Original Article

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Effect of accelerated aging on phase transformation and flexural strength of yttria-stabilized tetragonal zirconia dental ceramics

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Objectives: The objectives of this study were to investigate the effect of hydrothermal aging on the flexural strength and the transformation behavior of two translucent (Cercon xt ML and Ceramill Zolid FXML) and two conventional (Cercon ht and Ceramill ZI) dental zirconia (Y-TZP).

Materials and Methods: Five bar-shaped specimens (1.5 x 4 x 22 mm) of zirconia in each group were subjected to hydrothermal aging process at 134°C under 0.2 MPa for 0, 10 and 50 hours. After aging, the monoclinic-to-tetragonal phase transformation was evaluated using an x-ray diffraction technique. The alteration in material microstructure was observed using a scanning electron microscope. The flexural strength of all specimens was determined using a four-point bending test. Two-way ANOVA was used to analyze the flexural strength values to find the significant differences among groups at α =.05.

Results: The results showed that there was a significant increase in flexural strength of Ceramill ZI and Cercon ht after autoclaving for 10 hours and 50 hours. The flexural strengths of Ceramill Zolid FXML and Cercon xt ML were not significantly different at all autoclaving times. After 50 hours of autoclaving, the monoclinic phase increased from 0.2 to 69.40 wt% for Ceramill ZI, and from 0 to 70.03 wt% for Cercon ht. There was also no relevant effect of aging on grain size or topography at the surface of all Y-TZPs groups.

Conclusions: Prolonged hydrothermal aging caused an increase in monoclinic phase content in conventional dental zirconia and a significant increase in flexural strength was also observed in this group. However, prolonged hydrothermal aging had limited effect on flexural strength and amount of monoclinic phase change observed in the translucent dental zirconia group.

Keywords: accelerated aging, flexural strength, phase transformation, zirconia

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Introduction

In recent years, the use of ceramic restorations has increased remarkably due to their superior aesthetic appearance, excellent biocompatibility, and the absence of any metal content as compared to other materials. When combining with the well-established CAD/CAM procedures, an increase in demand of dental ceramics regarding the clinical use of these ceramics is recognized from both the dentist and patient. Zirconia-based ceramic materials, especially yttria-stabilized tetragonal zirconia polycrystals (Y-TZP), have been used as a core material for single crowns, dental implant abutments, and fixed partial dentures [1-3]. Zirconia has been shown to be an excellent material for use in the oral cavity. It is chemically stable, and the thermal conductivity is extremely low.

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Two main zirconia restoration categories include zirconia-based with a veneering material and monolithic zirconia restorations. Monolithic zirconia restorations, which are prepared from only zirconia ceramics, have some advantages over veneered-zirconia restorations, such as no need for layering and a simpler and shorter CAD/CAM process [4-5].

First-generation 3Y-TZP contains 0.25 wt % alumina (Al_2O_3) as a sintering aid. They exhibit high opacity because of the inherent birefringence of non-cubic zirconia phases, and light scattering from grain boundaries, pores, and additive inclusions [6]. Due to poor colour properties and less light transmission, veneering with a dental porcelain to ensure the aesthetics of restorations is required [7]. But the veneered Y-TZP has several problems, such as the multistep fabrication process, veneer chipping, and delamination after a duration of service [3, 8-10]. The composition of second-generation 3Y-TZP is refined mainly by reducing the amount of alumina to below 0.05% which does not significantly influence the mechanical properties of the material, and the porosity could be reduced by sintering at a higher temperature [11]. This results in the higher transmission of light with consistent strength [12]. However, the optical property of this second-generation zirconia is still unsatisfactory for use as monoliths in the anterior zone [6].

