



Evaluation of fracture resistance of zirconia monolithic restorations: an in vitro study

Avaliação da resistência à fratura de restaurações monolíticas de zircônia: um estudo in vitro

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ABSTRACT

Objective: was to evaluate fracture resistance of zirconia monolithic restorations after aging procedures. **Material and methods:** Monolithic translucent zirconia 3-unit FPDs were fabricated using Cerec inLab CAD/CAM system on 2 stainless steel dies with a uniform 120 degrees circumferential deep chamfer finish line of 1 mm width. FPDs were divided into 2 groups, first group (Group A) was subjected to aging procedures in an autoclave at hydrothermal conditions 134 °C /2 bars for 5 hours. Second group (Group B) was not subjected to any aging procedures (control group). All specimens of each group were loaded compressively in a universal testing machine at cross head speed 0.5 mm/min until fracture occurred. The percentage of monoclinic (*m*) phase was detected by XRD device. Scanning electron microscope (SEM) was used to examine the fractured surfaces for Aged TZI (Group A) and Non-aged TZI (Group B). Student's t-test was used to compare between fracture resistances of both groups. The significance level was set at $P \leq 0.05$. **Results:** The fracture resistance mean (SD) values of non-aged TZI (Group B) was 2406.9 ± 306.8 N which showed statistically significantly higher than that of aged group (Group A) which was 1964.5 ± 234.5 N. The percentage of monoclinic (*m*) phase detected by XRD device software in non-aged TZI (Group B) was nearly 0 weight % and in aged TZI (Group A) was about 42 weight %. **Conclusions:** Accelerated artificial aging decreases the fracture resistance of monolithic TZI FPDs.

KEYWORDS

Artificial aging; Fracture resistance; Monolithic zirconia.

RESUMO

Objetivo: Foi avaliar a resistência à fratura de restaurações de zircônia monolítica após procedimentos de envelhecimento. **Material e Métodos:** 3 unidades de FPDs de zircônia monolítica translúcida foram fabricadas utilizando o sistema Cerec inLab CAD/CAM com 2 matrizes de aço inoxidável com uma linha de acabamento de chanfro profundo circunferencial de 120 graus de largura de 1 mm. As FPDs foram divididos em dois grupos, primeiro grupo (Grupo A) foi submetido a procedimentos de envelhecimento em autoclave em condições hidrotérmicas de 134 °C /2 bars por 5 horas. O segundo grupo (Grupo B) não foi submetido a nenhum processo de envelhecimento (grupo controle). Todos os espécimes de cada grupo foram carregados compressivamente em uma máquina de teste universal na velocidade de 0.5 mm/min até a fratura ocorrer. A porcentagem da fase monoclinica (*m*) foi detectada pelo dispositivo XRD. O microscópio eletrônico de varredura (MEV) foi utilizado para examinar as superfícies fraturadas para TZI envelhecido (Grupo A) e TZI não envelhecido (Grupo B). O teste t de estudantes foi usado para comparar as resistências à fraturas de ambos os grupos. O nível de significância foi estabelecido em $P \leq 0.05$. **Resultados:** Os valores médios de resistência à fratura (SD) do TZI não envelhecido (Grupo B) foi 2406.9 ± 306.8 N, o qual se mostrou estatisticamente significativamente maior do que o grupo envelhecido (Grupo A), que foi 1964.5 ± 234.5 N. A porcentagem da fase monoclinica (*m*) detectada pelo software do dispositivo XRD em TZI não envelhecido (Grupo B) foi próximo de 0% em peso e em TZI envelhecido (Grupo A) foi cerca de 42% em peso. **Conclusão:** O envelhecimento artificial acelerado diminui a resistência à fratura de FPDs TZI monolítico.

PALAVRAS-CHAVE

Envelhecimento artificial; Resistência à fratura; Zircônia monolítica.

