

Influence of Low-Temperature Environmental Exposure on the Mechanical Properties and Structural Stability of Dental Zirconia

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Abstract

Purpose: The effect of dental fabrication procedures of zirconia monolithic restorations and changes in properties during low-temperature exposure in the oral environment is not completely understood. The purpose of this study was to investigate the effect of procedures for fabrication of dental restorations by low-temperature simulation and relative changes of flexural strength, nanoindentation hardness, Young's modulus, surface roughness, and structural stability of yttria-stabilized zirconia.

Materials and Methods: A total of 64 zirconia specimens were prepared to simulate dental practice. The specimens were divided into the control group and the accelerated aging group. The simulated group followed the same procedure as the control group except for the aging treatment. Atomic force microscopy was used to measure surface roughness. The degree of tetragonal-to-monoclinic transformation was determined using X-ray diffraction. Nanoindentation hardness and modulus measurements were carried out on the surface of the zirconia specimens using a nanoindenter XP/G200 system. The yttria levels for nonaged and aged specimens were measured using energy dispersive spectroscopy. Flexural strength was determined using the piston-on-three-ball test. The *t*-test was used to determine statistical significance.

Results: Means and standard deviations were calculated using all observations for each condition and evaluated using a group *t*-test (p < 0.05). The LTD treatment resulted in increased surface roughness (from 12.23 nm to 21.56 nm for Ra and 15.06 nm to 27.45 nm for RMS) and monoclinic phase fractions (from 2% to 21%), with a concomitant decrease in hardness (from 16.56 GPa to 15.14 GPa) and modulus (from 275.68 GPa to 256.56 GPa). Yttria content (from 4.43% to 4.46%) and flexural strength (from 586 MPa to 578 MPa) were not significantly altered, supporting longer term in vivo function without biomechanical fracture.

Conclusion: The LTD treatment induced the tetragonal-to-monoclinic transformation with surface roughening in zirconia prepared using dental procedures.

Zirconia is a polymorphic material that exists in three crystal structures: monoclinic, tetragonal, and cubic.¹ Pure zirconia is monoclinic from room temperature to 1170° C.² Above that temperature, it transforms into the tetragonal phase. At a temperature of 2370° C, zirconia transforms into a cubic phase. The tetragonal phase may be stabilized by adding small amounts of metallic oxides, such as Y_2O_3 , MgO, CeO, or CaO, but it is, in fact, metastable at room temperature. Processes such as grinding and sandblasting can trigger the tetragonal-to-monoclinic phase transformation.^{3,4} This transformation is accompanied by a 3% to 4% volume expansion that induces compressive stresses, thereby closing the crack tip and preventing further propagation.⁵ This characteristic, known as transformation toughening, leads to the increased fracture

strength and fracture toughness of Y-TZP ceramics compared with other dental ceramics.²

Zirconia is inert and has high-temperature applications. Kobayashi et al reported that the material can transform at approximately 250° C.⁶ This low-temperature transformation can have serious implications for clinical applications of zirconiabased materials. For example, more than 600,000 zirconia femoral heads were implanted worldwide, mainly in the United States and Europe. The zirconia manufacturers assumed that the problem of transformation was irrelevant until 2001, when several hundred hip prosthesis failures were reported sooner than anticipated.³

Transformation toughening caused by tetragonal-tomonoclinic transformation is desirable in the presence of a crack because the excess volume caused by the transformation reduces crack propagation. This transformation also occurs in the presence of hydrothermal stress such as water, blood, and synovial fluids over a long period of time, and is considered unfavorable because the excess volume is not compensated by crack space and causes micro- and macrocracking, reducing the mechanical properties. This phenomenon is called lowtemperature degradation (LTD) or aging.

