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Microstructure and nanoindentation analyses of low-temperature aging on the zirconia-porcelain interface

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ABSTRACT

The aim of the present study was to investigate the effects of low-temperature aging on the micro-mechanical and micro-structural properties of zirconia-porcelain interface. In total, thirty-three Y-TZP zirconia blocks were fabricated by using CAD/CAM technology, veneered with porcelains. Specimens were submitted to low-temperature aging in an autoclave at 134 °C, additional 0.2 MPa pressure for 0 h, 5 h, or 10 h. Flexural strength was obtained by using three-point bending test. Micro-mechanical properties (nano-hardness (H) and reduced modulus (E_r)) were investigated by nanoindentation tests. Scanning electron microscopy and X-ray diffraction analyses were performed to identify the micro-structure and fracture behavior. The flexure strength, modulus and hardness of zirconia increased after 5 h aging and decreased after 10 h aging. No significant alterations of the reduced modulus or hardness of porcelain were detected in the whole aging duration. Width of the zirconia-porcelain interface was extended towards the bulk of zirconia. The detachment and cracks could be observed in zirconia, and the crystal alignment was disorganized in porcelain after 5 h aging and 10 h aging. Mechanical properties of the veneering porcelain are not affected by low-temperature aging. However, the expansion and the alterations of micro-mechanical and micro-structural properties of zirconia-porcelain interface were detected.

1. Introduction

Zirconia-based restorations, exhibiting excellent mechanical strength, superior biocompatibility, and potential esthetics, have attracted extensive attention in prosthetic dentistry (Ferrari et al., 2015). Fatigue and/or aging can cause a significant reduction of the mechanical properties of different commercial zirconia materials (Flinn et al., 2012; Siarampi et al., 2014). However, the fracture resistance of the zirconia frameworks was still sufficient to withstand the loading conditions, even in the molar regions (Nakamura et al., 2015). The reliability of zirconia application in dentistry has been comprehensively accepted. The primary concern of zirconia-based prostheses was not related to framework integrity, but rather to edge chipping (“cohesive” failure) (Chai et al., 2014) or interfacial fracture (“delamination” failure) (Costa et al., 2014) of zirconia-veneering porcelain interface. Many factors are involved in the failure of interface, such as the coefficient of thermal expansion (CTE) (Mainjot et al., 2015), residual stress in the fabricating procedures, and fatigue (Kohorst et al., 2013; Tang et al., 2015). More recently, low-temperature degradation (LTD) of Y-TZP has been considered as an important issue for the lifetime of zirconia-based restorations(Lughi and Sergo, 2010).

LTD is known that the metastable tetragonal phase (t) transforms into the monoclinic phase (m) in a humid atmospheres at moderate temperatures below 400 °C, even at the room or body temperature. The phase transformation generates compressive stress for the volume expansion in localized areas around micro-cracks, resulting in arresting the crack propagation, known as transformation toughening (Chevalier et al., 2007). However, if the transformation occurs as a widespread surface feature, compressive stresses induced by volume extension of transformed grains may result in crack formation and propagation. This leads to a deterioration of the mechanical properties and may eventually result in ultimate failure of the material. Therefore, this t→m transition can be both beneficial and detrimental to the properties and the lifetime of the zirconia-based restorations. Clinical data and experimental investigations have shown that the occurrence of veneering porcelain chipping of zirconia-based restorations is relatively high after several years (Larsson and Vult Von Steyern, 2013; Pang et al., 2015; Quinn et al., 2010; Raigrodski et al., 2012). It has been proved that LTD begins at the surface of zirconia, and then enters the bulk of the zirconia material, which mainly influences the mechanical proper-

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ties of zirconia (Chevalier et al., 2007). However, whether the LTD effect occurred on the interface of zirconia-veneering porcelain, or contributed to the chipping of veneering porcelain has not been fully clarified yet. Tang et al. (2012) found that surface textures of five different veneering porcelains were changed after accelerated aging. It has been reported that the adhesion of VM9 layered porcelain to the zirconia framework was significantly decreased by increasing the autoclave cycles (Li et al., 2013). Nevertheless, controversial conclusion can be also available in the literature. Xie et al. (Xie et al., 2016) have declared that different veneering firing cycles did not influence the flexural strength or surface Vickers hardness of zirconia-based restorations.

Therefore, the aim of the present work was to investigate the influences of LTD on the mechanical and microstructural properties of zirconia-based restorations, especially on the interface between zirconia and veneering porcelain at nano-scale. The null hypothesis of the present study was that LTD does not affect the microstructural and mechanical properties of zirconia, veneering porcelain and the interface between zirconia and veneering porcelain.