In order to improve the translucency, later generations of Y-TZPs consist of a higher yttria content of 4 mol% (4Y-ZrO_2) or 5 mol% (5Y-ZrO_2) with a higher amount of nonbirefringent cubic phase. While the cubic structure is isotropic and the scattering of light is reduced, the strength and toughness are decreased because cubic zirconia cannot undergo the t→m phase transformation [13]. However, the more translucent 4Y-ZrO2 and 5Y-ZrO_2 materials are still widely used and indicated for broad usage as anterior crowns and FDPs. More recently, multi-layered zirconia ceramics have been developed as a solution for an esthetic enhancement of monolithic zirconia restorations. These zirconia blanks contain different layers of various translucencies and shades aimed to replicate the shade gradient observed in natural teeth, where the most translucent is observed in the incisal area and the more saturated in chroma and opacity would go towards the gingival region of the crown [4,14].

The low temperature degradation (LTD) of Y-TZP, where the metastable tetragonal phase spontaneously transforms to the monoclinic phase, appears to be a significant concern in dentistry because of the application in the wet oral environment together with a repeated chewing load. The results from few previous studies showed that the mechanical properties of the Y-TZP could be affected as a result of the tetragonal-to-monoclinic transformation, and this process would cause some surface roughening, grains pull-out, and microcracking [15,16]. To date, there is only limited data regarding the LTD of translucent Y-TZP for monolithic restorations.

The objectives of this study were to evaluate the effect of low temperature degradation on flexural strength of two commercially available translucent dental zirconia and two conventional Y-TZP. With the long exposure times that was far exceeding the expected life time of dental restorations, the transformation behavior and the resulting mechanical property could be investigated.

Materials and Methods

Four commercial yttria-stabilized zirconia ceramics were used in this study, i.e., Cercon ht, Cercon xt ML, Ceramill ZI, and Ceramill Zolid FXML. The composition and manufacturers of these materials are presented in Table 1.

Table 1 Chemical composition of dental ceramic used in this study (According to Manufacturers' information)

Manufacturer	Product	Composition wt%	
DENTSPLY SIRONA Inc., NC, USA	Cercon ht (Conventional)	ZrO_2	Not identified
		Y_2O_3	5%
		HfO_2	<3%
		Al_2O_3, SiO_2	<1%
DENTSPLY SIRONA Inc., NC, USA	Cercon xt ML (Translucency)	ZrO_2	Not identified
		Y_2O_3	9%
		HfO_2	<3%
		Al_2O_3oxide, SiO	<2%
Amann Girrach® Amann Girrbach GmbH Pforzheim, Germany	Ceramill ZI (Conventional)	$ZrO_2 + HfO_2 + Y_2O_3$	≥99.0%
		Y_2O_3	4.5–5.6%
		HfO_2	≤5%
		Al_2O_3	≤0.5
		Other oxides	≤1
Amann Girrach®	Ceramill Zolid FX ML (Translucency)	$ZrO_2 + HfO_2 + Y_2O_3$	≥99.0%
		Y_2O_3	8.5–9.5%
		HfO_2	≤5%
		Al_2O_3	≤0.5
		Other oxides	≤0.1

Bar-shaped specimens of each material were prepared according to ISO 6872: 2015. The bar specimens were cut from a pre-sintered block using a low-speed cutting machine and diamond-coated disk (Isomet®, BUEHLER®, Ilinois, USA) under water lubrication. All specimens were wet-polished using a grinding machine through 2000-grit silicon carbide (SiC) abrasive papers. The four sharp edges of each specimen were removed at 45° angle with 600-grit SiC paper. All specimens were sintered at a maximum temperature according to the program provided by the manufacturers. Final dimensions of all ceramic specimens were approximately 1.5 mm x 4.0. mm x 22.0 mm.

The specimens were artificially aged in an autoclave (Hiclave, HVA85, Hirayama, Japan) at 134°C under 2 bar pressure. (n=10 per group; at 0, 10 and 50 hours). The monoclinic phase transformation due to hydrothermal aging was determined using an x-ray diffraction technique with CuKα 1.5418 Å, at 40 kV, and 40 mA (D8, Bruker, Karlsruhe, Germany). The diffraction data were collected conventionally in the 2θ range of 20 to 80 degrees, with a step size of 0.01 degrees and a counting time of 1s/step. Three specimens from each group were analyzed with an x-ray diffractometer. Monoclinic phase fraction was determined from XRD data using the Match phase identification software version 3.3 (Crystal Impact, Bonn, Germany) with the standard models recorded in the Crystallography Open Database (COD) and Rietveld refinement method. The refined parameters were scale factors, specimen displacement, unit cell parameters, background, profile shape, overall isotropic displacement, and preferred orientation.