INTRODUCTION

Zirconia which is a polycrystalline ceramic with mechanical properties very close to metals [1]. Due to its white opaque color, zirconia cores or frameworks are veneered with feldspathic porcelain for a more acceptable esthetic outcome [2,3]. In clinical service, the most frequent failure of this combination is chipping of the veneer [1,4]. By increasing the translucency of zirconia ceramics, the monolithic translucent zirconia-based restorations become very attractive dental solution [5]. Zirconia based ceramics has three crystallographic forms. At room temperature pure zirconia is in monoclinic form (*m*), as the temperature material elevates above 1170 °C, the monoclinic form to 2370 °C changes to tetragonal form (*t*) then above 2370 °C till melting point 2680 °C, the cubic form (*c*) will exist. Upon cooling, any of these forms Tetragonal (*t*) or Cubic (*c*) form will return to monoclinic form. With addition of stabilizing oxides so called stabilizers such as Ceria (CeO₂), Magnesia (MgO), or Yttria (Y₂O₃) to zirconia, the tetragonal form (*t*) become stable at room temperature and this phase transformation could be prevented [6-9]. This spontaneous transformation of tetragonal (*t*) form to more stable monoclinic (*m*) form will be accompanied with a noticeable volume increase of the crystals (4-5%) creating favorable compressive stresses in the material. This mechanism has been defined "Phase Transformation toughening" (PTT). This localized compressive stresses around and at the tip of crack will counteract crack propagation. Tetragonal (*t*) form can also transform to monoclinic (*m*) form as a result of external applied forces either mechanical by (grinding or sandblasting) or thermal aging [10-12]. Among all other types of ceramics, Zirconia has the highest mechanical properties. It has flexural strength of (900-1200 MPa) [13] which is approximately equal to metal-ceramics (900-1000 MPa), twice as strong as alumina oxide ceramics (500 MPa) and 5 times greater than standard glass-ceramics

[14,15]. The low temperature degradation (LTD) or hydrothermal degradation or aging of zirconia is a well-known process strictly related to the **phase transformation toughening (PTT)** and represents the other side of a same coin. It results in a spontaneous, slow transformation of the crystals from the tetragonal (*t*) phase to the stable monoclinic (*m*) phase in absence of any mechanical stresses [7,16]. It initiates in isolated surface grains between which water is incorporated into the zirconia lattice through dissolution of Zr-O-Zr bonds and filling of oxygen vacancies [17]. After the surface is saturated with monoclinic (*m*) phase, LTD proceeds into bulk material [18]. This process may reduce the strength, toughness, and density of zirconia significantly [19]. By surface roughening, grain pull out and micro-cracking [20,21]. These micro-cracks offer a pathway for water to diffuse further into the bulk of the ceramic material. In 2001, the United State Food and Drug Administration (FDA) announced that as series of zirconia femoral heads had fractured due to LTD [22]. These premature failure of zirconia in hip implants have made considerable attention and concern about the lifetime of zirconia based restorations. Aging was first reported by Kobayashi et al. [23] in 1981 as a degradation that occur relatively low temperature 150-450 °C in a gaseous environment. In 1986, Lange et al. [24] linked this low temperature aging to a reaction between water vapor and the yttrium in Y-TZP. The authors reported that water vapor drew yttrium out to the tetragonal grain surface where monoclinic (*m*) nuclei were created. The nuclei grew by further depletion of yttrium until the tetragonal (*t*) grains transformed completely [25]. Although this transformation mechanism is very slow at oral temperature, other contributing factors, such as constant humidity resulting from saliva, temperature and PH changes due to various drinks and repeated high occlusal loads due to mastication, can accelerate the aging process and reduce mechanical properties of the materials [26,27]. Aging environments used

in the studies of low temperature degradation in zirconia ceramics vary from the relatively mild aging in humid air (for periods in excess of 3 years) [28] to the much more aggressive autoclave conditions [29,30].

All the studies of LTD reported a range of temperature between 125 °C and 400 °C. Sato et al. [31] reported that at 200-300 °C in dry air, while Sato et al. [32] and Lange et al. [24] reported that in humid atmospheres the highest rate is found between 125 °C and 225 °C. Degradation rates at room or body temperature of Y-TZP ceramics are currently not available, and accelerated tests at intermediate temperature (100-300 °C) are the only basis for extrapolating an estimate of the transformation and, hence, of product lifetime. This approach relies on the assumption that the transformation rate follows the same Arrhenius-like trend down to room/body temperature [12,24,31,32]. Several approaches are used to simulate aging in stabilized. The first one is autoclaving for 5 hours under 2 bar pressure and at temperature 134 °C [33,34]. The second one is prolonged boiling which can be accomplished by placing samples in boiling water and recycling the condensate to decrease evaporation [35]. Additional method used to age stabilized zirconia have included storage at 37 °C in Ringer's solution, saline solution, or distilled water. Since monolithic Y-TZP FPDs exposes the material directly to oral environment, consisting more in more hazardous condition (association between mechanical stimuli, water, and temperatures) [36-38]. Several studies have shown that the strength of zirconia test specimens has not been significantly affect by aging procedure according to the ISO standard 13356 (steam autoclave, 5 hours at 0.2 MPa and 134 °C) [39], Although significant amounts of monoclinic (*m*) phase were present [40-42]. Tanaka et al. [42] showed that after 190 hours in an autoclave at 121 °C and 0.15 MPa, Y-TZP specimens had flexural strength of 1054 ± 91 MPa even through a greater than 90%

