

Original research article

# Effect of thermal and mechanical aging on fracture toughness of Y-TZP core materials

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## ABSTRACT

**OBJECTIVE:** The purpose of this study was to evaluate the effect of thermal and mechanical aging on the fracture toughness of yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) core materials.

**MATERIALS AND METHOD:** Disc-shaped specimens for each core material (diameter: 15 mm, thickness: 1.2 mm), Cercon Base and Lava Plus, were randomly divided into three groups (control, thermal aging and mechanical aging). No aging was applied to control specimens. Thermal aging group specimens were subjected to  $10^4$  thermal cycles in water between 5-55 °C (dwell time of 30 s). Mechanical aging group specimens were subjected to a 200 N load for  $10^5$  times, with a frequency of 2 Hz. The fracture toughness of the control, thermal and the mechanical aging groups were measured by using the indentation fracture technique. Ten indentations were performed for each material. After each indentation, microscopic readings were recorded and the fracture toughness was calculated according to an established formula. Results were statistically analyzed by using a two-way analysis of variance (ANOVA) test.

**RESULTS:** Indentation fracture toughness values of the control, the thermal and the mechanical aging groups of Cercon Base were 6.28, 6.39 and 6.02  $\text{MPa}\sqrt{\text{m}}$ , respectively. There was no significant difference between the control group of Cercon Base and the aging groups ( $p>0.05$ ). However, significant difference was found between the thermal and the mechanical aging groups of Cercon Base ( $p<0.05$ ). Indentation fracture toughness values of the control, the thermal and the mechanical aging groups of Lava Plus were 4.79, 4.76 and 4.91  $\text{MPa}\sqrt{\text{m}}$ , respectively. Again, there was no significant difference between the control the aging groups of Lava Plus ( $p>0.05$ ).

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**CONCLUSION:** Compared to control, thermal and mechanical aging had no significant effect on the fracture toughness of the different Y-TZP core materials.

**KEYWORDS:** Aging; ceramics; fatigue fractures; yttria stabilized tetragonal zirconia; zirconium oxide

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## INTRODUCTION

Yttria-stabilized tetragonal zirconia polycrystals (Y-TZP) are commonly used core materials manufactured from fine zirconia ( $\text{ZrO}_2$ ) particles and 1.75 to 3.5 mol % yttrium oxide ( $\text{Y}_2\text{O}_3$ ).<sup>1,2</sup> Although zirconia based restorations have high strength, fracture has been reported as one of the most frequent complication for these restorations.<sup>3</sup> Before clinical application of a new dental material can be considered, fracture toughness value as well as bending strength of the material, defining together the mechanical strength, must be known. The fracture toughness is an important mechanical property, especially for zirconia-based fixed-partial dentures.<sup>4</sup>

Fracture toughness is a mechanical property defined as the fracture resistance against a propagating crack and stress intensity level at which catastrophic failure occurs due to microdefect.<sup>5,6</sup> The lower fracture toughness defines lower clinical reliability of the ceramic restorations.<sup>5</sup> Several toughening mechanisms with different effectiveness have been used to improve the properties of dental ceramics.<sup>7</sup> Fracture toughness can be measured by single edge precracked beam (SEPB), indentation strength and indentation fracture methods.<sup>8</sup> The SEPB method is a modification of the single edge notched beam method and it solves the precracking problem wherein an indented or saw cut flexure specimen is compression-loaded in a bridge until a precrack generates.<sup>9-10</sup> The precracked beam is then fractured in three point flexure test and fracture toughness is evaluated.<sup>8,11</sup> In the indentation strength method, a Vickers indentation is used to generate artificial flaw on the sur-

face of flexure specimen and the specimen is fractured in three or four point flexure test.<sup>8,12</sup> In the indentation fracture method, specimens are indented with Vickers indenter and the length of the median cracks are measured. The fracture toughness value depends on the indentation load, the size of the median cracks, the elastic modulus and the hardness of the material.<sup>8</sup> The indentation fracture method is a simple, easy, and economic method and has been widely used for measuring fracture toughness of dental ceramics.<sup>4,5,13</sup> Furthermore, using of a small amount of specimen area is the main asset of this technique.<sup>5,13</sup>

Microcracks and defects which grow inherently during thermal and mechanical processes may cause time-dependent aging of ceramic materials which can have detrimental effects on the mechanical properties of the material.<sup>5,6,14-17</sup> Therefore, aging tests are very essential to evaluate mechanical performance and clinical success of Y-TZP core materials.<sup>15-17</sup>

The purpose of this study is to evaluate the effect of thermal and mechanical aging on fracture toughness of commonly used Y-TZP core materials by using the indentation fracture method.