After hydrothermal aging, the flexural strength of all aged specimens was determined using a 4-point bending test (10 mm inner span, 20 mm outer span) according to ISO 6872:2015. A 4-point bending fixture was set up on a universal testing machine (Intron5566, Instron Ltd, Buckinghamshire, England). The load was applied on the specimen at the crosshead speed of 0.5 mm/min. The flexural strength was calculated using the following equation.

$$\sigma = \frac{3PL}{4wt^2}$$

where

σ = Maximum flexural strength (MPa)

P = Total applied load causing fracture (N)

L = Distance between lower supports (mm)

W = Width of the specimen at fracture (mm)

t = Thickness of the specimen (mm)

After flexural test, the representative fractured specimens from each group were prepared for the fracture surface evaluation using a scanning electron microscope (SEM, JSM-5410LV, Tokyo, Japan). All specimens were cleaned in an ultrasonic cleanser with absolute ethanol for 10 min and then air-blow dried, coated with gold in a sputter coater (Polaron SC7620, Quorum Technologies Ltd., East Sussex, UK). An SEM observation was made on the fracture surfaces and the outer surfaces of 3-5 representative specimens from each group.

Statistical Analysis

The Shapiro-Wilk test was used to test the normality of the flexural strength data obtained from the experiment and homogeneity of variances was determined using the Levene's test. Two-way analysis of variance was performed to determine if statistically significant differences of the mean values of flexural strength existed between four materials and various aging times, at α=0.05.

Results

Flexural strength

The mean flexural strengths with standard deviations of four dental zirconia are shown in Table 2. The results from the Shapiro-Wilk test showed that the flexural strength values of Ceramill ZI, Ceramill Zolid FX ML, Cercon ht and Cencon xt ML were normally distributed (p>0.05). The homogeneity of variances was confirmed using the Levene's test.

Table 2 The flexural strength of zirconia materials at control, 10 and 50 hours of autoclaving

Zirconia materials	Flexural strength (MPa)		
	control	10 hours	50 hours
Ceramill ZI (conventional)	724.9 (63.8) $^{(A,a)}$	909.9 (116.8) $^{(B,c)}$	879.4 (103.6) $^{(B,e)}$
Ceramill Zolid FXML (translucent)	464.4 (93.7) $^{(C,b)}$	485.6 (33.5) $^{(C,d)}$	447.3 (42.9) $^{(C,g)}$
Cercon ht (conventional)	705.9 (36.1) $^{(D,a)}$	908.3 (92.4) $^{(E,c)}$	943.7 (71.7) $^{(E,f)}$
Cercon xt ML (translucent)	438.4 (38.0) $^{(F,b)}$	429.0 (30.9) $^{(F,d)}$	408.1 (53.9) $^{(F,g)}$

the different capital letter superscripts represent statistical difference between different autoclaving times for each material

the different lowercase letter superscripts represent statistical difference between different materials at each autoclaving time

The results from two-way ANOVA showed that autoclaving time and type of material had an effect on the flexural strength of zirconia-based materials used in this study, with an interaction between both factors. The simple effect analysis was performed to determine the effect of each factor at the different levels of another factor. The significant increase in the flexural strengths was detected for Ceramill ZI and Cercon ht after autoclaving to 10 hours and 50 hours. ($p<.05$) The flexural strengths of Ceramill Zolid FXML and Cercon xt ML were not significantly different at all autoclaving times. ($p>.05$) Comparing between materials, the flexural strengths between Cercon and Ceramill zirconia were comparable for both conventional (Ceramill ZI and Cercon ht) and translucent group (Ceramill Zolid FXML and Cercon xt ML) as shown in Table 2 and Figure 1.