monoclinic (*m*) phase was detected by X-Ray Diffraction (XRD). Another study by Ban et al. [43] evaluated 0.5 mm thick specimens in various environments including autoclave (121°C and 0.15 MPa), physiological saline, and 4% acetic acid (80 days at 80 °C). Only specimens subjected to autoclave aging from 1046 MPa to 892 MPa, with measured increase in the monoclinic (*m*) phase from 0.3% to 49.9%. Alghazzawi et al. [44] evaluated zirconia specimens subjected to accelerated aging regarding relative changes in mechanical properties and structural stability. Accelerated aging was done boiling of specimens at 100 °C for 7 days in artificial saliva. It was found that the flexural strength was not significantly altered after LTD, although there was significant tetragonal (*t*) to monoclinic (*m*) transformation (from 2% to 21%) after accelerated aging in artificial saliva. The effect of accelerated aging (hydrothermal degradation) of Y-TZP was studied by Flinn et al. [20]. The experimental Y-TZP zirconia specimens were artificially aged under standard autoclave sterilization conditions, 134 °C at 0.2 MPa (at 5, 50, 100, 150, and 200 hours), and under standard industrial ceramic aging conditions, 180 °C at 1.0 MPa (at 8, 16, 24, and 48 hours). The tetragonal (*t*) to monoclinic (*m*) transformation was measured by using x-ray diffraction for all groups. Hydrothermal aging of zirconia caused a statistically significant decrease in flexural strength of thin bars of zirconia, which was the result of the transformation from tetragonal to monoclinic crystal structure from long-term degradation [20]. Siarampi et al. [45] studied the effect of in vitro aging (autoclaving at 121 °C and 2 bars) on the flexural strength of different Y-TZP specimens. It was found that slight increase of flexural strength of Y-TZP specimens after 5 hours and 10 hours of aging. Both Y-TZP specimens presented a tetragonal (*t*) to monoclinic (*m*) phase transformation with the monoclinic (*m*) increasing from 4 - 5% at 5 hours to around 15 % after 10 hours. The influence of low temperature degradation and cyclic loading

on the fracture resistance of monolithic Y-TZP zirconia crowns were studied by Nakamura et al. [46]. They used bar-shaped zirconia to evaluate surface fraction and penetration depth of monoclinic (*m*) phase using X-ray diffraction (XRD) and scanning electron microscopy (SEM) after being autoclaved at 134 °C for (0 – 200) hours to induce LTD. Monolithic Y-TZP molar crowns were used for the fracture testing. They found that monoclinic (*m*) phase fraction on the surface increased with autoclaving time and reached a plateau after 50 hours. The depth of the monoclinic (*m*) phase increased without reaching a plateau. The fracture load of the crowns significantly decreased from 5683 N (SD: 342) to 3975 N (SD: 194) after 100 hours of autoclaving. They concluded the even through LTD increased the monoclinic (*m*) phase, resulting in lower strength. The fracture resistance of the monolithic zirconia crowns was still sufficient to withstand the loading conditions in the molar regions [46].

METHODOLOGY

A total of 14 3-unit FPDs manufactured from monolithic TZI (Sirona dental systems, GmbH, D-64625, Bensheim, Germany) using Cerec inLab system (Sirona Dental Systems GmbH, D-64625 Bensheim, Germany) which were divided into two groups of 7 specimens each. The first aged TZI group (**Group A**) was subjected to accelerated artificial aging procedures in an autoclave at hydrothermal conditions 134 °C /2 bars for 5 hours. The second non-aged TZI group (**Group B**) was not subjected to any aging procedures and was designed as control group. Two stainless steel dies were designed to receive 3-unit FPDs. They were machine milled to the dimensions of 5 mm height (with a uniform 120 degrees circumferential chamfer finish line of 1 mm width, and a total occlusal convergence angle of 12 degrees). They were placed in pairs in a stainless steel holder, **Figure (1)**. All bridges sintered in Sirona inFire HTC furnace (Sirona Dental Systems GmbH, D-64625 Bensheim,

