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Multi-scale characterization of topographic modifications on metallic biomaterials induced by nanosecond Nd:YVO_4 laser structuring☆

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ABSTRACT

Laser surface structuring of biometals allows to achieve complex surface geometries at several scales since a rough surface is preferred to induce an optimal cellular response. A lack of standard measurement techniques to characterize implant surfaces was detected. This paper presents a comprehensive, multi-scale characterization of the topography of laser textured biometals with the ISO standard profile method; which allows to separate the contribution of the superimposed textures of the profile, the low frequency component (waviness) and the high frequency component (roughness), and to calculate the respective height parameters according to the standard definitions. Furthermore, the characterization was completed by the calculation of the spatial parameters obtained by FFT calculation in order to assess the horizontal characteristics of the surface. We focused on two common biometals, stainless steel 316L and titanium alloy Ti6Al4V, structured with an array of parallel grooves obtained by using a nanosecond Nd:YVO_4 laser. Regarding waviness, some differences in symmetry and sharpness were found according to the metal. Conversely, the roughness of the topography revealed that the average height of the micro-structures developed inside the grooves is similar in both metals. The spatial periodicity of the micro-structures showed that the aspect ratio resulted higher in AISI 316L than in Ti6Al4V. The analytical approach presented in this paper allows to establish an elementary comparison of the results obtained in the scientific community, contributing to a better understanding of *in vitro/vivo* cellular growing on textured surfaces.

1. Introduction

Laser structuring of a surface consists in making geometrical structures through ablation processes, in order to change the surface relief on a micro-metric or even nano-metric scale to meet a specific functional requirement. In the biomedical field, it has been applied to metallic bio-implants with the objective of controlling cell responses and promoting the bone integration of the implant [1]; in this sense, texturing with grooves or ridges and dimples increases the surface area and provides more opportunities for cell attachment [2]. Moreover, linear patterned surfaces can induce cells alignment, a phenomenon known as contact guidance, which reduces the extent of scar tissue formation and promotes osseointegration [3].

Laser structuring is an advantageous and flexible tool to process implants [4] due to the possibility of texturing complex surface geometries with high flexibility in the design of features at micro and nanoscale. In this sense, it has been demonstrated that, in the case of textured samples presenting multi-scale features, the resulting material interaction is affected by both the overall roughness or large scale morphology and the detailed features on all length scales [5].

Different approaches can be found in the literature to establish a classification of osseointegrated implant surfaces, which require both chemical and physical characterization [6]. Nevertheless, a thorough review shows a lack of comprehensive surface texture analysis protocols to lead to the design of an optimal implant surface [1]; in this sense, to the best of our knowledge, there are no standard measurement techniques or a standard set of surface texture parameters broadly employed in the characterization of implant surfaces [7]. This inconsistency in the methodology used to characterize and quantify the various scales of surface topography hinders the comparison of results between multiple studies [1].

In order to deal with surfaces which present stratified functional properties, ISO issued the three-part standard 13565. This approach is based on the extraction of profiles from the surface by filtering strategies; these profiles correspond to different texture scales. To understand a complex surface texture, firstly, the gross form should be removed and

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then the low frequency components (waviness, *W*) and the high frequency components (roughness, *R*) have to be separated [8]. Additionally, the implementation of spacing parameters, which assess the lateral or horizontal characteristics of a surface, can provide valuable insight into the overall surface texture. It has been common across many industries to handle these analytical approaches when assessing surface texture, furthermore, they have applied surface texture analysis based on ISO 25178-1:2016 standard [9–11]; however, this strategy is scarcely used within the biomedical implant literature and, frequently, only simple roughness parameters are used, which reduce the entire range of vertical hierarchical structure to a single value.

In previous works, we had studied the influence of laser processing parameters on the structuring of stainless steel 316L and titanium alloy Ti6Al4V, by means of a 355 nm nanosecond Nd:YVO_4 laser source. Different periodic patterns in a suitable feature range size for bioimplant applications were analysed [12,13]. The present work is focused on the multi-scale characterization of the topography of these laser textured biometals. The aim of this work is to present a more comprehensive approach to assess multi-scale topography of textured surfaces. This approach is based on the profile method, which allows to separate the contribution of the superimposed textures of the profile and to calculate the respective parameters, according to the standard definitions [14,15], and the spatial periodicity of the microstructures developed at the finest scale, in order to achieve a more complete 3D characterization of the structured surfaces.

2. Materials and methods

Experiments were performed on samples of 20 mm × 20 mm of commercially available metal sheets of stainless steel AISI 316L and titanium alloy Ti6Al4V (hereunder *SS* and *Ti*64). Metal surfaces were textured with a pattern of parallel grooves by using a nanosecond Nd:YVO_4 laser at the wavelength of 355 nm (Coherent AVIA Ultra 355–2000), as previously described [13]. Processing parameters were adjusted to attain feature size of grooves in the suitable range for biomedical applications (around 25 μm width and 10 μm depth). In brief, the parameters employed were as follows: a laser pulse rate of 10 kHz, a scan speed of 25 mm s^{-}1 and a separation between adjacent laser trajectories of 50 μm.

