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Low temperature degradation of single layers of multilayered zirconia in comparison to conventional unshaded zirconia: Phase transformation and flexural strength

Sebastian Wille[a,*], Paul Zumstrull[a], Victor Kaidas[b], Lea Katharina Jessen[b], Matthias Kern[a]

[a] *Department of Prosthodontics, Propaedeutics and Dental Materials, School of Dentistry, Christian-Albrechts University at Kiel, Germany*
[b] *Functional Nanomaterials, Institute for Materials Science, Faculty of Engineering, Christian-Albrechts University at Kiel, Germany*

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ABSTRACT

Objectives: The aim of this study was to evaluate the transformation of yttrium stabilized tetragonal zirconia (Y-TZP) to monoclinic phase and its change in flexural strength of the various layers of multilayered zirconia (enamel layer, transition layer 1, transition layer 2 and body layer) in comparison to two conventional zirconia ceramics. Additionally, the ball-on-three-balls test was compared with a conventional biaxial flexural strength test.

Methods: The crystallographic structure of the four layers of Katana Zirconia ML (Kuraray), e.max ZirCAD (Ivoclar Vivadent) and Lava Plus (3M ESPE) was investigated by x-ray-diffraction before and after hydrothermal aging in an autoclave for 5, 10, 15 and 20 h. The biaxial flexural strength was examined with a piston-on-three-ball test and a ball-on-three-balls test.

Results: There was a significant difference of the ratio of transformation with respect to the tested materials, also between the four different layers of multilayered zirconia. After 20 h of hydrothermal aging the mean ratio of monoclinic phase ranged from 9.9 vol% (LAVA Plus) to 44.2 vol% (Katana Zirconia ML, enamel layer). However, hydrothermal aging had no significant influence on the flexural strength of any material. Furthermore, the flexural strength measured by the ball-on-three-balls test was higher than that measured by the piston-on-three-ball test.

Conclusion: A ratio of 40% monoclinic phase at the surface did not significantly influence the biaxial flexural strength. Both test methods provide reliable results. However, as the ball-on-three balls test is less dependent on surface roughness, it might be preferred for investigations adapted to clinical reality.

1. Introduction

Yttria-stabilized tetragonal zirconia polycrystalline ceramics (Y-TZP) are commonly used as alternative for metal or metal-ceramic restorations, as they combine biocompatibility and very promising mechanical properties (Manicone et al., 2007; Piconi and Maccauro, 1999). Nowadays Y-TZP has also been introduced into the market for dental implants and implant abutments. Special zirconia ceramics have been developed for monolithic crowns and fixed dental prostheses (Beuer et al., 2012; Johansson et al., 2014). Monolithic zirconia restorations require optimized esthetic properties as they are not veneered. The esthetic properties are determined by the translucency and the color of the material.

However, as catastrophic failures of hip replacements containing a zirconia ceramic femoral head occurred (Hummer et al., 1995; Norton et al., 2002), aging of zirconia has to be considered also for the dental applications. The phase transition from tetragonal to monoclinic phase of Y-TZP under hydrothermal and/or mechanical loading is well known (Chevalier et al., 1999; Gupta et al., 1978) and called low temperature degradation (LTD). Various laboratory studies investigated Y-TZP ceramics by using a rapid aging method in an autoclave at 134 °C in water vapor with a pressure of 2 bar and measuring the ratio of monoclinic zirconia by x-ray diffraction (XRD) or Raman spectroscopy (Chevalier et al., 1999, 2007; Djaker et al., 2013; Inokoshi et al., 2015; Kosmac and Kocjan, 2012; Lughi and Sergo, 2010; Siarampi et al., 2014). It is well known that the grain size and also the exact chemical composition influence the phase transformation in zirconia ceramics (Flinn et al., 2012; Kosmac and Kocjan, 2012; Lughi and Sergo, 2010; Palmero et al., 2015).

* Corresponding author.
E-mail addresses: swille@proth.uni-kiel.de (S. Wille), paul.zumstrull@gmail.com (P. Zumstrull), v.kaidas@googlemail.com (V. Kaidas), leje@tf.uni-kiel.de (L.K. Jessen), mkern@proth.uni-kiel.de (M. Kern).

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Several studies considered different concepts for translucent zirconia and their influence on the aging resistivity and flexural strength (Nakamura et al., 2016b; Samodurova et al., 2015; Zhang et al., 2015; Zhang, 2014). There are two different methods to obtain the shaded zirconia before the final sintering process: One method uses metal oxides that are mixed into the zirconia powder, the other method utilizes coloring solutions before sintering in order to get the zirconia infiltrated with coloring ions (Sedda et al., 2015). Nakamura et al. (2016a) tested a shaded zirconia with both coloring methods in comparison to unshaded zirconia. Both coloring methods resulted in a lower transformation ratio after 10 and 100 h of aging in an autoclave. However, the coloring procedures did not significantly influence the biaxial flexural strength (Nakamura et al., 2016a). However, different coloring compositions with respect to their aging behavior have not been investigated in the cited study.

A multilayered zirconia (Katana Zirconia ML, Kuraray, Tokyo, Japan) consists of four layers with different shades, which are named enamel layer, transition layer 1, transition layer 2 and body layer. These shades are obtained by mixing metal oxide into the zirconia powder. In order to achieve different shades the amount and composition of the metal oxides vary, which may lead to different aging behavior.