Germany) as recommended by manufacturer and checked for passive fit on their corresponding dies, **Figure (2)**. All specimens of **Group A** were subjected to artificial aging procedures by placement in stainless steel tray of Class B standard autoclave (Tinhoero 12 European, Foshan Core Deep Medical Apparatus Co., Ltd, China) at 134 °C/2 bar for 5 hours. While **Group B** was not subject to any aging procedure after sintering and considered as control group. Then all specimens were loaded compressively in a universal testing machine (Instron, model 3345, England) at cross head speed 0.5 mm/min until fracture occurred. A tin foil was placed between the load piston and the specimen to ensure even stress distribution and minimization of the transmission of local force peaks, **Figure (3)**. After fracture testing procedures, the amount of (*m*) phase detected in a representative part (fracture line) taken from specimen of aged TZI group (**Group A**) and Non-aged TZI group (**Group B**) was detected by XRD device software. Also, The fracture site of the tested specimens Aged TZI (**Group A**) and Non-aged TZI (**Group B**) subjected to fracture loading was examined by using scanning electron microscope (SEM)(JEOL-JSM-5500 LV).

RESULTS

Student's t-test was used to compare between fracture resistance of Zirconia and aged Zirconia. The significance level was set at $P \leq 0.05$. The Non-aged TZI (**Group B**) showed statistically significantly higher mean fracture resistance than Aged TZI (**Group A**). The fracture resistance mean (SD) values of non-aged TZI (**Group B**) was 2406.9 ± 306.8 N which showed statistically significantly higher than that of aged group (**Group A**) which was 1964.5 ± 234.5 N, **Figure (4,5) Table (1)**. The percentage of monoclinic (*m*) phase detected by XRD device software. It was nearly 0 weight % in Non-aged TZI (**Group B**) and about 42 weight % in Aged TZI (**Group A**).

Table I - Descriptive statistics and results of comparison between fracture resistance values (N) of the two groups

Group	Mean	SD	Median	Minimum	Maximum	95% CI		P-value
						Lower bound	Upper bound	
Non-aged TZI (Group B)	2406.9	306.8	2307.0	1993.0	2856.3	2123.1	2690.7	0.010*
Aged TZI (Group A)	1964.5	234.5	1971.8	1525.2	2205.8	1747.7	2181.3	

*: Significant at $P \leq 0.05$



Figure 1 - The machined stainless steel dies placed in stainless steel holder.



Figure 2 - Monolithic TZI 3-unit FPD seated on the stainless dies.



Figure 3 - Tin foil placed between the load piston and Monolithic 3-unit FPD.

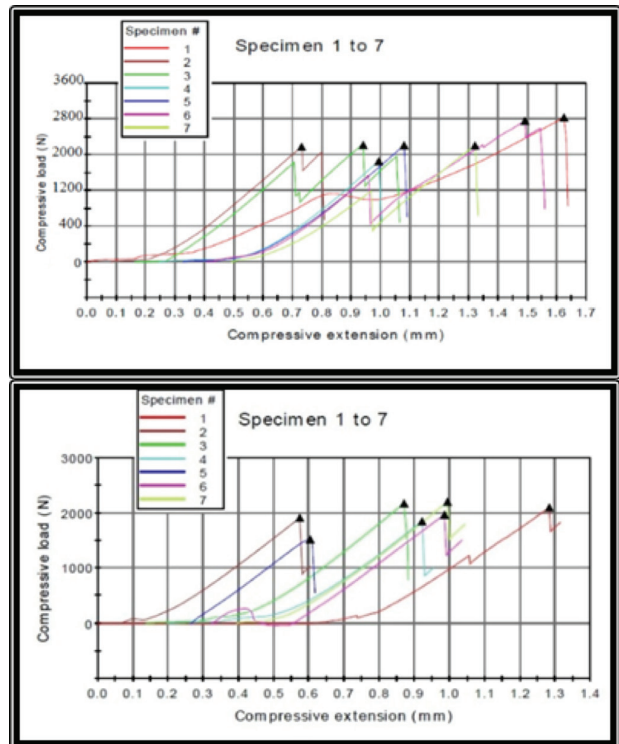


Figure 4 - Load-deflection curves of: **Left**, Non-aged TZI (Group B), **Right**, Aged TZI (Group A).