The following features for LTD have been established:³ (1) the tetragonal-to-monoclinic transformation starts on the surface and progresses into the material; (2) reduction in grain size and/or increase in concentration of stabilizer reduces transformation rate; (3) greater tetragonal-to-monoclinic transformation when the yttria-stabilized zirconia is aged in water (at a temperature of 65°C to 120°C) compared with air,⁷ and (4) degradation is time dependent and proceeds more rapidly at temperatures between 200°C and 300°C. LTD is responsible for grain push-out,^{8,9} increased surface roughening,^{10,11} increased wear, decreased hardness,¹² and loss of strength (20% decrease in the fracture strength),¹³⁻¹⁶ which may lead to performance deterioration.¹⁷

Dental restorations function in an aggressive environment with saliva, pH changes, and cyclic loading. Coping/framework has dual protection against aging through veneering porcelain on the external surface and luting cement on the internal surface; however, it has been shown¹⁸ that common luting cements absorb water via dentinal tubules, thereby exposing the zirconia coping to moisture, which, in turn, may lead to aging problems over a shorter period of time than anticipated. It is critical to determine if LTD of zirconia prepared using dental procedures occurs in conditions that simulate extended use in the oral environment. The surface finish of the restoration is critical, and fabrication with CAD/CAM technology and veneering of the ZrO₂ framework is a multistep process with the potential for operator variability. These surface treatments may affect the long-term stability and the aging sensitivity of zirconia and the success of the restoration. Both the aging environment and fabrication details for the dental zirconia restoration are very different from those of zirconia femoral heads. Thus, there is a need for basic material studies of zirconia for dental applications.

The purposes of this study were to (1) investigate the effect of aging treatment on the flexural strength, nanoindentation hardness, Young's modulus, surface roughness, and structural stability of yttria-stabilized zirconia (Y-TZP); and Table 1 Chemical composition of yttria-stabilized zirconia

Chemical component	Mol%
Zirconium dioxide (ZrO ₂)	92.642
Yttrium oxide (Y_2O_3)	5.3
Hafnium oxide (H _f O ₂)	1.78
Aluminum oxide (Al_2O_3)	0.253
Others	0.025

Table 2 Material specifications of yttria-stabilized zirconia

Specification	Value
Density ($ ho$)	6.05 g/cm ³
Thermal expansion coefficient (TEC)	$10 \times 10^{-6} \text{ K}^{-1}$
Flexural strength	1200 MPa
Fracture toughness (K _{IC})	8 MN/m ^(1/2)
Modulus of elasticity (E)	210 GPa
Grain size	0.35 μm
Vickers hardness (HV 10)	1200 Hv
Melting point	2680°C
Shrinkage after sintering	20.8%

(2) determine the depth distribution of the transformation. The hypotheses were that the aging treatment would cause yttria loss and decrease the tetragonal phase stability of the dental zirconia, leading to tetragonal-to-monoclinic transformation, increased surface roughness, lower hardness, and lower modulus of elasticity, and that the flexural strength would not be affected.

Materials and methods

Sixty-four zirconia disk-shaped specimens (11.78 mm diameter, 1.35 mm thick) were fabricated by the TurboDent system (Pou-Yuen Technology Co., Ltd., Fusing Township, Taiwan). The disks were oversized to compensate for 20.8% shrinkage during sintering. Manufacturer data on the chemical composition and material specifications of this zirconia are listed in Tables 1 and 2.

The specimens were prepared to simulate laboratory dental procedures for the fabrication of monolithic zirconia restorations. They were finished using the Exact-Micro-Grinding System (Model number 300-310, Norderstedt, Germany) with $35 \,\mu\text{m}$ diamond lapping film (Allied High Tech Products, Inc., Rancho Dominguez, CA) at standard speed and pressure without water. The specimens were colored with A1 dyeing liquid according to the manufacturer's instructions. The dyed disks were then sintered to full density as recommended by the manufacturer (the TurboDent system requires a 7.5-hour firing cycle at maximum temperature of 1530°C to include heating and cooling). The specimens were further ground with 35 μ m continuous diamond lapping film, and then were polished with 0.5 μ m diamond lapping film. Each disk was then fired at a temperature of 910°C to restore the tetragonal phase, as documented in the literature.19,20

The specimens were divided into two equal groups – the control group and the accelerated aging group, with 32 specimens for each group. Four specimens were used for XRD, surface roughness, hardness, modulus, and elemental analysis, and 28 specimens for flexural strength for each group (control and aged specimens). Multiple measurements were made in each specimen for XRD (1 data point), surface roughness (16 data points), hardness (97 data points), modulus (97 data points), and elemental analysis (12 data points).

