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Aging resistance of surface-treated dental zirconia

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ARTICLE INFO

Article history:
Received 18 March 2014
Received in revised form
19 July 2014
Accepted 28 November 2014

Keywords:
Zirconia
Alumina sandblasting
Tribochemical silica sandblasting
Aging
Rough polishing
Low-temperature degradation

ABSTRACT

The influence of surface treatment on the low-temperature degradation (LTD) of tetragonal zirconia polycrystalline (TZP) is still unclear.

Objectives. The effect of surface treatments on the LTD behavior of zirconia was investigated.

Methods. Fully-sintered specimens of seven commercial dental zirconia (Aadva, GC; In-CeramYZ, VITA; IPS e.max ZirCAD, Ivoclar Vivadent; LAVA Frame and LAVA Plus, 3M ESPE; NANOZR, Panasonic; ZirTough, Kuraray Noritake) were provided by the manufacturers with specimen dimensions of approximately 10 mm × 5 mm × 3 mm. For each zirconia grade, samples were kept 'as sintered' (untreated) or were subjected to one of the three surface treatments: rough polished, sandblasted with Al_2O_3, tribochemical silica sandblasted ($n = 3$/group). The tetragonal to monoclinic transformation was evaluated by X-ray diffraction at several intervals during LTD testing up to 40 h in steam in an autoclave (134 °C, 2 bar).

Results. The five yttria-stabilized TZP (Y-TZP: Aadva, In-CeramYZ, IPS e.max ZirCAD, LAVA Frame, LAVA Plus) zirconia showed a similar trend in LTD behavior. The Al_2O_3 sandblasted zirconia showed the highest monoclinic volume fraction. The as sintered (untreated) zirconia degraded faster than the surface-treated zirconia. Although the surface-treated ceria-stabilized TZP/alumina (Ce-TZP/Al_2O_3: NANOZR) zirconia had a higher initial monoclinic volume fraction compared to the Y-TZP zirconia, it showed a stronger aging resistance. The as sintered (untreated) Y-TZP/alumina (Y-TZP/Al_2O_3: ZirTough) zirconia showed a strong aging resistance, whereas the surface-treated Y-TZP/Al_2O_3 zirconia degraded slightly.

Significance. Surface treatment improved the aging resistance of Y-TZP zirconia. Surface treatment did not affect the LTD behavior of Ce-TZP/Al_2O_3 zirconia, while surface treatment decreased the aging resistance of Y-TZP/Al_2O_3 zirconia.

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http://dx.doi.org/10.1016/j.dental.2014.11.018
0109-5641/

1. Introduction

All-ceramic restorations have well been adopted in daily clinical practice as fixed dental prostheses or FDPs. Thanks to its biocompatibility and promising mechanical properties, yttria-stabilized tetragonal zirconia polycrystalline (Y-TZP) ceramics can be used as an alternative for conventional metal or metal-ceramic restorations. More recently, dental zirconia has also been introduced for full-contour 'all-zirconia' crowns and bridges, as well as for implant and implant abutments.

In most cases, all-ceramic restorations are CAD/CAM machined from pre-sintered ceramic blocks, and sintered to full density afterwards. To improve the shape of the zirconia core, the dental technician often needs to additionally abrade the zirconia core [1]. Furthermore, due to the high chemical inertness, different mechanical surface pre-treatments have been recommended to improve the bonding of composite cement to zirconia, this in light of an adhesive luting procedure. For instance, aluminum-oxide (Al_2O_3) sandblasting or tribochemical silica sandblasting with 30- and 110-μm silica-coated Al_2O_3 particles have been shown not only to roughen, but also to chemically activate zirconia, the latter thus making it more receptive for chemical bonding via silane coupling agents [2]. Because zirconia ceramics exhibit a stress-induced transformation, the surface of the abraded or sandblasted zirconia will be transformed, i.e., constrained as well as damaged; this may influence its long-term performance under clinical conditions [1].