XRD Phase analysis

The x-ray diffraction patterns of all zirconia-based materials are shown in Figure 2. Monoclinic peaks in the control group of all materials were negligible, but the peak intensity was amplified after an aging time was increased to 10 and 50 hrs in Ceramill ZI and Cercon ht (Figure 2). For Ceramill Zolid FXML and Cercon xt ML, the XRD patterns of all aged specimens were similar to those obtained from the control group. (Figure 2)

The phase fractions of all zirconia-based materials are shown in Table 3. The amount of tetragonal phase was higher in Ceramill ZI and Cercon ht, ranging between 84.9 to 86.1% at the control time. The cubic phase of Ceramill Zolid FXML and Cercon xt ML was 51.1-57.1 %. The amount of monoclinic phase was increased from 0.8 to 70.5 % for Ceramill ZI, and from 0 to 70.4 % for Cercon ht after 50 hours of autoclaving (Table 3). There was a slight change in the amount of monoclinic phase in Ceramill Zolid FXML and Cercon xt ML, which increased from 0 to 2.4 wt% and 0 to 2.8 wt%, respectively, after 50 hours of autoclaving.

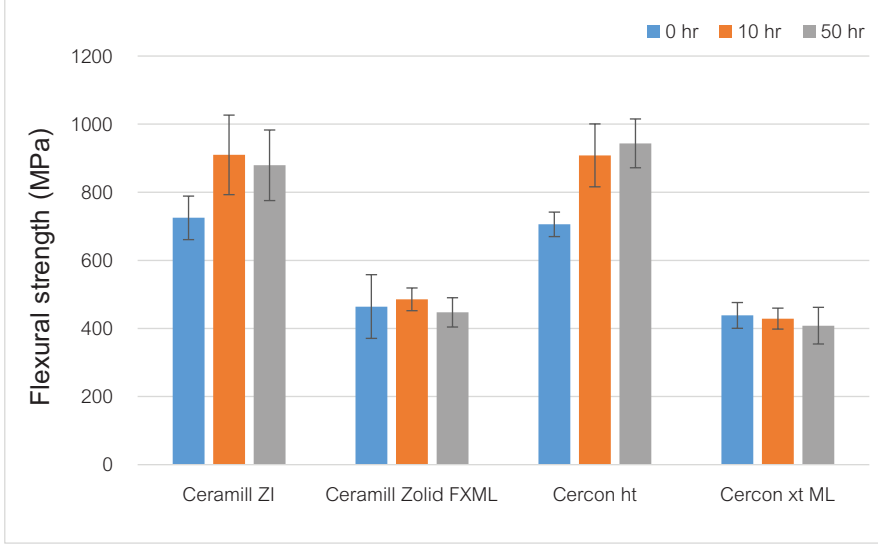


Figure 1 Flexural strength of zirconia-based material at different autoclaving times