## MATERIALS AND METHOD

### Specimen preparation

Two commercially used zirconia-based core materials (Cercon base; Cercon, DeguDent, Hanau, Germany and Lava All-Ceramic System; Lava Plus, 3M ESPE, Seefeld, Germany) were selected for this study. Cercon Base consisted of zirconia, yttrium oxide: 5%, hafnium oxide: <2%, and aluminium oxide and silicon oxide: <1%. Lava Plus consisted of zirconia, yttrium oxide: 3%, and aluminium oxide: 0.1 wt %.

For each material, disc-shaped specimens (diameter: 15 mm and thickness: 1.2 mm) were fabricated from partially stabilized-zirconia blanks and sintered by the manufacturers. The dimensions of the specimens were controlled by using a digital micrometer (Powertechtools, Zhejiang, China). Specimens from each material were divided into three groups randomly. Each group consisted of three specimens. Group 1 was the control group. Group 2 was the thermal aging group; the specimens were subjected to  $10^4$  cycles in water between 5-55 °C in a thermal cycling machine (Nuve, Bursa, Turkey) with a dwell time of 30 s. Group 3 consisted of mechanical aging group; the specimens were subjected to a 200 N load for  $10^5$  times in a mechanical cycling machine (Instron 8801, Instron, Canton, MA, USA) with a frequency of 2 Hz. The cycling was conducted at room conditions ( $22\pm 1$  °C and  $60\pm 5\%$  relative humidity). All measurements were performed on the compression side of the mechanically-cycled specimens.

### Fracture toughness determination

The fracture toughness of materials was determined by using the indentation fracture technique.<sup>13</sup> The disc-shaped specimens were embedded in acrylic resin cylinders (Panacryl; Arma Dental, Istanbul, Turkey). Surfaces of the specimens were polished with a ceramic surface polishing kit (Sof-Lex Finishing and Polishing System, 3M ESPE, Seefeld, Germany). Each polishing wheel was used approximately for 20 s at a speed of 350 rpm. After surface grinding and polishing, all specimens were ultrasonically cleaned in distilled water for 10 min (Euronda, Eurosonic Energy, Vicenza, Italy).

Experiments were conducted according the indentation fracture technique by pressing a pyramid Vickers indenter onto the test specimen to generate cracks around the indentation. Standard Vickers loads of the testing machine (Vickers; Fritz Heckert, Leipzig, Germany) were evaluated to determine the optimum load which generate acceptable crack pattern on each material. Optimal load for each material was determined by comparing the crack length from center of the indent to length of half diagonal. The acceptable cracks were evaluated according to the following criteria:<sup>13,18</sup> all cracks emanated from the corners of the Vickers indent, presence of radial cracks with crack length two-times longer than the half diagonal of the indentation and the absence of chipping.

The average crack length ( $c$ ) was obtained from the measurement of the radial cracks ( $2c$ ). A load of 30 kgF (294 N) was used to generate acceptable radial cracks on specimens of both groups. Each specimen was indented at ten locations with a load holding time of 15 s. After each indentation, readings were recorded immediately under the microscope of the indentation machine. The fracture toughness was calculated according to the following formula as described in a previous study by Anstis *et al.*<sup>13</sup>:

$K=0.016\times(E/H)^{1/2}\times P/c^{3/2}$  where,  $H=P/2a^2$ , in which  $K$  is the fracture toughness of the material ( $\text{MPa} \times \text{m}^{1/2}$ ),  $E$  is the elastic modulus,  $P$  is the load applied (N),  $a$  is the indent half diagonal (m) and  $c$  is the crack length measured from the center of the indent (m). Elastic modulus values were 215 GPa for Cercon and 210 GPa for Lava.<sup>1,14</sup>

### Statistical analysis

The fracture toughness results were statistically analyzed by using two-way analysis of variance (ANOVA) with a significance level set at 0.05 (SPSS 18, SPSS, Chicago, IL, USA).

