiae was shaved prior to performing a 1.5 cm incision along the tibial crest. The subcutaneous tissue, muscles and ligaments were dissected to expose the lateral external surface of the diaphyseal bone. An end-cutting bur was used to drill a hole of 1.5 mm in diameter with manual rotating movements to avoid overheating and necrosis of the bone tissue [3]. A laser treated Ti laminar implant (T01) was placed in the hematopoietic bone marrow compartment of the right tibia. A laser treated Ti laminar implant (T02) was placed in the left tibia. The reverse side of each treated laminar implant was used as control. No antibiotic therapy was administered. At 14 days post-implantation the animals were sacrificed by ether overdose, the tibiae were resected, fixed in formalin and radiographed.

Histological processing: The tibiae were processed for embedding in methyl methacrylate, and sectioned with a saw at the level of the metal implant to obtain 3 cross-sections transversal to the central long axis of the tibia. The sections were ground with a polishing machine and completed manually with sandpaper. Fifty-micrometer sections were stained with 1% toluidine blue.

3. Results and Discussion

3.1 Surface characterization

Figure 1 shows the grazing angle X-ray diffraction patterns of laser treated c.p. Ti foils in air atmosphere with 200 and 500 pulses per site. It can be seen that besides metallic Ti reflections, marked with asterisks, TiN peaks also appear. During laser surface melting under air atmosphere, the nitrogen atoms dissolve into the high temperature Ti melt, resulting in alloying of the melt-pool with nitrogen. However, no signals of Ti oxides were detected within the accuracy of the technique, in spite of the ambient oxygen presence. The TiN peaks are shifted to lower 2$\theta$ values, which is an indication of compressive stress being built up in the coating after surface melting and alloying.

Due to the difference in thermal coefficients between pure Ti and TiN, some cracks could be observed on the surfaces that were absent when the treatments were performed under Ar atmosphere.

The AFM surface topography pictures allow a quantitative analysis of the surface roughness, as shown in figure 2.
The surface roughness of the laser-irradiated Ti foils with 200 laser pulses and with a fluence of 2.5 J/cm² resulted $R_{p-p}$ (peak to peak) = 650 +/- 150 nm and and $R_a$ (average) = 130 nm, while for the 500 pulses irradiated samples, $R_{p-p}$ = 1250 +/- 160 and $R_a = 180$nm. The most important difference between the 200 and 500 pulses treatments is the spatial distribution of peaks and valleys. The T02 (500 p/site) treatment gives average distances between peaks and valleys of about 40 nm, while for T01 these are about 200 nm. The importance or not of such difference is evaluated at the light of the biological tests results.

3.2 Biological Tests

None of the animals showed alterations in body weight, behavior or general health. In all cases the histological analysis of the sections evidenced the presence of lamellar bone around the metallic implants. No macrophages or related inflammatory cells were observed in the interface regions.
Figure 3 shows three cross-sections transversal to the central long axis of the tibia. From all the photos that were taken, we choose for control surface the one that presents more bone tissue formation in order to demonstrate that even in the case of an acceptable bone tissue amount, the interlocking with Ti surface is deficient. In group T01 the surface of metal implants was irregular with uneven and indented edges. The presence of important areas of soft tissue without osseointegration was observed compared to T02. In some cases metal particles were found in the peri-implant interface. In group T02 regular implant surfaces and substantial areas of bone in close contact with the implant (osseointegration) were observed compared to control samples and group T01. A tendency to more bone formation was also found in group T02 compared to T01.

![Figure 3. Histological sections showing the interface between control and experimental groups (T01 and T02). Orig. Mag. X100. I: implant B: Bone OI: Osseointegration](image)

4. Conclusions
Laser irradiation has here been demonstrated to be a suitable, clean and easy method to improve bone response. A tendency to more bone formation was found for the laser treated implants compared to control implants. It can be due to the formation of TiN on the surface that improves biocompatibility.

On the other hand, the T02 treatment showed a more beneficial response and more areas of bone interlocking compared to T01. Due to the high difference in topography between T01 and T02 surfaces, it indicates the importance of both spatial and height dimension of surface roughness for implant incorporation. Further investigations are needed to find optimal structure on Ti implants.

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