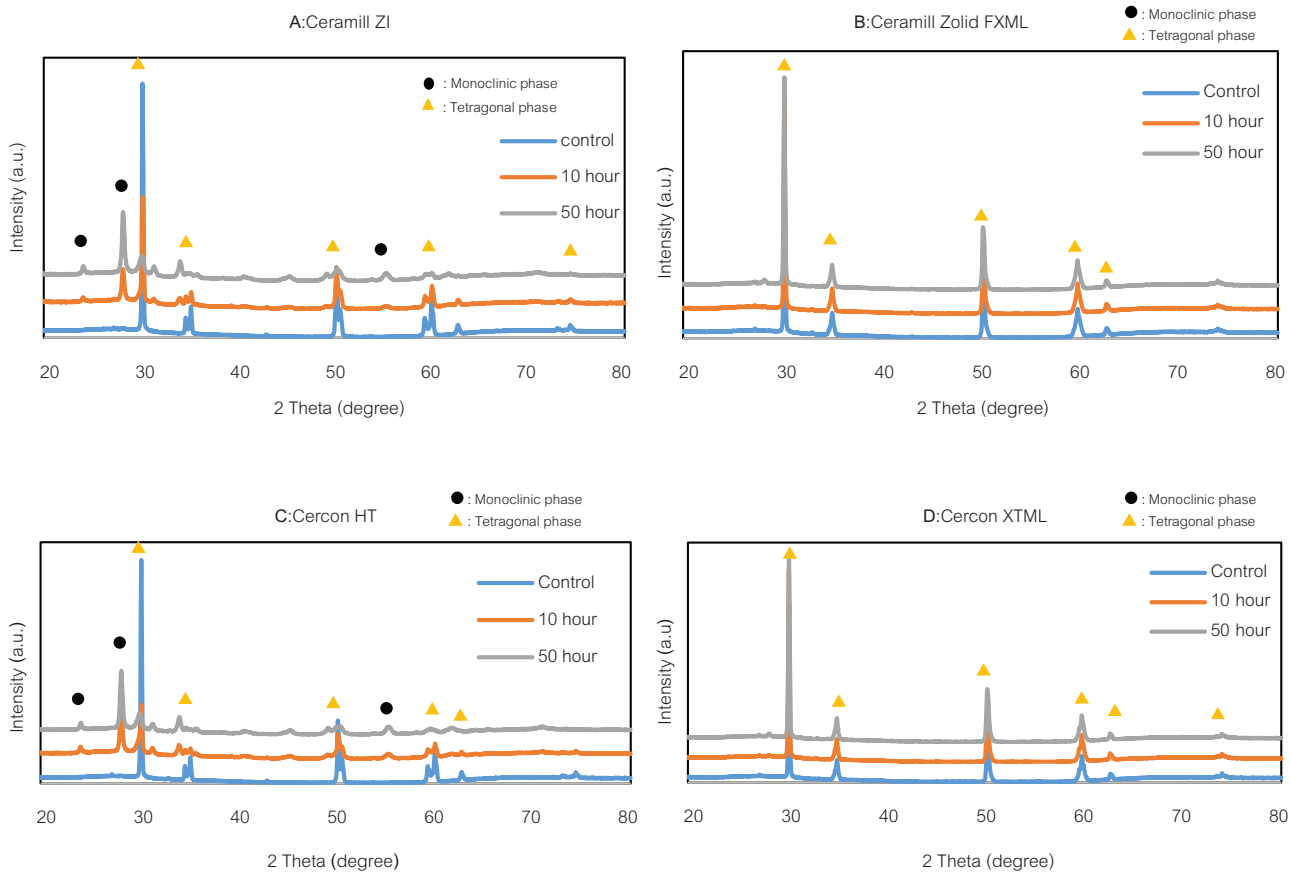


Figure 2 X-ray diffraction patterns of (A) Ceramill ZI, (B) Ceramill Zolid FXML, (C) Cercon ht and (D) Cercon xt ML

Table 3 The phase fraction and reference details of all zirconia materials

Materials	Phase Fraction (%Tetragonal/Cubic/Monoclinic)		
	0 hour	10 hour	50 hour
Ceramill ZI	86.1/13.1/0.8	63.5/11.2/25.3	26.1/3.4/70.5
Ceramill Zolid FXML	42.9/57.1/0	40.6/59.4/0	40.6/57.0/2.4
Cercon ht	84.9/15.1/0	49.5/9.4/41.1	21.4/8.2/70.4
Cercon xt ML	48.9/51.1/0	50.1/48/1.9	55.7/41.5/2.8
Reference database used COD-Inorg 2021.06.14			
Example of Entry number for each phase:			
Tetragonal Zr; 96-152-1478, 96-152-6428			
Cubic Zr; 96-500-0039, 96-210-1235, 96-152-8645			
Monoclinic Zr; 96-900-7449, 96-152-2144			

Surface morphology observation

The result from scanning electron microscopy showed that aging in an autoclave appeared to have no relevant effect on grain size or topography at the surface of all zirconia groups (Figure 3). However, the degradation layer, approximately 10 μm in thickness, was observed on the fracture surfaces of 50-hour autoclaving specimens in the conventional zirconia groups (Figure 4). The spongy-like microstructure at the near-surface was clearly distinguished from the normal feature of zirconia microstructure. From the results obtained from an energy dispersive spectroscope (EDS) analysis (Figure 5), the yttrium content in this transformed was reduced to 0 wt%.

