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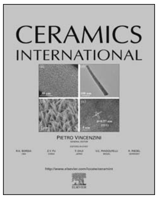

Effect of laser surface texturing on primary stability and surface properties of zirconia implants

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ABSTRACT

The aim of this study was to investigate the influence of different laser surface texturing parameters on static and dynamic coefficient of friction values of Yttria-tetragonal zirconia polycrystals (3Y-TZP) against bone to assess the primary stability of implants. The ability of the textures to promote osseointegration, as measured by wettability, and eventual changes in roughness, due to possible aging during sterilization, were also assessed. 3Y-TZP disks were divided into four groups: as sintered sample, sandblasting and etching treatment, laser irradiation using two output power of 0.9 and 1.8 W. The friction tests were carried out by using a pin-on-plate configuration, being the pins the 3Y-TZP disks and the plates the bone. The surfaces were inspected by SEM/EDS and by surface roughness profilometer. Wettability characteristics were also evaluated by drop contact angle, and aging was assessed, after laser treatments, by XRD analysis. Results demonstrate that with laser surface texturing of zirconia it is possible to combine better wettability, better aging performance, and better primary stability, as compared to traditional - Sand blasting and Etching treatments. Thus, it is shown that the laser irradiation technique is a promising alternative to conventional sandblasting and etching procedures.

1. Introduction

Yttria-stabilized tetragonal zirconia polycrystalline (Y-TZP) was introduced in the biomaterials field several years ago, mainly due to its combination of mechanical properties and aesthetic quality and durability [1,2]. Nowadays, zirconia bioceramics are often applied as implants in knee, bone screws, and dental applications [2]. Moreover, in vitro and in vivo studies also reported that the osseointegration capability of zirconia Y-TZP implants has been shown to be very similar to titanium implants, proving its suitability as an implant material [3,4]. This is possible notwithstanding the poor chemical connection with bone [5], since zirconia is a chemically inert material when introduced in human body [6].

To enhance zirconia surface properties, several surface treatments have been applied and the most common are etching, sandblasting, polishing and coating.

The conventional approach is performed by a sandblasting followed by an acid-etching, which is known to be the gold standard surface treatment [7]. It allows achieving micro-roughness values from 0.5 until 4 µm (Ra) [8]. However, the main disadvantage of this method is the potential undesirable chemical modification of the base material [9].

More recently, laser treatments have been proposed as a solution to modify the surface properties of biomaterials. This is an easily controllable and very flexible technique that promotes a rapid and oriented modification of the surface [10]. Although there are several attempts to modify zirconia surface and investigate its influence on biological response and mechanical properties, there is still little knowledge about the effects of surface modification on primary stability, as measured by static and dynamic friction response against bone. Moreover, the effect of sterilization (aging) on surface roughness and wettability is also scarce.

There are two important factors to ensure that the prosthesis mimics the mechanical and physical functions of a natural joint, which is important to mention: are the primary and secondary stability. The press-fit procedure during the total hip arthroplasty, for instance, guarantees that both the acetabulum cup and stem are anchored to the bone – referred to as primary stability – and the subsequent bone ingrowth insures the long-term bonding between the prosthesis and bone – referred to as secondary stability [11]. The integration between

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implant and bone is conducted by several events (e.g. surface topography and chemistry), which determines the performance of implant and take place largely at the tissue-implant interface. The development of this interface depends on interactions of bone matrix and osteoblast with the biomaterial. Thus, the improvement of initial attachment of osteoblasts precursor cells to implant surfaces plays an important role in the bone integration of the implant and longer-term stability [12].

Furthermore, protein adsorption and subsequent cell behaviour will depend on surface topography, wettability and electric charge [13,14]. The relation between the hydrophilicity of a material surface and cell adhesion has been widely investigated, and several works have reported that high surface wettability, which means high surface energy, promote higher cell adhesion in comparison to low surface energy [15,16]. Tarumi et al. [17] studied the relation between surface roughness and wettability of zirconia after various treatments (silicone wheel polishing treatment, sandblast treatment, tribochemical treatment), and reported that by increasing the surface roughness (obtained in the case of sandblasting treatment, tribochemical treatment) the water contact angle decreases thus increasing wettability.

Besides, in an implant design, other aspects have to be taken into accounts such as mechanical properties and tribological behaviour [18]. Tribological aspects are important, mainly during insertion of the prosthesis, which produces a friction pressure in the bone-implant interface. Primary stability of the implants is dependent on the friction between implant and bone, and in the case of screw type implants (screws, dental implants) depend mainly on the upper surface of the threads and the bone [11].

