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A parametric study of laser interference surface patterning of dental zirconia: Effects of laser parameters on topography and surface quality

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ABSTRACT

Objective. The aim of this work is to generate micrometric linear patterns with different topography on dental grade zirconia by means of UV laser interference and to assess the quality of the produced surface, both in term of the geometry produced and of the surface damage induced in the material.

Methods. The third harmonic of a Q-switched Nd:YAG laser (355 nm, pulse duration of 10 ns and repetition rate of 1 Hz) was employed to pattern the surface of 3Y-TZP with micrometric-spaced lines. The resulting topography was characterized with White Light Interferometry and Scanning electron microscopy: pattern depth (H), amplitude roughness parameters (S_a , filtered- S_a), Fourier spatial analysis and collateral damages were related to laser fluence and number of pulses employed.

Results. With our experimental setup, line-patterning of zirconia surfaces can be achieved with periodicities comprised within 5 and 15 μm . Tuning laser parameters allows varying independently pattern depth, overall roughness and surface finish. Increasing both fluence and number of pulses allows producing deeper patterns (maximum achievable depth of 1 μm). However, increasing the number of pulses has a detrimental effect on the quality of the produced lines. Surface damage (intergranular cracking, open porosity and nano-droplets formation) can be generated, depending on laser parameters.

Significance. This work provides a parametric analysis of surface patterning by laser interference on 3Y-TZP. Best conditions in terms of quality of the produced pattern and minimum material damage are obtained for low number of pulses with high laser fluence. With the employed method we can produce zirconia materials with controlled topography that are expected to enhance biological response and mechanical performance of dental components.

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1. Introduction

Ceramic materials are commonly used for dental applications including veneering material for metal substructures, all-ceramic posts and cores, frameworks for crowns and bridge-works [1]. At the moment, there is an increasing interest in using ceramics also as materials for oral implants [2] as an alternative to titanium due to their higher resistance to bio-corrosion and superior esthetic properties [3,4]. Tetragonal Zirconia Polycrystal stabilized with 3% molar of Yttrium oxide (3Y-TZP) is considered an excellent material for all these applications because of its bio-stability, mechanical properties (good strength, hardness and high fracture toughness) and esthetic appearance in combination with porcelain enamel (white ivory color, close to natural teeth) [4,5]. Furthermore, it has been demonstrated that zirconia ceramic exhibits low plaque accumulation and displays similar bacterial binding properties to titanium [6].

Despite the aforementioned advantages, the suitability of 3Y-TZP to be used in the dental prosthetic field depends on the feasibility of functionalizing its surface to improve the biological response or the mechanical adhesion to other materials (like dental cements and resins) [7]. This should be carried out by techniques that do not compromise material's bulk properties and its long-term stability. Common strategies to achieve these goals are the modification of the roughness or the introduction of a controlled topography. For instance, micrometric silica reliefs on zirconia implant surfaces have been employed to study endothelial cells and fibroblast response in Ref. [8], where it was demonstrated that parallel grooved surfaces were able to control cells spatial distribution and to guide their growth. Also an increment in roughness has proved to be beneficial in the osseointegration of zirconia-based implants [9] as well as improving the adhesion to dental cements [10–12].

The most common physical surface modification methods available for ceramics are machine-aided approaches such as sandblasting [13], grinding [14], acid etching [15] or laser micromachining [16,17]. The last rises above the others as suggested by Holthaus et al. [18] in a comparative study of different techniques to pattern the surface of ceramics materials such as alumina, zirconia, silica and hydroxyapatite. They concluded that laser treatment processes are a suitable alternative to classical methods since they have the advantage of being fast and with a high control on the final desired topography in contrast with contact techniques, as mechanical micromachining or stamp transfer molding. Moreover, using conventional methods the fabrication of defined patterns smaller than 100 μm is still challenging due to the high hardness and brittleness [19] of ceramics.

Direct Laser Interference Patterning (DLIP) offers a fast and accurate alternative to introduce controlled topography at the micrometric and sub-micrometric scale [19]. In this technique a periodical intensity distribution is produced by beam interference on the surface of the material to be treated. Depending on the number of interfering laser beams and the optical setup, different geometries can be produced (lines or dots). Commonly, nanosecond, picosecond and femtosecond pulsed lasers are used in order to reach high energy density at the interference maxima position. This high peak power permits

to locally melt, vaporize or ablate the substrate to engrave the desired geometry [20]. Further details about the technique and the achievable patterns can be found in Ref. [21]. DLIP allows great precision and flexibility in the produced topography and is fast enough to modify large areas, especially if compared to other laser micromachining techniques that require the scanning of the beam through the surface by opto-mechanical methods [22]. DLIP has been successfully used to pattern surfaces of different materials: from metals [23–26] and ceramics [27–29] to polymers [30,31]. Most common applications are in the field of tribology and biomaterials: topographical, chemical and microstructural modifications induced by DLIP are exploited to modify surface wettability [23], to introduce texture [25] or to tune the interaction with biological species [30,31].

DLIP technique has been successfully applied to produce micrometric line-patterning onto Ytria-stabilized zirconia with neither significant collateral damage induced by the laser treatment [28] nor detrimental effect on mechanical properties [32]. However, there has not been any systematic study about the influence of laser parameters on the topographies produced with DLIP on 3Y-TZP. As demonstrated for other ceramic materials, changing laser parameters employed often results in different surface topographies [27]. Moreover, a detailed roughness analysis at different scales is essential to accurately describe the topography of the produced patterns, especially when dealing with biomedical applications [33].

The objective of this work is to correlate the morphology and the quality of the generated pattern to the laser parameters employed (fluence and number of pulses). The topography and roughness of the patterns are characterized by means of 2D- and 3D-amplitude parameters and spatial distribution analysis. Surface finish and quality are assessed in terms of collateral damages induced by laser treatment. Finally, the processability ranges are determined as a function of laser parameters.

2. Experimental

2.1. Material processing

Commercially available powder of Tetragonal Polycrystalline Zirconia stabilized with 3% molar Y_2O_3 (TZ3YSB-E, Tosoh Co.) was employed. The powder was isostatically pressed at 200 MPa in a cylindrical mold and subsequently sintered in an alumina tube furnace at 1450 °C for two hours (heating rate: 3 °C/min), as described in previous work [34]. The resulting rods (10 mm in diameter) were cut into discs of approximately 2 mm thickness. The surface of the discs was grinded and polished with diamond suspensions of decreasing particle size (30–6–3 μm) and with colloidal silica as a final step. The samples had a final density of $6.03 \pm 0.02 \text{ g/cm}^3$ (99.67% of theoretical density) and a grain size (intercept distance) of $0.31 \pm 0.08 \mu\text{m}$. The obtained material has biomedical grade, according to ISO 13356:2013 [35].

2.2. Laser treatment

A Q-switched Nd:YAG laser (Spectra Physics Quanta-Ray PRO210) with a fundamental wavelength of 1064 nm and an

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