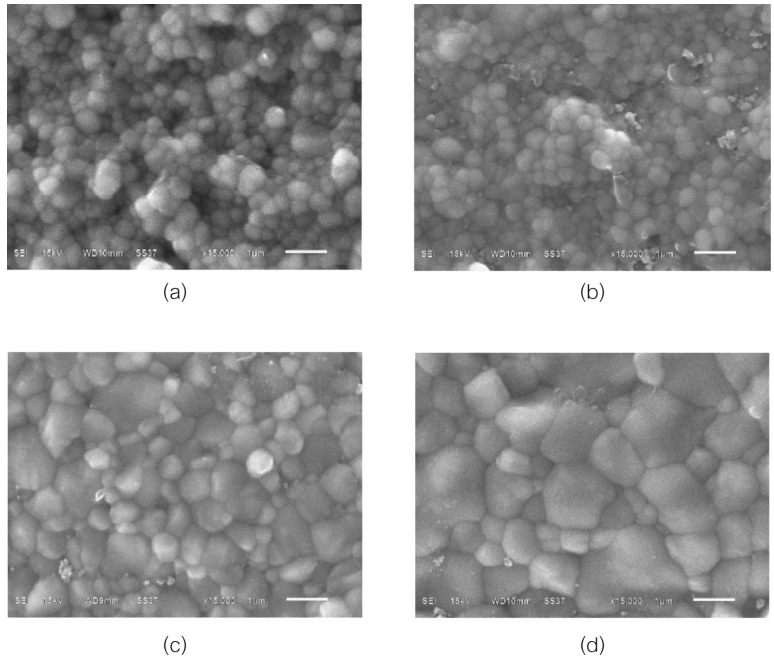


Figure 3 The representative SEM images of (a) Ceramill ZI, (b) Ceramill Zolid FXML, (c) Cercon ht and (d) Cercon xt ML (control group)

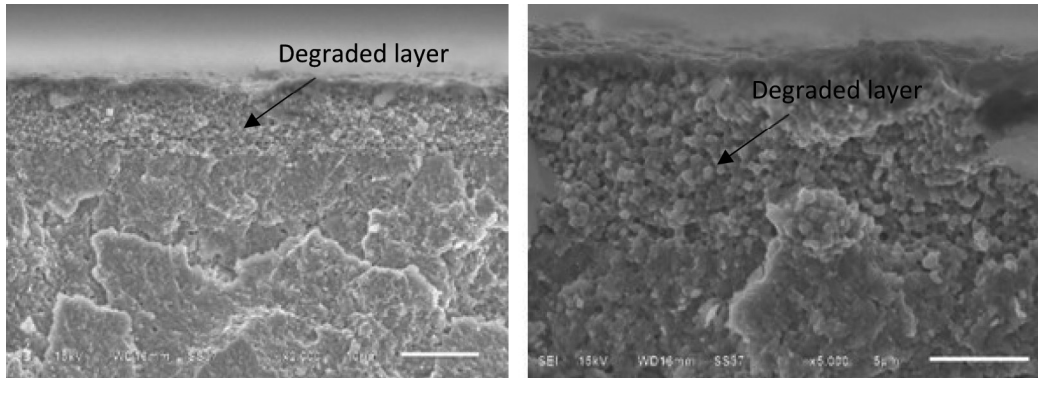
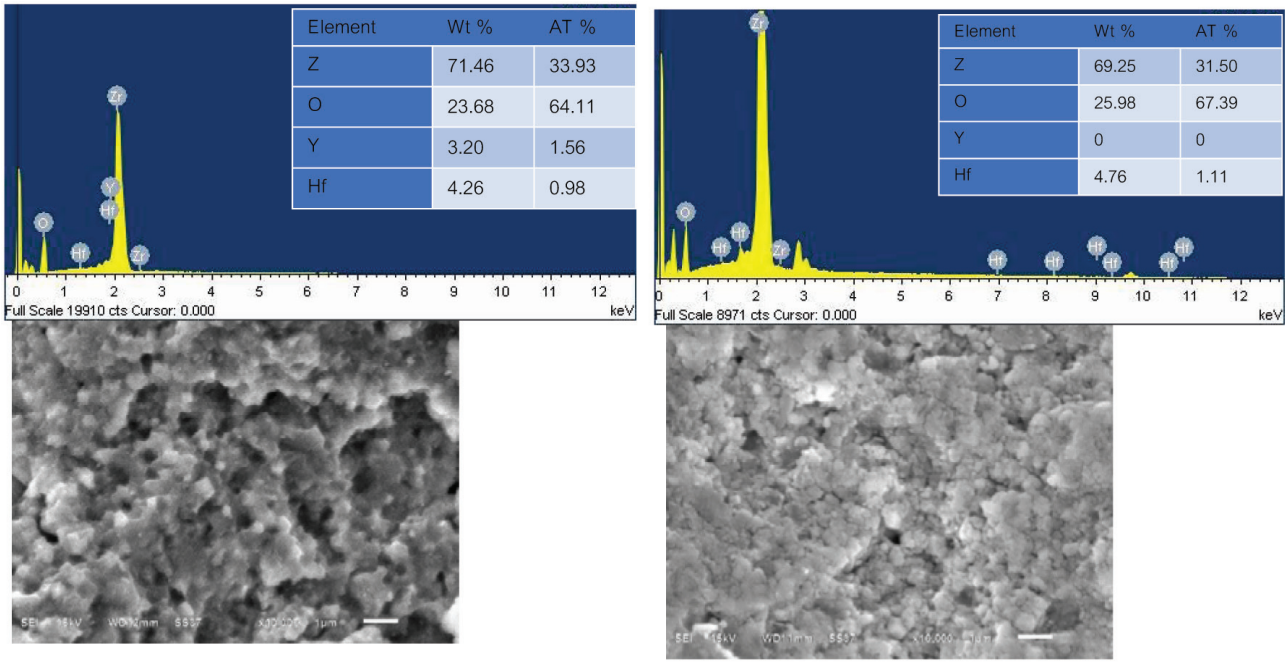