After laser processing, the samples were analysed by scanning electron microscopy SEM (JEOL JSM-6400) to obtain a qualitative characterization of the surface. Topographical data were acquired with an optical imaging profiler (Sensofar® PLu 2300) and different objectives were used: a 20× EPI objective, with a field of view of 637 μm × 477 μm and a pixel size of 0.83 μm, and a 100× EPI objective with a field of view of 124 μm × 92 μm and a pixel size of 0.17 μm. Data processing, analysis and visualization were implemented by using MATLAB®.

3. Surface topography characterization

Images provided by SEM of both *SS* and *Ti*64 samples textured with a pattern of parallel grooves show the characteristic features of photothermal ablation, and allow us to appreciate different scales in the topography [13]. In this sense, transverse cuts (Fig. 1a) depict the wavy profile of valleys and peaks, which constitutes the texture at the larger scale. At a higher magnification, (Fig. 1b), top views show the distinctive pillar micro-structures, with a size of 34 μm, developed on the inner walls of the grooves, which conform the fine scale texture. These pillar structures are oriented along the groove, in the laser scan direction and, for a fixed set of laser processing parameters, their shape and size depend on the material.

These features highlight that a complete characterization of the textured surfaces requires a multi-scale analysis, focused not only on the distribution of heights (out of the plane) but also on how the heights are distributed in the plane of the surface. Therefore, the approach can be developed in two steps: firstly, from height distribution maps and by applying filtering strategies, the standard parameters of waviness (large scale texture) and roughness (fine scale texture) are extracted according to the standard ISO profile method [14]; secondly, the spacing parameters of the fine scale texture can be obtained by a bi-dimensional Fourier Transform (FFT). It should be taken into account that, throughout this work, we will use the term roughness and waviness to refer, respectively, to the high frequency and low frequency components of the arbitrary profile of the surface treated with laser and, specially in the case of waviness, not in the traditional sense of the term, concerning the lack of uniformity after tool processing of the surface.

3.1. Large scale characterization: waviness parameters

In order to extract the low-frequency parameters of roughness, i.e. waviness, from topographic images obtained with the 20× EPI objective, the primary profile perpendicular to the main axis of the grooves (*x*-axis) was obtained, as it is shown in Fig. 2: regular topography of the *Ti*64 structured sample (Fig. 2a), and the transversal profile (Fig. 2b) are depicted; 3D representation of the topography is also included (Fig. 2c). Afterwards, the primary profile was filtered by using a digital Gaussian filter with a low cut-off λ_c = 0.008 mm and a high cut-off λ_f = 0.25 mm, in order to remove higher-order deviations and the form-errors [15,16].

Three topographic images at different points of the textured area were acquired, and the standard parameters of waviness were calculated by averaging ten equally spaced profiles per image. Fig. 3 depicts the different stages involved in this calculation.

3.2. Fine scale characterization: roughness parameters

Due to the limit of resolution of the optical profiler used it was not feasible to measure dimensions of the micro-structures, developed inside the grooves, by simply filtering the primary profile. In terms of signals, the equipment was not capable to measure accurately a low amplitude and high frequency component when it was superimposed to a high amplitude and low frequency signal; which corresponds to the grooves themselves. In order to overcome this difficulty, the analysis was focused on the bottom plane of the groove, and a primary profile along its main axis (*x* coordinate) was extracted.

Eight topographic images, acquired with the 100× EPI objective, were stitched to obtain an extended topography of 600 μm, according to the recommended evaluation length [14]. As in the case of waviness, standard parameters were calculated by averaging ten equally spaced profiles, once the off-set was removed. An example of the extended topography and the conditioning of the primary profile to obtain roughness parameters is depicted in Fig. 4.

3.3. Fine scale characterization: spacing parameters

As it was mentioned before, due to the preferential orientation of the pillar micro-structures, which conform the fine scale topography, the distribution of heights (z coordinate) in the plane $x-y$ is not random and thus the roughness parameters calculated along the *X*-coordinate are insufficient for a complete description of the texture. Further information can be obtained from the 2D Fourier Transform (FFT); therefore, the wavelength of the FFT gives the spatial periodicity $L(x, y)$ of the micro-structures along the longitudinal (L_x) and transversal (L_y) directions of the groove; the quotient (L_x/L_y) obtained from the 50% isoline (ellipse) in $k - space$, corresponds to the aspect ratio of these pillar structures.

Fig. 5 depicts an example of computation of the FFT; the zone of interest (Fig. 5a) was re-sampled to obtain a smoother distribution (Fig. 5b), and the heights from the $x-y$ plane were represented as isolines in a contour plot. The 50% isoline was used to calculate L_x/L_y