Aging process

A device was used to provide boiling conditions at a constant temperature. The zirconia specimens were placed in a flask containing the artificial saliva and heated to boiling.²¹ The accelerated aging group was boiled (100°C, 7 days in artificial saliva) for aging of the zirconia to simulate long-term LTD in the oral environment.

Atomic force microscopy (AFM)

Surface roughness was determined using a DI3100 microscope (Digital Instruments, Inc., Chapel Hill, NC) in contact mode with oxide-sharpened silicon nitride probes and an average scanning speed of 50 μ m/s without any additional surface preparation. Average roughness (Ra) (the arithmetic average of the profile ordinates within the measured section) and root mean square roughness (RMS) (the root mean square value of the profile ordinates within the measured section) were measured.²²

X-ray diffraction (XRD)

The phase distribution was analyzed on an X-ray diffractometer (Siemens D500 Bruker AXS, Madison, WI). These experiments were conducted primarily with Bragg-Brentano geometry, between 27° and 32° (2 θ), with K α radiation. Scans were performed at 40 kV, 30 mA, step size of 0.005°/step, and a scan time of 8 seconds/step. The tetragonal-to-monoclinic transformation was detected on the top surface of the specimens. The relative amount (X_M) of the transformed monoclinic zirconia in the specimens was calculated from the integral intensities of the monoclinic ($\overline{111}$), (111) and the tetragonal (101) peaks. This characterization was based on the equation proposed by Garvie & Nicholson:²³

$$X_{m} = \frac{I_{111}^{m} + I_{111}^{m}}{I_{111}^{m} + I_{111}^{m} + I_{101}^{t}}$$

The monoclinic fractions were calculated as a function of different X-ray incidence angles $(14^\circ, 10^\circ, 8^\circ, 5^\circ, 3^\circ)$ to determine the depth distribution of tetragonal-to-monoclinic transformation from the surface into the depth of the material after the aging process.

Nanoindentation hardness and Young's modulus

Nanoindentation and Young's modulus measurements were made on the surface of the zirconia specimens using a Nanoindenter[®] XP/G200 (Oak Ridge, TN) system calibrated using Corning 7980 (Corning Incorporated, Canton, NY). A Berkovich diamond indenter with 120° for each facet was used for all the measurements. The loading/unloading rate was 0.3 mN/s. A 10-second hold time at a maximum load, and 10 seconds at 10% of maximum load during unloading were used to minimize thermal drift. The 5×5 matrix was taken for each specimen using 500 nm as maximum penetration depth with 35 nm distance, and the Poisson ratio was 0.25. The data were processed using vendor software to produce load-displacement curves, and the mechanical properties were calculated using Testworks[®] software (MTS Systems Corporation, Eden Prairie, MN).

Scanning electron microscopy (SEM)

Scanning electron micrographs (Philips 515 Scanning Electron Microscope; Philips Electronics, Eindhoven, the Netherlands) of the specimens were obtained with an accelerating voltage of 30 kV. The specimens were coated with carbon for elemental analysis with energy dispersive spectroscopy (EDS) to quantify material composition (yttrium, zirconium, oxygen, aluminum, and hafnium) for the control and aged specimens.