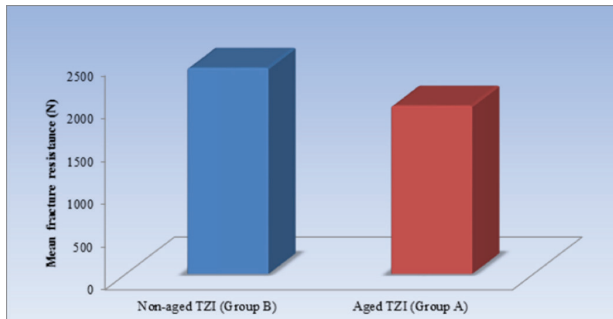


Figure 5 - Bar chart representing mean fracture resistance in the two groups.

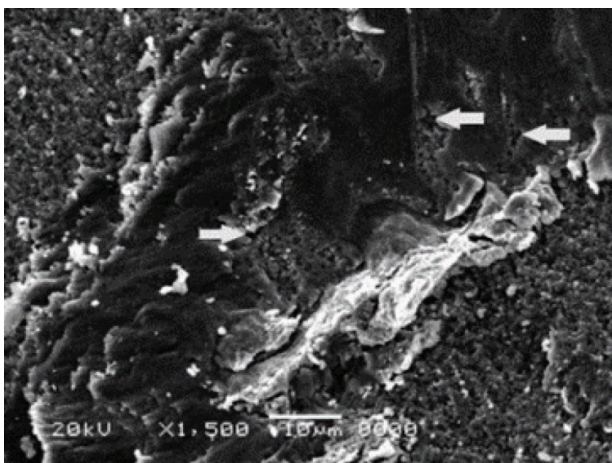
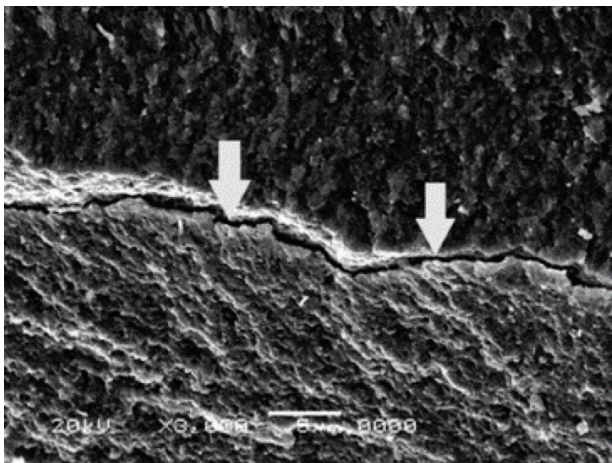


Figure 6 - SEM images **Left**, Non-aged TZI (**Group B**) showing white arrows pointed to crack line. **Right**, Aged TZI (**Group A**) showing white arrows pointed to multiple voids.

DISCUSSION

The monolithic translucent zirconia-based restorations become very attractive dental solution to avoid the porcelain chipping problem commonly encountered in ceramic restoration of bilayer structure [5]. This will put it in direct contact with opposing natural dentition and saliva at oral temperature [47]. The low temperature degradation (LTD) or hydrothermal degradation or aging of zirconia is a well-known process strictly related to the phase transformation toughening (PTT). It results in a spontaneous, slow transformation of the crystals from the tetragonal (*t*) phase to the stable monoclinic (*m*) phase in absence of any mechanical stresses. It means instability at low temperature in presence of water. The transformation starts from surface into the bulk of the material and is associated with micro-cracking and decline of the surface or bulk mechanical properties [7,24,29,48]. The development of high strength oxide ceramics such as zirconium oxide coupled with CAD/CAM technology extended the use of the monolithic restorations in oral cavity without veneering material. This put the restoration in direct contact with opposing natural dentition and saliva at oral temperature with subsequent exposure to occlusal loads and thermal fluctuation from fluids and foods [33]. This study was conducted to evaluate the fracture resistance of monolithic TZI restorations (3-unit FPDs) before and after accelerated aging tests in autoclave at 134 °C and 2 bar pressure for 5 hours which has theoretically the same effect as 3-4 years in vivo studies [29]. In the present study, the abutment teeth were made of stainless steel with elastic modulus 200 MPa, which is superior to that of dentin 12 MPa [49-51]. The stainless steel dies were machinery milled with uniform 120 degrees circumferential deep chamfer finish line of 1 mm width, and convergence angle 12 degree to simulate a second lower premolar and a second lower molar to receive 3-unit FPDs.

