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Structural studies of degradation process of zirconium dioxide tetragonal phase induced by grinding with dental bur

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ABSTRACT

Zirconium dioxide has been widely used in dental prosthetics. However, the improper mechanical treatment can induce changes in the microstructure of zirconium dioxide. From the viewpoint of mechanical properties and performance, the phase transitions of ZrO_2 from the tetragonal to the monoclinic phase induced by mechanical processing, are particularly undesirable. In this study, the phase transitions of yttrium stabilized zirconium dioxide (Y-TZP) induced by mechanical treatment are investigated by the scanning electron microscopy (SEM), atomic force microscopy (AFM) and powder diffraction (XRD). Mechanical stress was induced by different types of drills used presently in dentistry. At the same time the surface temperature was monitored during milling using a thermal imaging camera. Diffraction analysis allowed determination of the effect of temperature and mechanical processing on the scale of induced changes. The observed phase transition to the monoclinic phase was correlated with the methods of mechanical processing.

1. Introduction

The first medical use (1969) of zirconium dioxide (zirconia) involved the orthopedic hip replacement surgery of monkey femur where instead of titanium prostheses zirconia was used. In dental applications, zirconium dioxide competes even with titanium, which utility for dental implants and abutments is well established. Since then, numerous studies have demonstrated its many advantages such as lack of cytotoxicity or mutagenicity. Especially important feature of zirconia, when compared to titanium is its improved biocompatibility [1–3]. Less pronounced inflammation process around the zirconia implants and smaller accumulations of bacteria on surface as compared to those near titanium have also been observed [4]. Ceramics based on zirconia have gained popularity in dentistry, especially in the performance of fixed prosthodontics. It is related to its biocompatibility, good mechanical properties, aesthetics and the development of new CAD/CAM technology (Computer Aided Design/Computer Aided Manufacturing) enabling the production of fully ceramic crowns and bridges with very high precision and an excellent marginal fit [5–7]. Therefore in dentistry zirconia is mainly used as a substructure for ceramic crowns and bridges, fully zirconia crowns and bridges implant superstructures (implant abutments), dental implants, post and cores, orthodontic braces and even partial dentures [1]. During the preparation of dental crowns, bridges or implants it may be necessary to perform certain adaptations of the shape (using dental diamond bur) and surface conditioning to achieve better connection with cement fixing (e.g. sandblasting, tribochemical treatment, chemical treatment like etching or modification using silane coupling agents). The next step is firing process of porcelain veneering on zirconia core, which means treatment of ceramics via thermal cycles with precise observance of temperature regimes. The impact of these factors on the physical and chemical properties of zirconia has not been yet fully understood [8–14]. Nowadays, due to the evolution of zirconium dioxide applications, a photofunctionalization method was introduced in order to modify zirconia surface without compromising its properties [15].

Zirconium dioxide is a polymorphic material and exists in three polymorphic crystal forms: monoclinic, tetragonal and cubic. At room temperature, zirconia occurs in the monoclinic phase. Heated to a temperature above 1170 °C it is converted into the tetragonal phase, and above 2370 °C into the cubic one. The most preferred from the biomechanical point of view, is the tetragonal form. The mechanical properties depend largely on the zirconia phase

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transition mechanism [1]. The reinforcing transformation occurs when the particles are in a metastable zirconia tetragonal phase (just about to undergo the transition). Metastable state depends on the composition, size, shape of zirconia particles, the type and amount of stabilizing oxides, zirconia interaction with the other phases and processing method. The phase transition may be induced by an external force such as grinding, cooling, striking. The transformation from the tetragonal to the monoclinic phase causes a volume increase by 4%, inducing compression strain. It is accompanied by a number of micromechanical processes, which in varying degrees contribute to the strengthening of zirconia ceramics [16–19]. The impact of different grinding procedures on selected medical zirconia samples was studied by Lee et al. [20] and Mohammadi-Bassir et al. [21].

The aim of the study was to evaluate the impact of mechanical processing i.e. grinding using diamond bur with and without the presence of water cooling on the microstructure and some physical properties of zirconia used in dentistry. However, as far as we know, the treatment temperature has been often assumed to be correlated with the phase transitions of zirconium dioxide, however, no measurements have been performed to actually support this assumption. In this study such an attempt was made.