Biaxial flexural strength

Fifty-six specimens were subjected to a biaxial flexural strength test (piston-on-three balls) according to ISO standard 6872 for dental ceramics²⁴ using a universal testing machine (Instron, Satec Systems Inc., Model: Apex T5000, Grove, PA). Each specimen was placed with the treated surface under tension on three 3.18 mm diameter hardened steel balls positioned 120° apart on a support circle with a diameter of 10 mm. A thin plastic sheet (0.05 mm thick) was placed between the punch and the specimen to facilitate an even load distribution. The specimens were loaded with a flat piston with a diameter of 1.5 mm at the center of the specimen at a 0.5 mm/min crosshead speed until failure. The fracture load was recorded (N), and the biaxial flexural strength for each specimen was calculated.

Statistical analysis

The measurements of the various regions of each group were averaged for the control and aged sample sets. The sample size was based on past publications and resources.^{11,25,26} The *t*-test was used to compare the control and aged specimens. Data were evaluated for normality, and, despite the small sample size, there was minimal deviation from normality. Further evaluation using nonparametric Mann-Whitney U procedures were implemented for the small sample size, and the results were similar to the *t*-test. Thus the results are presented as *t*-tests. A *p*-value of 0.05 was considered statistically significant. The power of all comparisons was computed for an alpha = 0.05. The individual points were taken as the unit of analysis and adjusted for the correlation among the specimens. Power was 0.75 to 0.85 for all significant comparisons, which was judged to be adequate.

Results

Elemental analysis (Table 3) using EDS of the control specimens indicated high concentrations of zirconium (Zr) and oxygen (O), and small concentrations of yttrium (Y), hafnium (Hf), and aluminum (Al). The p-value comparing control and

U U	Table 3	Mean and standard	d deviations of the	control and aged	I specimens for a	all characterization	procedures and	flexural strength
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		Mean \pm SD (control specimens)	Mean \pm SD (aged specimens)	<i>p</i> -value
Surface roughness	Ra (nm)	12.23 ± 6.16	21.56 ± 13.39	0.017
	RMS (nm)	15.06 ± 7.23	27.45 ± 16.16	0.009
Amount of monoclinic (%)		2.4 ± 0.6	21.0 ± 2.0	< 0.001
Hardness (GPa)		16.56 ± 0.81	15.14 ± 1.83	< 0.001
Young's modulus (GPa)		275.68 ± 12.26	256.56 ± 21.56	< 0.001
Elemental analysis (wt %)	Zr	80.73 ± 3.18	80.68 ± 2.51	0.966
	0	11.41 ± 3.50	11.45 ± 2.47	0.974
	Y	4.43 ± 0.32	4.46 ± 0.43	0.848
	Hf	3.09 ± 0.27	3.13 ± 0.31	0.739
	Al	0.305 ± 0.158	0.305 ± 0.239	0.999
Flexural strength (MPa)		586.86 ± 71.47	578.31 ± 75.25	0.678

Note: The independent specimen *t*-test was used for all comparisons. Homogeneity of variances was checked in each case, and the appropriate *t*-test (equal or unequal variances) was applied.

aged values comes from a group *t*-test. The aged specimens indicated very similar concentrations without any significant reduction in the concentration of yttrium or zirconium.

The aging treatment resulted in an increase in both the monoclinic fraction and surface roughness. The XRD analysis performed on control specimens indicated a small monoclinic fraction $(2.4 \pm 0.6\%)$; however, the aged specimens showed a substantial amount of monoclinic fraction $(21.0 \pm 2.0\%)$ (Fig 1). The surface roughness was measured, and there was a significant increase in the surface roughness (Ra: 12.23 ± 6.16 nm to 21.56 ± 13.39 nm and RMS: 15.06 ± 7.23 nm to 27.45 ± 16.16 nm) between control and aged specimens.

The hardness and modulus were measured, and there was a significant decrease in the hardness and modulus between the control and aged specimens. The flexural strength was not affected between the control and aged specimens (Table 3).