## RESULTS

All Y-TZP specimens subjected to aging survived. Indentation fracture toughness of all groups are shown in

**Table 1.** Indentation fracture toughness values of the tested materials; mean (Standard error)

	Cercon Base	Lava Plus
Control	6.28(0.13) AB*	4.79(0.04) A
Thermal aging	6.39(0.12) A	4.76(0.06) A
Mechanical aging	6.02(0.17) B	4.91(0.04) A

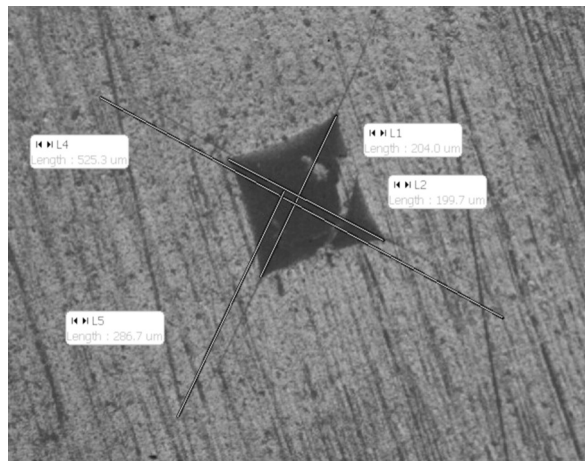
\*For each column, no significant difference exists between groups marked with the identical capital letter ( $p>0.05$ ). Note that statistically significant difference exists between Cercon Base and Lava Plus specimens at each control, thermal aging and mechanical aging groups (horizontal comparisons;  $p<0.05$ ).

Table 1. Indentation fracture toughness values of the control, thermal and mechanical aging groups of Cercon Base were 6.28, 6.39 and 6.02  $\text{MPa}\sqrt{\text{m}}$ , respectively. There was no significant difference between the control group of Cercon Base and the aging groups ( $p>0.05$ ). However, there was a significant difference between the Cercon Base thermal aging and mechani-

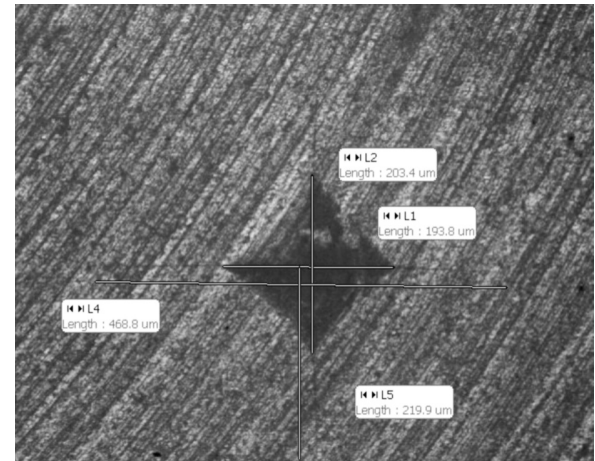
cal aging groups ( $p<0.05$ ). Indentation fracture toughness values of the control, thermal and mechanical aging groups of Lava Plus were 4.79, 4.76 and 4.91  $\text{MPa}\sqrt{\text{m}}$ , respectively. There was no significant difference between the groups of Lava Plus ( $p>0.05$ ). When the groups of Cercon Base and Lava Plus were compared, fracture toughness values were found to be significantly different ( $p<0.05$ ). Images of the indentation fracture tests for one specimen from the control, thermal and the mechanical aging groups are shown in Figures 1-3 for Cercon Base and Figures 4-6 for Lava Plus, respectively.

**DISCUSSION**

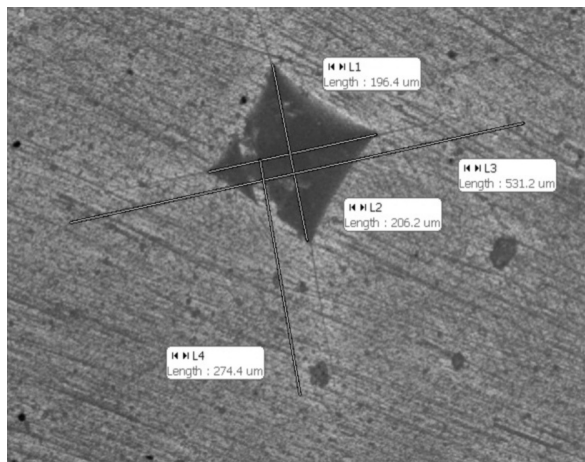
Zirconia restorations are exposed to moisture, mechanical and thermal fatigue known to induce deformations within the materials and their interfaces during the mas-



