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Influence of hydrothermal and mechanical conditions on the strength of zirconia

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ABSTRACT

Low temperature degradation and mechanical and thermal cycling may decrease the strength of zirconia and jeopardize the long-term success of dental restorations made of this material. The objective of this study was to reveal the influence of different environmental and loading conditions on the strength of 3 mol.% yttria-stabilized polycrystalline tetragonal zirconia (3Y-TZP). A total of 144 disk specimens were produced from each of two 3Y-TZP materials, and subjected to one of the following conditions: (A) no further treatment (control); (B and C) 10^6 and 5×10^6 mechanical cycles, respectively, with an upper load limit of 100 N; (D) 10^4 thermal cycles between 5 and 55 °C; (E) 200 days storage in water at 36 °C; (F) a successive combination of conditions B, D and E; (G) storage in water at 80 °C for 64 days; (H) storage in an autoclave at 134 °C for 8 h. Monoclinic phase content was evaluated by X-ray diffraction (XRD) analysis. Specimen strength was determined in a biaxial bending test. The two ceramics exhibited average strengths of 995 and 1239 MPa, respectively. No statistically significant influence of any treatment on strength was demonstrated for either material. However, XRD measurements revealed a substantial increase in monoclinic phase content, from an initial 2% (control) to up to 10%, according to storage conditions. As a consequence of hydrothermal loading a tetragonal to monoclinic phase transformation took place at the surface of the 3Y-TZP materials investigated, but, like thermal and mechanical cycling, this did not lead to significant changes in bulk strength.

1. Introduction

Over the past years, 3 mol.% yttria-stabilized polycrystalline tetragonal zirconia (3Y-TZP) has been increasingly used for the fabrication of all-ceramic restorations in dentistry. This type of zirconia ceramic combines high esthetics, excellent biocompatibility, low plaque accumulation and low thermal conductivity with remarkable strength characteristics which had only been achieved before by metallic materials [1,2]. The high strength of zirconia even permits the application of all-ceramic fixed dental prostheses or implant abutments in the posterior region and is founded on a characteristic reinforcement process [3,4]. Zirconia exists in three crystallographic polymorphs. The monoclinic phase (m) is stable at room temperature, the tetragonal phase (t) is stable between 1170 and 2370 °C while the cubic phase (c) is the high temperature structure [5]. The tetragonal form can be retained in a metastable state at room temperature by adding various oxides, in particular yttria, which is the oxide commonly used for dental applications [1]. Under high localized stress, in particular at flaws on the surface or within the lattice, a structural transformation from the tetragonal to monoclinic phase takes place. This complex mechanism is called transformation toughening. The associated expansion of 4% in volume induces localized compressive stresses and microcracks around the transformed particles, which effectively oppose the opening of cracks and increase the resistance to crack propagation [5,6]. However, aside from structural defects within the lattice, a slow t → m transformation may also be caused by fatigue at low temperatures in the humid environment of the oral cavity [7,8]. This phenomenon is commonly referred to as ageing or low temperature degradation (LTD). LTD is initiated from isolated surface grains at which water is incorporated into the zirconia lattice by dissolving Zr–O–Zr bonds and filling oxygen vacancies [9–11]. This reduces the energy barrier for t → m transformation and thus increases the rate of transformation, which gradually spreads along the surface and proceeds into the bulk. Although this mechanism is very slow at oral temperatures, it may result in significant reductions in strength, toughness and density [12,13]. The reduction in strength due to LTD may be enhanced by applying cyclic stresses (e.g. chewing forces) [14,15]. Strength may also be degraded by repeated thermal stress, leading to the generation of tensions within the ceramic material, which are manifested as slow subcritical

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crack growth [16,17]. As zirconia restorations are exposed to both a humid environment and to cyclic loading in the oral cavity, evaluation of the fatigue characteristics under such conditions is essential for the successful application of zirconia restorations in dentistry.

In a recent investigation [3], four unit all-ceramic fixed dental prostheses (FDP) with zirconia frameworks were shown to withstand the occlusal forces normally acting during function. However, a considerable decrease in load-bearing capacity, by up to 40%, was observed after combined mechanical and thermal cycling and long-term storage in water at body temperature [18–20]. The question arose as to what extent this decrease was due to ageing of the framework, possibly related to LTD of zirconia, or to fatigue, either of the veneering ceramic or of its bond to the framework, both leading to reduced stability of the compound structure. In order to answer this question with respect to the ageing of zirconia we evaluated the strength of zirconia specimens after the same combined treatment as in previous investigations and, to obtain a more detailed picture of the behavior of zirconia, after subjecting it to several mechanical, thermal and hydrothermal conditions alone. The hypothesis to be tested was that specimen treatment had a significant influence on the strength of zirconia.