The distribution of the tetragonal-to-monoclinic phase fraction within the surface can also be measured with XRD. A low incident angle will limit the X-ray penetration depth and will yield data more reflective of the material closest to the surface. Figure 2 shows that as the X-ray incidence angle was decreased from 14° to 3° , the fraction of the monoclinic phase was increased (14°: 20.7%, 10°: 23.6%, 8°: 27.2%, 5°: 31.5%, 3° : 39.3%). This confirms that the tetragonal-to-monoclinic transformation is decreasing from the surface into the depth of the material where the surface has a larger fraction of the monoclinic phase (39.3% at 3°) than the deeper area (20.7% at 14°). These results show that the transformation occurred more at the surface compared to 5 μ m into the bulk zirconia. Thus, the immediate surface, where fracture is most likely to initiate, is most transformed, which could influence flexural strength.

Discussion

Zirconia copings/frameworks are usually fabricated using partially sintered Y-TZP blocks and then subjected to different surface treatments. In our study, a minimal fraction of the monoclinic phase (2%) was detected for the control specimens, as these specimens were exposed to various processing treatments that included final heat treatment to a temperature of 910°C used in porcelain fabrication. This finding is in agreement with several authors who reported that heat treatments in the temperature range of 900°C to 1000°C induce the reverse transformation from monoclinic to tetragonal after aging, grinding, or sandblasting of Y-TZP.^{19,20}

There is no universally accepted mechanism to explain the origins of the tetragonal-to-monoclinic transformation in the presence of moisture, but three mechanisms are proposed in the literature. The first is that water (H₂O) reacts with yttria (Y_2O_3) to form yttrium hydroxide $(Y(OH)_3)$, which depletes the stabilizing oxide sufficiently to cause transformation to the monoclinic phase.²⁷ The second mechanism is water attack of the Zr-O bond, leading to stress accumulation due to movement of -OH into the crystal structure. This motion generates lattice defects that act as nucleating agents for subsequent transformation from the tetragonal-to-monoclinic phase.²⁸ Finally, O_2 – (not OH⁻) from water dissociation fills oxygen vacancies.²⁹ Papanagiotou et al²¹ reported that LTD resulted in a loss of yttria (from 6.76 wt% to 4.83 wt%) when Vita In-Ceram YZ was aged in boiled water for 7 days, which supports the first mechanism. In our study, there was a significant amount of tetragonal-to-monoclinic transformation, but the yttria and zirconia content were unchanged within the sensitivity of EDS (the same experimental method was used by Papanagiotou et al). This apparently contradictory result may be due to the different compositions of zirconia used. Thus, the data lend indirect support to the mechanisms of either attack of the Zr-O bonds or the O_{2-} filling oxygen vacancies.

The strength after LTD is affected by the thickness of the transformed layer on the surface, which is related to the amount of monoclinic phase observed,¹³ and the extension of the cracks. In the present study, there was no significant difference in flexural strength among the specimens (p = 0.678). Furthermore, the flexural strength was not affected by the amount of the monoclinic transformed (from 2% to 21%) after accelerated aging in artificial saliva because tetragonal-to-monoclinic transformation occurred in the external surface only with shallower depth, and the internal flaws were not critical enough to affect the flexural strength. This finding is in agreement with another study.²¹ The values for flexural strength (586.86 ± 71.47 MPa for control specimens and 578.31 ± 75.25 MPa



Figure 1 XRD analysis for the control (A) and aged (B) specimens. Comparison of the relative peak heights shows a substantial increase of monoclinic phase ($\overline{111}$) for the aged specimens compared with the control.

for aged specimens) in this study were much lower than in other studies.^{21,30} Lower values could be due to the less-thanideal specimen dimensions (thick specimens with smaller diameter) used in this study to calculate the flexural strength,

which was still within the minimal accepted flexural strength (500 MPa) as recommended by ISO13356;2008.²⁴ In the present study, a piston-on-three-ball test was used instead of piston-on-ring according to ISO13356;2008 for evaluating the



Figure 2 The fraction of monoclinic phase for different incidence angles $(14^\circ, 10^\circ, 8^\circ, 5^\circ, 3^\circ)$. The 3° incident angle had the highest monoclinic fraction, while the 14° angle had the lowest monoclinic fraction.