Figure 4 The representative SEM images Ceramill ZI after autoclaving for 50 hours : (a) a fracture surface, (b) degraded layer at the surface of the specimen



(a) (b)

Figure 5 SEM–EDS analysis of composition of (a) Ceramill ZI 0 hour and (b) Ceramill ZI 50 hour

Discussion

Low temperature degradation (LTD) or aging of zirconia is defined as the spontaneous t→m transformation occurring over time in water, and it is unlike phase transformation toughening which the t–m transformation is triggered by the local stress produced at the tip of an advancing crack [15,17]. In the presence of water or water vapours, aging occurs by a slow transformation at the surface from tetragonal to a stable monoclinic phase. The transformation of one grain causes the stresses on the neighbouring grains, and microcracking is generated. This provides a path for the water to penetrate down into the specimen and exacerbates the transformation, which progresses from neighbor to neighbor [18].

It has been shown that the main factors affecting the LTD are (i) the stabilizer type and content, (ii) the residual stress, and (iii) the grain size [1]. Because zirconia materials used in this study were stabilized with Y_2O_3, and the grain sizes of these materials depended partly on the amount of doped Y_2O_3. Even though the grain size of Ceramill groups appeared to be smaller than that of Cercon group, as shown in Figure 3, the aging pattern of these two brands seemed to be comparable for both the conventional and translucent groups. For the residual stresses, some dental laboratory procedures such as, sandblasting, fast cooling rates, coupling with mismatch in coefficient of thermal expansion materials (CTE) could generate some stresses and cause t→m phase transformation [1,18-21]. In addition, the t→m phase transformation also induced some compressive stresses within the transformation area [22].