Studies concerning the tribological behaviour, in particular, friction coefficient of zirconia against bone is very scarce. Notwithstanding, there are a few studies on this field, but only Ti and its alloys, as well as other metallic materials, against bone, are included. Mischler and co-authors reported the tribological behaviour of Ti sliding against cow bone, in a lubricating medium and their results showed a severe wear of the bone, evidenced by the great amount of bone in the metal surface. The values of friction coefficient varied from 0.34 to 0.39 in their study [19]. Davim and co-authors studied the friction and wear behaviour of bovine cancellous bone against stainless steel, under water lubrication. They showed that the prevalent mechanism found was abrasion and their average friction coefficient value was 0.25 [20]. Dantas and co-authors studied the tribological performance of Ti6Al4V bioactive composites to evaluate the friction response and surface damage. Surface treatment was also used and their results showed that the static friction increased with the surface roughness (from 0.20 to 0.60), when compared to polished samples [11].

Finally, and regarding eventual surface topography changes, due to aging as a consequence of common sterilization processes, after laser surface treatments, there is also no information in the literature. Thus, in this study a preliminary analysis of the aging effect on surface properties, namely roughness, was performed.

It is worth to mention at this stage that the effect of laser surface treatments or textures in mechanical properties and in aging is out of the scope of this paper. Another paper is being prepared with detailed information on mechanical properties as a function of laser treatments and aging. However, it may be said at this moment that laser treatment, including texturing, is not necessarily detrimental in terms of mechanical properties.

The main aim of this study is to comprehend the friction behaviour on the implant-bone interface during the implant insertion in the bone and to evaluate the influence of surface properties of zirconia modified by laser treatment in comparison to the conventional treatment.

2. Experimental details

2.1. Preparation and surface treatment of zirconia disks

A commercial powder of 3% mol yttria stabilized zirconia – 3Y-TZP (TZ-3YB-E Tosoh, Japan) was used to produce disks with 14.5 mm radius by biaxial pressing under 200 MPa. Zirconia disks were sintered in air for 2 h at 1500 °C in a heating rate of 8 °C/min, resulting in a grain size of 0.42 ± 0.031 µm. A total of 12 disks were prepared.

The 3Y-TZP disks were divided into four groups according to a surface treatment:

(i) AS – as sintered sample;
(ii) SE - sandblasting and etching treatment. The 3Y-TZP disks were subjected to the grit-blasting procedure, using 100 µm alumina particles. The grit-blasting was carried out at a constant pressure of 6 bars, at a distance of 10 mm from the blasting nozzle and with an impact angle of 90° for 30 s. Then, the samples were immersed in hydrofluoridine acid (48%) for 30 min;
(iii) LI - laser irradiation using an output power of 0.9 W.
(iv) LII - laser irradiation using an output power of 1.8 W.

The samples were subjected to a laser treatment in order to produce a moderate and high roughness topography. For this, a Nd: YAG laser (OEM Plus 6 W, Italy) working in a fundamental wavelength of 1064 nm and a repetition rate of 20 kHz. was employed to carry out the laser treatments. The nominal focal length of the focusing lens was 160 mm. The surface samples were irradiated using two output laser power values, 0.9 W (LI) and 1.8 W (LII), with a scan speed of 15 mm/s at room temperature in air. To relieve the stresses caused by laser irradiation and recover the oxygen content to the zirconia surface, the laser irradiation groups (LI and LII) were subjected to a thermal treatment (in air) of 1200 °C for 1 h with a heating rate of 5 °C/min (named here LIT and LIIT).

2.2. Preparation of the plates (bone)

The plates used in this study were prepared from a fresh young femur of bovine (8 months). The bone was cut into rectangular samples (4×16×20). The samples were continuously kept wet with PBS [21,22] solution during sample preparation. After machining, the bone plates were covered with gauge immersed in PBS solution and kept in the freezer until the friction tests. Before the friction tests, the samples were defrosted.

2.3. Surface roughness measurements

After the surface treatments, the roughness was measured using a contact profilometer (Surftest SJ 201, Mitutoyo, Tokyo, Japan) at 2.5 mm measurement length and 0.25 mm/s. In the case of samples treated by laser, the profilometer ran the samples in the perpendicular direction in relation to the direction of the textures. Five measured were performed for each test sample. The measured surface roughness parameters were: (i) average roughness, R_a, (average obtained between peaks and valleys distance), (ii) skewness, *Rsk*, (the curve asymmetry in terms of frequency of valleys and peaks along to the profile, i.e. this parameter indicates the proportion between the number of peaks and valleys) and (iii) kurtosis, *Rku*, (the distortion of the curve regarding the normal distribution).

2.4. Autoclave procedure

The standard steam sterilization procedure, according to ISO 13356 (134 °C, under 2 bars pressure for 1 h) was performed after all the surface treatments. The monoclinic content was monitored after each treatment, including autoclave test, through XRD analysis with an incidence angle held in 2°, using Toraya modification of Garvie & Nicholson equation, considering the maximum intensities of peaks [23,24].