It is well known that hundreds of zirconia total hip prosthesis heads failed catastrophically between 1999 and 2001, which led to its withdrawal from the market soon after [3–5]. Later in 2007, the problem of catastrophic failures was attributed to low-temperature degradation (LTD), i.e. transformation of the metastable tetragonal to monoclinic phase (at 20–250 °C), initiated and accelerated by water penetration [6–8]. The cause of the failures was related to an accelerated tetragonal to monoclinic phase transformation of zirconia in a limited number of batches [8]. Although the manufacturing process of those orthopedic zirconia femoral heads is significantly different from that of dental zirconia, more research attention is recently also devoted to LTD of dental zirconia [9–13]. At the moment, however, only few papers reported on the influence of surface treatment on LTD of dental zirconia [1,9,14–16], whereas any direct comparison among different dental zirconia is currently missing. The objective of this study was therefore to evaluate the influence of different surface treatments on the LTD behavior of dental zirconia. The null hypothesis tested was that different surface pre-treatments do not affect the LTD behavior of dental zirconia.

2. Materials and methods

The study design is schematically explained in Fig. 1. Fully-sintered zirconia specimens, five so-called 'Y-TZP' zirconia (Aadva, GC, Tokyo, Japan; In-Ceram YZ, VITA, Bad Säckingen, Germany; IPS e.max ZirCAD, Ivoclar Vivadent, Schaan, Lichtenstein; LAVA Frame and LAVA Plus, 3M ESPE, Seefeld, Germany), one ceria-stabilized tetragonal zirconia polycrystalline (Ce-TZP)/Alumina (Al_2O_3) zirconia (NANOZR, Panasonic, Osaka, Japan), and one Y-TZP/Al_2O_3 zirconia (ZirTough, Kuraray-Noritake Dental, Tokyo, Japan) were provided by the manufacturers. All specimens were obtained in the form of sintered rectangular bars (10.0 mm × 5.0 mm × 3.0 mm) from the different suppliers and all surface treatments were directly applied to these as received samples without additional machining operations. Those specimens were ultrasonically cleaned in acetone for 10 min and thoroughly dried with compressed air. All specimens of each grade were assigned into four groups of four specimens each, and either were kept as sintered (untreated) or were rough polished using a polishing disk (MD Allegro 250, Struers, Ballerup, Denmark) with a diamond suspension (15 μm water-based diamond suspension, Kemet Europe, Kapellen, Belgium), sandblasted with 50 μm Al_2O_3 particles (Danville Engineering, San Ramon, CA, USA), or tribochemical silica sandblasted with 30 μm Al_2O_3 particles using CoJet (3M ESPE). Details of the surface treatments are summarized in Table 1. Both the top and bottom surfaces received the same surface treatment. One specimen from each zirconia grade was used for microstructural investigation using scanning electron microscopy (SEM, XL30-FEG, FEI, Eindhoven, Netherlands) employing the following conditions (gold coated, 10^{-5} mbar pressure, 10 kV energy range, 144 μA beam current, secondary electron image).

Surface roughness was measured on the three remaining specimens of each grade using an optical interferometer (Wyko NT3300, Veeco, Tuscon, AZ, USA) with 5× magnification. Quantification of the three-dimensional (3D) surface roughness parameter Sa (arithmetic mean deviation) was performed using Vision32 software (Veeco). For each specimen, five regions (effective field of view was 1.210 mm × 0.921 mm) on the surface-treated sides were selected. For surface roughness, a linear mixed-effects model (nlme package, R3.01, R foundation for Statistical Computing, Vienna, Austria) was constructed to assess the influence of the different surface treatments. For this model, the 'as sintered' (untreated) condition was excluded, since data largely varied, depending on the sample preparation process conducted by the manufacturers. In this model, two fixed effects, 'ZIRCONIA GRADE' and 'SURFACE TREATMENT' were considered, as well as their mutual interaction. The specimen measured was considered as a random effect.

Micro-Raman spectroscopy was next performed in order to detect potential residual stress on the surface [17]. Raman spectra (SENTERRA, BrukerOptik, Ettlingen, Germany) were collected using the following conditions: Ar-ion laser with a wavelength of 532 nm, 20 mW power at sample and 100× objective. The spectrum integration time was 20 s with the recorded spectra averaged over three successive measurements. For each specimen, at least 12 measurements were performed using a pinhole aperture of 50 μm. The degree of correlation between the Raman wavenumber of the tetragonal (t-ZrO_2) band around 147 cm^{-1} and the monoclinic (m-ZrO_2) volume fraction was calculated (R3.01, R Foundation for Statistical Computing) for each grade.

Cu K_α (40 kV, 40 mA) X-ray diffraction (XRD, Seifert 3003 T/T, Seifert, Ahrensburg, Germany) analysis was used for phase identification and calculation of the relative phase content of m-ZrO_2 and t-ZrO_2. Both the top and bottom surfaces of