As the influence of different colors on the aging behavior of Y-TZP has not been tested yet, one aim of this study was to investigate, whether the aging behavior of shaded zirconia is influenced by the color in comparison to two conventional unshaded zirconia ceramics. The first hypothesis of this study was that the ratio of phase transformation on the surface and the flexural strength are not significantly influenced by the coloring method.

Many studies concerning zirconia utilize the piston-on-three-ball technique to investigate the flexural strength of the zirconia (Amaral et al., 2013; Ebeid et al., 2014; Hallmann et al., 2016; Nakamura et al., 2016a; Özcan et al., 2013; Pereira et al., 2015a; Song et al., 2013; Su et al., 2017; Sulaiman et al., 2015; Yilmaz et al., 2007). Three-point bending tests (Egilmez et al., 2014; Moon et al., 2016) and four-point bending tests (Abi-Rached et al., 2015; Flinn et al., 2017; Michida et al., 2015; Passos et al., 2015) can also be found in literature. However, Börger et al. (2002) investigated the stress distribution in a ball-on-three-balls test and concluded that this test is independent from the surface roughness of the tested specimens. This is also a major advantage for the investigation of the LTD of zirconia, since the aging resistivity of zirconia is highly dependent from the surface treatment (Inokoshi et al., 2015). However, the measurement technique might influence the measured flexural strength. For example it was stated that for ceramics the flexural strength measured by a three-point-bending test is higher than by a four-point-bending test (Rodrigues et al., 2008).

Accordingly, another aim of this study was to investigate if there is any difference between the biaxial flexural strength measured by the ball-on-three-balls test and the piston-on-three-balls test. The second hypothesis proclaims that there is no difference in the results of these two measurement methods.

2. Materials and methods

In this study the four different layers of Katana Zirconia ML (Kuraray, Tokyo, Japan), e.max ZirCAD (Ivoclar Vivadent, Schaan, Liechtenstein) and LAVA Plus (3M ESPE, Seefeld, Germany) were investigated. All investigated materials and their corresponding group codes and LOT numbers are given in Table 1. For each investigated material 103 discs with a diameter of 12 mm and a thickness of 1.2 mm were produced. Both surfaces of the discs were ground in two steps with SiC paper under ample cooling with water. In the first step 600 grit and finally 1200 grit paper was used. The specimens of each group were randomly distributed into 5 subgroups of 20 specimens according to the aging procedure for flexural strength tests and one subgroup containing 3 specimens for XRD measurements.

Table 1
Group codes of the used materials and their LOT numbers.

Group code	Material	Company	LOT number
Kat 1	Katana Zirconia ML, enamel layer	Kuraray	DJAQJ
Kat 2	Katana Zirconia ML, transition layer 1	Kuraray	DJAQJ
Kat 3	Katana Zirconia ML, transition layer 2	Kuraray	DJAQJ
Kat 4	Katana Zirconia ML, body layer	Kuraray	DJAQJ
Lava	LAVA Plus	3M ESPE	584237
Zir	e.max ZirCAD	Ivoclar Vivadent	U26548

2.1. Aging process

The aging was performed in an autoclave (CS, WEBECO, Bad Schwartau, Germany) at 134 °C in water vapor atmosphere at a pressure of 0.2 MPa. The first subgroup for flexural strength tests was not treated in this way and served as control group. The other four subgroups for these tests underwent this treatment for 5, 10, 15 and 20 h according to the subgroups the specimens.

2.2. Flexural strength tests

After the treatment in the autoclave each of the five subgroups was randomly distributed into two groups of same size (N = 10). Half of the specimens were tested by a standard biaxial flexural strength test using the piston-on-three balls technique. The diameter of the cylindrical piston was 1.5 mm. The specimens were placed centrally on three balls with a diameter of 3.4 mm that were placed equidistantly on a circle with a diameter of 10 mm. The flexural strength was calculated from the recorded fracture load using the following formulas:

$$S = \frac{-0.2387P}{d^2}(X - Y)$$

$$X = (1 + \upsilon)ln\left(\frac{r_2}{r_3}\right)^2 + \frac{1 - \upsilon}{2}\left(\frac{r_2}{r_3}\right)^2$$

$$Y = (1 + \upsilon)\left(1 + ln\left(\frac{r_1}{r_3}\right)^2\right) + (1 - \upsilon)\left(\frac{r_1}{r_3}\right)^2$$

S: biaxial flexural strength (MPa); P: fracture load (N); d: specimen disk thickness at fracture origin (mm). υ is Poisson's ratio (0.25), r_1 is the radius of the support circle, r_2 is the radius of the loaded area, and r_3 is the radius of the specimen.

The ball-on-three balls technique was used for the other half of the specimens. All balls were made of stainless steel and had a diameter of 8 mm. The flexural strength was calculated according to Börger et al. (2002) using the applet of the ISFK of the Montan University Leoben (Austria) (Funktionskeramik, 2011).

Both testing methods for flexural strength were conducted with a universal testing machine (Z010, Zwick, Ulm, Germany) with a crosshead speed of 0.5 mm/min. The results of both methods were statistically evaluated separately by one-way ANOVA followed by a Games-Howell test for pairwise post-hoc comparison. In this context the Levene test revealed that the variances of the groups were significantly different ($p < 0.05$).

2.3. X-ray diffraction (XRD)

The three specimens of the sixth subgroup were used for XRD measurements with a PTS 3000 (Seifert). The specimens were investigated before and after applying the aging process for 5, 10, 15 and 20 h to the same specimens. The measurement range for 2θ was 20° to 40° with a step size of 0.04° and a measurement time of 10 s. The whole