biaxial flexural strength because these type of tests are not affected by the contacting conditions; however, the piston-on-ring test will produce a lower value of the maximum tensile stress when there is friction at the contact points, and this will cause overestimation of the tensile strength at the disk.³¹

The phase transformation of artificially aged Y-TZP disks illustrated in the current study increased the surface roughness, and this is comparable to previous studies^{11,15} because the volume expansion (3% to 5%) associated with the tetragonal-to-monoclinic transformation leads to grain pushout and surface uplift, which imparted the surface roughening. Although the roughness of Y-TZP significantly increased with aging, a roughness of Ra = 0.021 μ m and RMS = 0.027 μ m are still considered to be very smooth compared with acceptable surface roughness for bacterial colonization (0.2 μ m)³² and within the range of roughness of 0.25 μ m to 0.5 μ m to be undetected by a patient's tongue.³³

Santos et al¹¹ confirmed a drop in hardness (nanoindentation testing) with the zirconia femoral head (from 18 GPa to 11 GPa) caused by an extensive monoclinic transformation (from 0% to 78%). In the present study, there was a decrease in the hardness (from 16.56 GPa to 15.14 GPa) and modulus (from 275.68 GPa to 256.56 GPa). This happened due to the induced micro-cracks from transformation after artificial aging.²⁹

The specimens of the present study were prepared with procedures similar to those used by $Ardlin^{34}$ and showed similar results for the monoclinic fraction for nonaged specimens (2% or less of monoclinic content), monoclinic fraction for aged specimens (20% to 25% of monoclinic content compared with 21% in the present study), no statistical difference in the flexural strength after aging procedures, and significant changes in the surface roughness after the aging process, although different aging conditions were used. In the present study, artificial saliva was used as the aging environment, because it has a very similar composition of ions to natural saliva and then aged at 100° for 7 days as recommended for aging zirconia to simulate the long-term use in the oral environment.²¹ However, Ardlin used a 4% acetic acid solution at 80°C for 168 hours.

All in vitro simulations were intended to represent known changes experienced in vivo. Y-TZP ceramics used in medicine for arthroplasties showed rapid atomic structure changes, which influenced clinical outcomes. Multiple methods have been developed previously, including temperature-environment-time cycles to simulate changes found in vivo. The current study used the same ceramic used for dental restorations. Prior studies have developed simulations applicable to the oral environment, and this study was designed to best simulate oral conditions and to test changes in atomic structure and mechanical properties including dental procedures routinely used for oral restorations. Therefore, the temperature, environment, and time cycles were selected from prior dental and medical publications specific to in vitro simulations.

The amount of transformation was highest at the surface and decreased into the bulk of the zirconia. Therefore, the surface would be proportionally rougher. Thus, the collection of food debris could be a clinical concern because of this increased surface roughness. The limitation of this study was that the effect of LTD was not tested for wear and influence on opposing dentition. Future research should be conducted to test the effect of the surface topography of monolithic zirconia restorations opposing natural dentition after the aging process, and correlated with in vivo studies to evaluate clinical significance.

Conclusions

Within the limitation of the in vitro simulation aspects of this study, the following can be concluded:

- 1. The LTD treatment of dental zirconia specimens prepared using conditions replicating the fabrication of zirconia monolithic dental restorations induced a tetragonal-to-monoclinic transformation (21%), which was greater at the surface, a significant increase of surface roughness from 12 nm to 22 nm (Ra) (p = 0.017), decreased hardness (17 GPa to 15 GPa, $p \le 0.001$), and decreased surface modulus (276 GPa to 257 GPa, $p \le$ 0.001).
- 2. The flexural strength (586 MPa to 578 MPa) and yttria composition (4.43% to 4.46%) of the bulk zirconia were not significantly altered.

These results show that the procedures for dental restorations did not significantly change the bulk properties of the zirconia, which represents an important factor for longer-term in vivo function without biomechanical fracture.

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