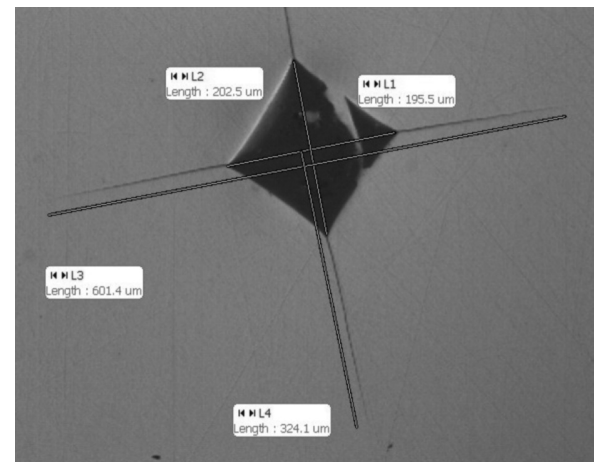
**Figure 1.** Vickers indentations and cracks on one specimen from the Cercon Base control group



**Figure 2.** Vickers indentations and cracks on one specimen from the Cercon Base-thermal aging group

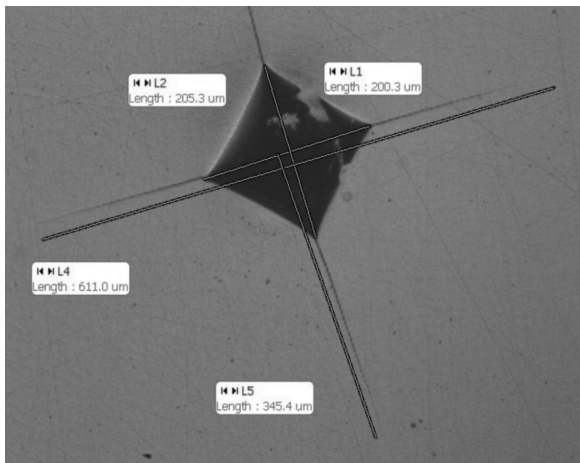


**Figure 3.** Vickers indentations and cracks on one specimen from the Cercon Base-mechanical aging group



**Figure 4.** Vickers indentations and cracks on one specimen from Lava Plus control group



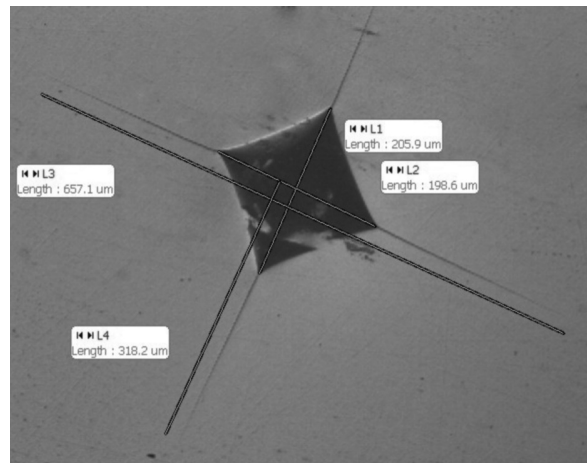


**Figure 5.** Vickers indentations and cracks on one specimen from the Lava Plus-thermal aging group

tatory function in oral cavity.<sup>15,19-21</sup> Beside the thermal and mechanical fatigue, factors such as the grain size and shape, the type of stabilizing elements, the size and distribution of internal flaws, and presence of prestresses may affect the clinical failure of ceramic based restorations.<sup>1,22</sup>

The oral cavity is a dynamic environment where changes in forces and temperatures occur. These factors affect mechanical properties of restorations.<sup>23</sup> Fontijn-Tekamp *et al.*<sup>24</sup> reported that physiological biting and chewing forces range from 60 to 75 N in the anterior dentition and from 110 to 125 N in the posterior dentition. The cycling load (200 N) applied in this study might be used for the estimation of restorations' performance under severe conditions like bruxism.<sup>14</sup> During loading, the material is subjected to highly localized stresses. The stresses not only cause crack formation but also lead to change of the crystal structure and transformation to amorphous phase.<sup>25</sup> This transformation also occurs in the presence of cyclic hydrothermal stresses such as water or water-based conditions.<sup>26</sup> These stresses are often stimulated by thermal cycling with temperature changes.<sup>27</sup> The temperature varies due to the intake of hot and cold food in the oral cavity.<sup>16,20</sup> The reported lowest comfortable temperature is 15 °C and the highest ranges 50-55 °C.<sup>28,29</sup> Beside the temperature ranges, the cycle number is also important to evaluate the effect of thermal aging.<sup>27</sup> Gale & Darwell<sup>27</sup> reported that 10<sup>4</sup> thermal cycles represents one year clinical function in the oral cavity. In this study, thermal aging was subjected for 10<sup>4</sup> times in water with bath helds 5-55 °C.