2. Materials and methods

Thirty-three rectangular samples of zirconium dioxide were prepared. The initial block (in pre-sintered form) was zirconia Lava Plus (Lava™ Plus, 3M ESPE), which was sliced into samples using a water cooled diamond circular saw. The samples were polished to the desired size of 37 × 15 × 3.6 mm and then were subjected to sintering process in a dedicated furnace for dental ceramics (Lava Furnace, 3M ESPE) in accordance to the manufacturer's instructions. The sintering procedure caused a reduction in the sample size to 29.5 × 12 × 3.3 mm, as approximately 20% contraction in volume of the material took place. According to Garvie and Nicholson, the diffraction pattern is affected by the thickness of the sample, therefore the thickness of the sample should not be less than 2 mm [18]. The samples were processed using diamond burs commonly used in dentistry (881/016, Drux GmbH, Germany), mounted on dental 1:5 Contra Angle Handpiece (Ti-Max M95, NSK, UK) with different grit size: 135 μm (manufacture's symbol K4, grit size – coarse), 50 μm (manufacture's symbol K2, grit size – fine), 30 μm (manufacture's symbol K1, grit size – fine) and 15 μm (manufacture's symbol K0, grit size – very fine).

In order to achieve a direct relation between the model and clinical procedures and to eliminate the human effect, a special measuring setup had to be designed and built. The measuring setup had standardized possibilities of setting to ensure an accurate determination of temperature distribution in the studied samples. The setup also ensured the possibility of mounting the standard tools used in dentist surgery such as dental drill 1:5 with a microengine. Another requirement of the setup was to make sure that the sample would be able to perform to- and -from motion over a constant path and at constant frequency, under a constant pressure of the drill head to the samples. A measuring setup that meets all these requirements was designed and constructed with the elements produced in rapid prototyping technology (a 3D printer) [22]. The stand allowed to standardize and adjust advisable conditions for the experiments and to clearly visualize temperature range of the processes, by means of infrared camera (Fig. 1) [22].

Variable parameters used in the treatment of zirconium dioxide surface include: different diamond bur grits (135 μm, 50 μm, 30 μm, 15 μm), the contact force of the drill to the sample (4.5 N and 9 N), the speed of the drill (40000 rpm and 200000 rpm), the presence of or absence of cooling water (W+ and W−). We have examined samples with all possible combinations of mentioned variables. All the samples were processed for 300 s. This time was estimated based on the time needed for clinical and laboratory corrections while fabrication of dental appliances. For example, shorter time is needed for correction of a CAD/CAM substructure for a prosthetic crown however for zirconia abutments corrections longer time is needed.

2.1. Thermographic measurements

Thermographic measurements were performed using a Flir SC 620 camera equipped with an non-cooled microbolometric detector with a resolution (IR resolution) 640 × 480 pixels, and noise equivalent differential temperature sensitivity (NEDT) < 40 mK. The camera was equipped with an objective characterised by field of view (FOV) 24° × 18°. Thermograms were recorded at a 30 Hz frequency. All thermographic sequences were recorded under identical conditions. Measurements were carried out in the temperature range 0–500 °C and in the spectral range of 7.5–13 μm. The treatment process of Y-TZP samples was recorded at an angle of approximately 10°.

2.2. X-ray diffraction analysis (XRD)

A series of diffraction measurements were performed using a modified diffraction system HZG-4 and high-voltage generator TUR (30 mA, 34 kV). Diffraction studies were carried out using CuK_{α}X-rays (λ = 0.154178 nm). The diffraction data were recorded for the angle 2θ in the range 20–40° at the scanning rate of 0.24°/min. The range of Bragg angle was chosen because preliminary studies revealed that the most visible changes are located in this area and also according to other authors' observations [18,23–25]. Individual diffraction peaks were fitted with a Gaussian shape function using the PeakFIT software (version 4.12, Gambit). The obtained absolute values of the area under the Gaussian profiles of selected diffraction peaks (2θ = 28, 30, 34.5 and 35°) were converted into percentage values and normalized to 100%. The analysis of percentage changes was related to the reference sample (not submitted to machining).

To compare the changes of diffraction peak areas for samples processed with various parameters the statistical analyses were applied using Statistica programme. In the first step, the data distribution was evaluated, in particular whether the data distribution is normal, using standard Shapiro-Wilk's test. Because there was disagreement with normal distribution, therefore non-parametric tests were applied. To find statistically significant differences among the normalized peak areas of samples studied the Kruskal–Wallis tests were conducted. Then to assess the differences between various parameters, tested during our experiments, the Mann-Whitney testes for particular pairs of parameters were conducted.

Additionally, we performed further analyses of XRD patterns, fitting diffraction peaks using Gaussian/Pearson functions. We conducted analyses, based on the angular range from 28 to 32° and on Garvie-Nicholson's method [18] originally used to calculate molar fraction of monoclinic relative to the zirconia cubic phase which can be determine by the following formula:

$$X_{m/c} = \frac{I(\bar{1}11)_m + I(111)_m}{I(\bar{1}11)_m + I(111)_m + I(111)_c} \quad (1)$$

where I represents the intensity of the XRD peaks and the numbers in brackets are the Miller indices of the pertinent crystallographic planes. We adopted it to our data to determine the

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