The use of a deep chamfer in this study found to be sufficient in theoretical studies [52]. The monolithic TZI FPDs were fabricated using CEREC inLab CAD/CAM system without any polishing. The pre-shaded monolithic TZI blocks were used in this study to avoid disadvantages of using of coloring solution [53,54]. The accelerated low temperature degradation (LTD) of zirconia based restorations relative to the oral environment used in the present study was the most widely accepted zirconia aging mechanism is cited in ISO standard [39] 13356: 2008, which states that autoclave aging under 2 bar pressure for 5 hours. Also, this mechanism of accelerated aging was recommended by several studies [12,35,39]. In the present study, the mean value and the standard deviations of the fracture resistance of monolithic TZI FPDs (3 units) for (**Group B**) non-aged TZI which was not subjected to any aging process was 2406.9 N (SD \pm 306.8) while for (**Group A**) aged TZI which was 1964.4 N (SD \pm 243). Non-aged TZI (**Group B**) showed statistically significantly higher mean value of fracture resistance than aged zirconia ($p \leq 0.05$).

The hypothesis of this study stated that subjecting zirconia based restorations to aging process may affect its final fracture resistance was accepted. Still fracture resistance of aged TZI (**Group A**) higher than the biting force found in the molar region which vary from 342-1280 N [55]. In this study, the percentage of monoclinic (m) phase detected by XRD device in non-aged TZI (**Group B**) was nearly 0 weight % and in aged TZI (**Group A**) was about 42 weight %. This high monoclinic (m) percentage can be explained by absence of any polishing procedure performed in this study. The results of the present study were in accordance with Ban et al. [43] who evaluated 0.5 mm thick specimens in various environments including autoclave (121°C and 0.15 MPa), physiological saline, and 4% acetic acid (80 days at 80 °C). Only specimens subjected to autoclave aging showed significant decrease

from 1046 MPa to 892 MPa, with measured increase in the monoclinic (m) phase from 0.3% to 49.9%. Also, the results of this study were in accordance with study by Flinn et al. [20] in which Y-TZP zirconia bars were exposed to standard autoclave sterilization conditions (134 °C at 0.2 MPa) and under standard industrial ceramic aging conditions (180 °C at 0.1 MPa). It was found that the flexural strength of zirconia specimens was significantly decreased after hydrothermal aging and this was attributed to the transformation from Tetragonal (t) to monoclinic (m) crystal structure [20]. On the opposite side, the results of this study were against results found by Amaral et al. [38] who found that zirconia specimens aged at 127 ± 1 °C and 150 KPa air pressure for 12 hours had load strength significantly increased in value after LTD. Another study by siarampi et al. [45] had results against this study who studied the effect of in vitro aging (autoclaving at 121 °C and 2 bar pressure) on the flexural strength of the Y-TZP ceramics after 5 hours and 10 hours. It was found a slight increased flexural strength values after 5 hours but decreased values after 10 hours of aging. Although the (m) phase was increased to 4-5% after aging for 5 hours and to around 15% after aging for 10 hours. Another study by Kim et al. [19] also investigated the degradation of flexural strength of Y-TZP ceramic after various low temperature treatments and reported that the point of monoclinic (m) phase concentration at which the flexural strength begins to decrease is between 12 weight % and 54 weight %. The decrease in fracture resistance found by this study can be explained by the presence of flaws, microcracks and pores that may have been created during manufacturing and processing located inside the bulk of the zirconia based ceramic material. These flaws can act as stress concentration sites potential for crack initiation and propagation [19,56], and enhance the LTD process facilitating water penetration [57]. Also, if (t) to (m) phase transformation is confined superficially, it may cause initial increase in

the strength of the material. If, however, it propagates for inside the bulk of the material, the failure is unavoidable especially under occlusal loads in the oral environment [45].

CONCLUSIONS

Accelerated artificial aging decreases the fracture resistance of monolithic TZI FPDs.

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