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The effect of different laser irradiation on rugometric and microtopographic features in zirconia ceramics: Study of surface statistical metrics

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The purpose of this in-vitro study is to evaluate and compare the effects of different laser irradiation routines to the surface morphology of zirconia-based ceramics. Sample prepared by a copy milling technique were irradiated using Nd-YAG, Er-YAG, and CO_2 infrared lasers. Surface topography of the samples was studied by means of AFM and SEM methods, and changes in their spatial characteristics were analyzed comparing statistical and fractal parameters. Depending on the absorption of incident energy, CO_2 laser was found to exert dominant influence on the surface geometry concerning changes in the surface texture, roughness and fractality of the specimens.

1. Introduction

In the recent decade, numerous biomedical applications of ceramics, metals, and polymers were found, especially in the field of dentistry. Good mechanical properties, excellent corrosion resistance, and high biocompatibility are only few examples of significant properties of metals such as Ti which makes them useful in biomedicine. However, there are some obstacles concerning biotoxicity, unsatisfactory wear resistance, and elastic modulus which recently motivated scientist to use ceramics instead of traditional alloys [1–3].

Crystalline zirconia is a well-known polymorph that takes one of three possible forms: monoclinic (M), which is stable from room temperature up to 1170 °C, tetragonal (T), which becomes stable from 1170 to 2370 °C, and cubic (C), which is favorable above 2370 °C [4–6].

In recent years, zirconia-based ceramics have gained large attention in dental industry and therapy due to their interesting properties. Unlike other ceramics, zirconia exhibits very high resistance to crack propagation, high thermal expansion and low thermal conductivity [7,8], which make it material of choice, for example, abutments for ceramic and steel. Apart from that, due to excellent wear properties and biocompatibility, zirconia is found suitable material for other dental prosthetic products that imitate that of natural tooth: crowns, bridges and veneers [9]. Unfortunately, zirconia suffers from age-related hydrolytic fatigue; hence it

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needs careful surface treatment for biomedical grade [10,11]. In addition, aging kinetics of zirconia induces detrimental microstructural and crystallographic phase changes that reduce the lifetime of dental restorations [12].

Laser processing is one of the important methods for fabrication of materials. There are advanced technologies such as selective laser melting (SLM) and developed additive manufacturing (AM), by which producing complex-shape materials become possible by using a high intensity laser beam [13,14]. Two significant benefits of SLM manufacturing techniques are high material utilization rate and high speed production [15]. Apart from that, however, the laser processing is also used for surface treatment. Recently, a number of surface modifications have been proposed to improve structural properties of ceramic surfaces: roughness, texture, morphology, and proliferation [16]. Among others, surface treatment with laser beams turned out to be one of the most effective routines in this respect. Since its development by Maiman in 1960, lasers have become multifunctional tools with a spectrum of applications in medicine and specifically in dentistry [17,18].

The use of solid state lasers, such as: Nd-YAG (neodymium yttrium-aluminum-garnet), and Er-YAG (erbium yttrium-aluminum-garnet), as well as gas lasers, such as carbon dioxide (CO_2), are active in the infrared range and opened new perspectives for engineering of surface finish of various materials [19,20]. For example, the effect of laser re-melting of a surface of alumina-covered steel substrate was found to improve mechanical and tribological properties of the coating [21], whereas laser irradiation of a steel specimen was found to suppress the development of fatigue cracks increasing the duty cycle of the machinery [22].

Nd:YAG lasers were found helpful in management of tooth hypersensitivity and bleaching [23,24]. On the other hand, Er:YAG lasers appeared suitable to remove caries, prepare cavities, and modify surfaces of ceramics [25]. Unlike them, CO_2 lasers are used in dentistry for soft tissue and periodontal surgery with significant postsurgical benefits [26]. Apart from that, an enhancement in mechanical and tribological properties of zirconia was observed upon irradiation with CO_2 laser beam. Petibon et al. reported their results on laser melting that caused surface densification, hence improved wear, corrosion and thermal shock resistances [27], while Lee et al. demonstrated that laser glazing changes phase composition of zirconia-based thermal barrier coatings associated with disappearance of a monoclinic phase, and increases the lifetime of that coating due to increased strain accommodation ability in laser-induced segmented cracks [28].