In this study, the long exposure times as 10 and 50 hours of autoclaving were used, and they were far exceeding the expected life time of dental restorations, which Chevalier proposed that autoclaving for 1 hour at 134°C, at a 2 bar of zirconia resembled the actual exposure in the oral environment for three to four years [18]. However, the transformation behavior and the resulting mechanical property could be investigated deliberately. For the mechanical property, aging for such a long time appeared to enhance the fracture resistances of dental zirconia. The significant increase in flexural strength was as a result of the compressive stress arisen from the extensive t→m transformation at the surface after hydrothermal aging [22]. From the XRD results, an increase in the monoclinic content was profound, from 0.8 to 70.5 % for Ceramill ZI, and from 0 to 70.4 % for Cercon ht after 50 hours of autoclaving. Though the changes in outer surface morphology was not clearly observed, the porous-like zone parallel to the external surface was evidently detected on the fracture surfaces of 50-hour aging specimens (Figure 4). This layer was approximately 10 μm in depth. Despite the different appearance, it did not appear to enhance the growth of the fracture-initiating flaw. As a result, the fracture pattern was comparable in all tested groups. However, the complete depletion of Y_2O_3 at the aging zone was noticeable, and the mechanism of the Y_2O_3 reduction was unclear. The possible explanation could be the interaction between the OH^- from water and Y_2O_3, which caused the dissociation of Y_2O_3 molecules [17, 23]. Another reason could be the oxygen vacancy annihilation coupled with the reduction of Y_2O_3 that led to the destabilization of the tetragonal zirconia [24]. This Y_2O_3 reduction could activate more t-to-m transformation, especially in the 3Y-ZrO_2 groups. The high Y_2O_3 content in 5Y-ZrO_2 was an important factor for the LTD resistance even a slight increase in monoclinic fraction was observed in these groups. The reason for a more tolerance to phase transformation was suggested to be the higher oxygen vacancy number of a cubic phase [15]. However, some tetragonal phase was still affected by the extensive hydrothermal aging.

For 5Y-ZrO_2, there was no significant difference in both flexural strength and monoclinic phase content after a long autoclaving time. The cubic phase fraction of Ceramill Zolid FXML was 57% while the cubic phase fraction of Cercon xt ML was 51% before autoclaving. The reason is well published that an increase in cubic zirconia is the explanation for the resistance in aging effects because the cubic phase would not undergo the phase transformation [25-26]. For a multilayer translucent zirconia used in this study, the variation in Y_2O_3 content seemed to affect the mechanical property insignificantly because the composition of these materials was 5Y-ZrO_2, and the multi-layer structure was a shade-gradient arrangement [27-28]. Unlike the strength-gradient zirconia, which are the combination of 3Y-TZP/5Y-TZP or 4Y-TZP/5Y-TZP, that the mechanical property would vary depending upon the Y_2O_3 content in each layer [29].

The low temperature degradation of dental zirconia using the hydrothermal aging can cause the t→m transformation, and the characteristics of the degradation process could be investigated. In dental applications, stress-induced t→m transformation using a mechanical fatigue test has also been used to examine the effect of this phase transformation. Even the mechanical fatigue could cause less phase transformation than the accelerated hydrothermal aging, but the surface degradation with grain pullout and

crack generation also occurred [30]. An increase in the surface roughness may lead to the adherence of microbes onto the zirconia surface, and this surface roughness is also conducive to the wearing of the natural opposing tooth structure [31].

The results from few previous studies showed that the t→m transformation would not significantly affect the longevity of dental zirconia. [30,32] Due to the limited information on the long-term behavior of dental zirconia in the oral environment, coupled with the complexity and rapid modification of zirconia composition and microstructure, future research is required regarding the failure related to the t→m transformation of these zirconia-based materials.

Conclusion

Within the limitations of this study, some conclusions could be drawn as follows,

1. Prolonged autoclaving time could increase the phase transformation of yttria-stabilized tetragonal zirconia (3Y-TZP) dental ceramic, and the monoclinic content was increased significantly after 10 and 50 hours of autoclaving.

2. Prolonged autoclaving between 10 to 50 hours could significantly enhance the flexural strength of yttria-stabilized tetragonal zirconia (3Y-TZP) dental ceramic.

3. Various autoclaving times had an insignificant effect on the flexural strength and the phase transformation of 5Y-ZrO_2 tested in this study.

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