2. Materials and methods

2.1. Specimen preparation

An yttria-stabilized tetragonal zirconia polycrystal ceramic (Y-TZP, CTE=$10.5\times10^{-6}\ K^{-1}$, Cercon, DeguDent, Hanau, Germany), and a veneering porcelain (Cerabien ZR, CTE=$9.1\times10^{-6}\ K^{-1}$, Noritake, Nagoya, Japan) were used in this study. Thirty-three Y-TZP bars were cut from blocks, and sintered at 1450 °C for 2 h, according to the manufacturer's instructions. After sintering, the dimensions of the zirconia bars were 24 mm×4 mm×1 mm. The bars were sequently polished by using 9 µm, 6 µm, and 1 µm diamond suspensions. Veneering of the porcelain on the zirconia substrate was performed with assistance of a metal template using the traditional layering technique. A total of four firings including the final glaze firing (shade base, body and enamel dentin firings) were required to produce the layer of veneering porcelain. In addition, an extended drying time of 12 min with the body and enamel dentin firings were used to compensate for the large volume of porcelain. This procedure is not usually performed because typical restorations are smaller and are sufficiently dried with the normal drying time. The firing procedures were recommended by the manufacturer, without internal or external staining. The veneering porcelain surface was wet-ground using 320–1200 grit silicon carbide abrasive paper to obtain a flat surface and consistent dimensions of 24 mm×4 mm×2 mm.

2.2. Low-temperature degradation simulation

Specimens were ultrasonically cleaned in distilled water for 3 min. And then accelerated aging test was performed in an standard autoclave with the conditions of 134 °C and 0.2 MPa for 5 h, or 10 h (n=11). Specimens without aging procedure were served as control group. Guideline of ISO 13356 (2008) was referred in this study.

2.3. Characterization of mechanical properties

2.3.1. Flexural strength

Flexural strength was determined by using a three-point bending test. Specimens were tested by using a universal testing machine (3510, Bose, American), with a crosshead speed of 0.5 mm/min at room temperature (20 ± 1 °C). Initial fracture stress δf (n=8) was calculated according to ISO 6872 (2008):

$$\delta f = 3Fl/2wh^2 \quad (1)$$

where F is the fracture load, l is the roller span (20 mm), w and h represent the width and the height of the specimen. Image acquisition was performed by using a high-speed camera (frame rate 50,000 fps, FASTCAM SA5, Photron, American) to record the process of deformation and fracture during loading.

2.3.2. Nanoindentation

Nanoindentation was performed by using a TriboScope nanomechanical testing system (Tribolab, Hysitron Incorporation, Minneapolis, USA) equipped with an in situ imaging mode. A pyramidal diamond Berkovich indenter with a total included angle of 142.3°, and a tip with a radius of curvature of approximately 120 nm, was utilized. Trapezoidal loading–unloading profile was used, as defined by 5 seconds loading, unloading segments, and a holding time of 2 s at a maximum load of 8 mN. Hardness (H) and reduced modulus (E_r) values were calculated from the experimental unload-displacement curves using the Oliver and Pharr model (1992). For a Berkovich indenter, the hardness is defined as:

$$H = P_{max}/A \quad (2)$$

where H is hardness, P_{max} is the maximum applied force and A is the projected contact area.

The reduced modulus, E_r, is calculated from the unloading data as:

$$E_r = \sqrt{\pi}\ S/2\beta\ \sqrt{A} \quad (3)$$

where β is equal to 1.034 for a Berkovich indentation, S is contact stiffness. The modulus, E, of the sample can easily be determined from the following relationship:

$$1/E_r = 1-\nu^2/E + 1-\nu_i^2/E_i \quad (4)$$

where E_i and υ_i are Young's modulus and Poisson's ratio of the material and the indenter, respectively (E_i=1141 GPa, and υ_i=0.07).

Statistical grid nanoindentation tests were carried out across the interface of zirconia-veneering porcelain. There were 49 indents per specimen with a distance of 8 µm at the lateral and vertical directions. For the transition region of veneering porcelain and zirconia (approximate of 8 µm), intensive intensity of indents were adopted with the inter-distance of 3 µm. The distribution of indents was shown in Fig. 1. After indentation, in situ scanning probe microscopy (SPF) imaging was performed to obtain the surface topography using a very light loading force (2 µN) without causing any damage to the surface.

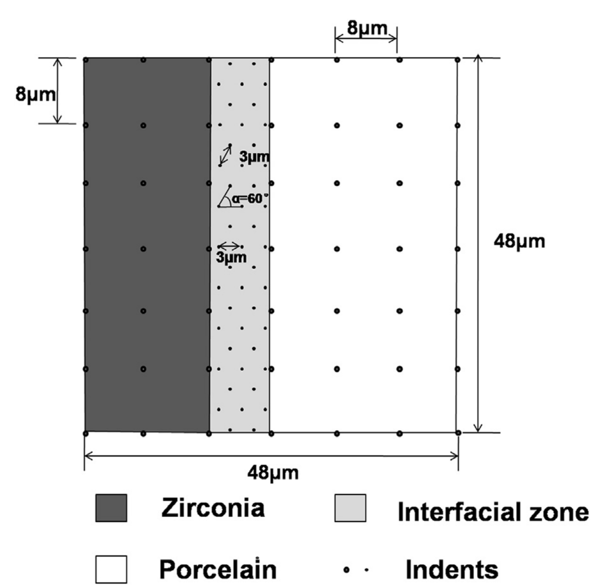

Fig. 1. Grid nanoindentation across the interface between zirconia and porcelain.