In addition, the effects of Er:YAG laser irradiation on the surface nanomorphology of human dental enamel, were studied using multifractal analysis and statistical functions [29,30].

Published research have undoubtedly demonstrated that the geometrical structure of the surfaces, and more precisely various aspects of the surface irregularities, play key role in determining the boundary phenomena concerning, for example, interaction dynamics, multiscale tribology, contact effects and others. The problem of bridging a gap between fundamental mechanisms behind observed changes and accurate descriptors of the surface morphology at different levels appears extremely important for biomedical applications. The roughness associated with the degree of the surface development affects osseointegration through modification of the true contact area and adhesion forces. Specific features of the surface topography, for example grooves, not only influence penetration area of the boundary, but also control the fluid retention and even drive desired cell reactions [31,32]. In turn, the surface treatment might change multiscale geometrical structure specific of most natural objects, which is way to tune the surface characteristics for better compatibility. In ceramics, specific surface-to-volume ratio and porosity expressed in terms of fractal measures are found to correlate with absorption of various species [33].

The main goal of this work was to study changes in zirconia surface texture caused by Nd-YAG, Er:YAG, and CO_2 lasers irradiation procedures. To this end, atomic force microscopy (AFM) and scanning electron microscopy (SEM) images were taken that were further processed to determine statistical and fractal characteristics of the samples.

2. Experimental details

In this study, 75 zirconia ceramic discs with 7 mm diameter and 3 mm thickness (Zirkon Zahn GmbH, Bruneck, Italy) were prepared by a copy milling technique. All samples were annealed in the furnace at 1500 °C prior to wet polishing for 15 s using silicon carbide abrasive paper (English Abrasives, London, UK) with 600, 800 and 1200 grits. Depending on further treatment, the disks were divided into four groups referred to as:

1) REF – control (reference) samples.
2) ND – samples irradiated with Nd-YAG laser.
3) ER – samples irradiated with Er-YAG laser.
4) CO_2 – samples irradiated with CO_2 laser.

The details of the irradiations procedures with the infrared lasers are given in Table 1 ordered with increasing wavelength and pulse width. After irradiation the samples were cooled down to room temperature in ambient air.

Absorptance data presented in Table 1 were estimated from the reflectance and transmittance spectra published elsewhere assuming flat sample surface and perfect crystalline structure [34,35]. Note that samples irradiated with solid state lasers absorb only around 10% of energy of the incident light, whereas those irradiated with CO_2 laser absorb nine times larger energy. In addition, the beams are significantly different in terms of their spectral characteristics: the wavelength, repetition frequency and pulse width. As a result, the effectiveness of the laser irradiation cannot be assessed through comparison of relevant parameters of the characteristics of the beams. Instead, an alternative approach need to be adopted in the current research, in which the irradiation threshold was established before the appropriate treatment, that is such a laser beam characteristics that still remains the surface structure intact. Then, the beams were tuned up slightly above that threshold and the irradiation procedure was carried out.

2.1. Characterization of surface topography

AFM relies on moving very sharp tip on elastic cantilever over the surface of a solid material and probing the tip-surface interactions in order to sample the surface heights. On the other hand, Scanning Electron Microscope (SEM) uses a focused beam of high-energy electrons to drive many forms of emission from the surface it is illuminating (including secondary and backscattered electrons) to build up greyscale maps of a surface topography and elemental composition. Regardless of the method, however, obtained images can be analyzed to derive spatial correlations between distant samples (patterning). To this end a number of methods were proposed that explored, for example, statistical dependencies within a data series, scaling invariance etc. The latter property leads us to the concept of fractal parameters, which appear to be highly appropriate for description of irregular and rough data series, such as those related to surface topography [36–40]. In the following, simple but reliable two-step algorithm for accurate estimation of fractal parameters is described, which was thoroughly tested previously [41].