2. Materials and methods

Two commercially available zirconia framework ceramics, each stabilized by the addition of 3 mol.% yttrium oxide (3Y-TZP), were chosen for this study. The materials were Lava Frame (3M ESPE, Seefeld, Germany) and VITA In-Ceram YZ for inLab (Vita Zahnfabrik, Bad Säckingen, Germany), referred to below as LA and YZ, respectively. These were the same materials as had been used in a previous study by Kohorst et al. [18]. A total of 144 disk-shaped specimens (final diameter 14 mm, thickness approximately 1.3 mm) were produced from each material by machining and cutting white blanks, followed by sintering at 1500 °C (LA) or 1530 °C (YZ). LA specimens supplied by the manufacturer had been parallel ground and subjected to additional thermal etching (30 min at 1350 °C) in order to achieve a state comparable with "as fired". Subsequently, specimens of both materials were ground and finished bilaterally with diamond media of decreasing grit size down to 15 μm (MD Piano 220, MD Piano 600 and DP suspension P, Struers A/S, Ballerup, Denmark) for 3 min each on a grinding wheel (DAP7, Struers A/S, Ballerup, Denmark) at a rotational speed of 250 min^{-1} and a pressure of 4.4 N cm^{-2}. Each grinding step was accompanied by cooling with water or, in the case of the diamond suspension, with an alcohol-based lubricant (DP lubricant blue, Struers A/S, Ballerup, Denmark), and completed by cleansing under running water. Surface preparation followed the prescriptions of ISO 6872 [21]. The surface quality of both the sample disks and of a remaining, non-veneered framework specimen from the previous study by Kohorst et al. [18] was assessed as the arithmetic mean roughness value (R_a) by profilometry (radius of stylus point curvature 5 μm, evaluation length 4 mm, cut-off wavelength 0.8 mm, measurement on three different locations per specimen) (contact stylus instrument TR6, Hommelwerke, Villingen-Schwenningen, Germany). The disk specimens were randomly allotted to 1 of 10 groups which received the following treatments (see also Table 1): (A) no further treatment (control); (B and C) 10^6 and 5×10^6 cycles, respectively, of biaxial bending in a piston on three balls set-up with an upper load limit of 100 N (corresponding to 110 MPa); (D) 10^4 thermal cycles between 5 and 55 °C, dwell time 30 s; (E) 200 days storage in distilled water at 36 °C; (F) a successive combination of conditions B, D and E; (G) storage in distilled water at 80 °C for 64 days; (H) storage in water in an autoclave (StM-MCS-J, SANOclav, Bad Überkingen-Hausen, Germany) at 134 °C and 3 bar absolute pressure for 8 h. Specimen strength after treatment was determined in quasistatic biaxial flexural tests (see Fig. 1) according to ISO 6872 [21] in a universal mechanical testing machine (20 K, UTS Testsysteme, Ulm, Germany) at a cross-head speed of 1 mm min^{-1}. The set-ups for dynamic and quasistatic biaxial flexural loading comprised three stainless steel balls (diameter 4.0 mm, material X46Cr13) on a support circle with a diameter of 12.0 mm and a piston (high speed steel, diameter 1.4 mm). The load was raised until fracture of the specimen and the maximum force recorded. This was later used to determine the biaxial flexural strength, using the formula given in the standard ISO 6872 [21] and taking the Poisson number for zirconia as 0.25 [22]. Group mean strengths were calculated and the influence of treatment on strength determined by ANOVA with 0.05 chosen as the level of significance. All statistical evaluations were performed with the software package SPSS version 16 (SPSS Inc., Chicago, IL).

Additionally, four selected LA specimens in groups G and H were subjected to X-ray diffraction (XRD) analyses before and after hydrothermal treatment and, in the case of group G, at different

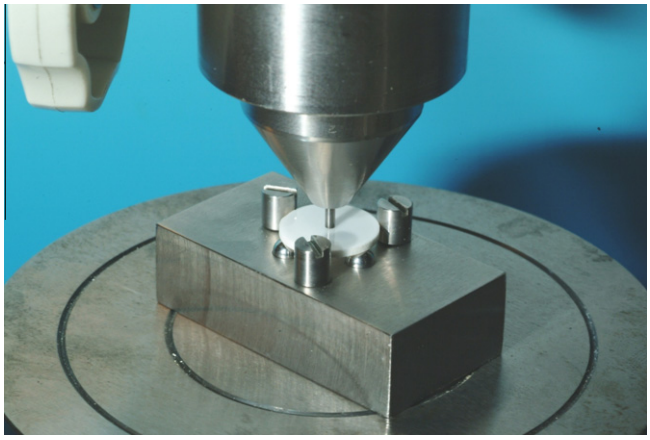

Fig. 1. Specimen in piston on three balls set-up for biaxial strength testing. Poly(ethylene) foils were usually interposed but are not shown for clarity.

Table 1
Treatments for different specimen groups.

Group	Treatment	Temperature (°C)	Special conditions	Duration
A	Control	23		
B	Mechanical cycling	36	σ_{max} = 110 MPa[a]	1,000,000 cycles
C	Mechanical cycling	36	σ_{max} = 110 MPa[a]	5,000,000 cycles
D	Thermal cycling in distilled water	5–55	Dwell time 30 s	10,000 cycles
E	Storage in distilled water	36		200 days
F	Combination of B, D and E	–		200 days
G	Storage in distilled water	80		64 days
H	Storage in water	134	Autoclave, 3 bar	8 h

[a] Maximum applied stress in biaxial cyclic bending.

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