Studies have focused on the effect of fatigue on strength,<sup>16,17,30</sup> however there is limited data on the influence of aging on fracture toughness, which indicate success of the material in the oral cavity.<sup>14</sup> The clinical success of all ceramic restorations is very often associated with their brittleness and low fracture tough-



**Figure 6.** Vickers indentations and cracks on one specimen from the Lava Plus-mechanical aging group

ness.<sup>7</sup> The improvement of reliability of a material in service can be achieved by increasing its fracture toughness.<sup>7,31</sup>

There are some methods to evaluate the fracture toughness of zirconia based materials.<sup>8</sup> Single edge precracked beam (SEPB) method is recommended by ISO for testing fracture toughness of dental ceramics.<sup>32</sup> However, it is difficult to generate experimental cracks and obtain Young modulus of the specimens by performing different experiments with single-edge precracked beam method.<sup>4</sup> When a Vickers indenter is pressed on the surface of a specimen, crack is generated around the indentation. The indentation fracture method is a test method to evaluate fracture toughness of ceramic-based materials by measuring the size of these cracks. The indentation fracture method is easier to apply than the SEPB method.<sup>4,8</sup> The main asset of this technique is the small amount of specimen area needed. Therefore, typically hundred indentations can be made on a single surface edge dimension of 10 mm.<sup>13</sup> In this study, this technique was used for testing the fracture toughness of the materials and ten indentations were made on each specimen to evaluate the fracture toughness values.

The fracture toughness was determined using the indentation fracture method in several studies on dental ceramics.<sup>7,14,33-35</sup> In the present study, acceptable precracks were generated from 30 kgF (294 N) load with 15 s of load holding time. Harada *et al.*<sup>4</sup>, also stated that both 196 N and 294 N loads were optimum experimental conditions of the indentation fracture test for zirconia-based ceramics. And also, it was stated that in the SEM observation of the indentation and the cracks, no chipping and fracture were observed. Similar to these results, chipping or fracture were not observed under 30 kgF load in this study. Okada *et al.*<sup>36</sup> stated that 5 s holding time was not enough to generate adequate cracks and there was not any difference between 15 s and 30

s holding times. Furthermore, 15 s holding time was used in another study.<sup>4</sup> In this study, the 15 s of load holding time was selected.

The indentation fracture toughness of Cercon Base was slightly increased in the thermal aging group and the values were lower in the mechanical aging group. However, there was no significant difference between the Cercon Base control and the aging groups ( $p>0.05$ ). There was significant difference between the Cercon Base thermal and mechanical aging groups ( $p<0.05$ ). Karakoca Nemli *et al.*<sup>14</sup> evaluated the effect of mechanical fatigue with  $20^4$  cycles on fracture toughness and phase transformation of Y-TZP ceramics (Lava Frame and Cercon) and found no significant difference between control and fatigue groups of Cercon. In the present study, fracture toughness of Cercon Base was lower in the mechanical aging group compared to the control. The difference between the values of Cercon Base after mechanical aging, in comparison to control, may have resulted from higher mechanical cycles used in this study. Yılmaz *et al.*<sup>5</sup> presented similar fracture toughness values for Cercon to Cercon Base control group used in this study.

It was observed that mechanical or thermal aging did not significantly affect the fracture toughness of Lava Plus compared with the control group ( $p>0.05$ ). Karakoca Nemli *et al.*<sup>14</sup> found that fracture toughness of mechanical-aged Lava was significantly greater than control Lava. In the present study, the fracture toughness of Lava Plus showed an increase; but the difference was not significant ( $p>0.05$ ).

Two common approaches have been used to enhance the fracture toughness and mechanical properties of zirconia-based core materials. One of them is reducing the grain size of components, and the second is addition of secondary elements known as doping phase.<sup>37-39</sup> This secondary phase causes changes in the microstructure of the material and alters its mechanical and physical properties. Furthermore, alumina is one of the common dopant materials for increasing both the strength and the fracture toughness of zirconia-based restorations.<sup>40</sup> This may be the reason for observing different indentation fracture toughness for Lava Frame specimens and Lava Plus specimens with decreased alumina content. The fracture toughnesses of Cercon Base and Lava Plus specimens were significantly different in all groups ( $p<0.05$ ). The difference in fracture toughness between Cercon Base and Lava Plus specimens may be caused by the differences in the phase transformation and the microstructure of the materials.<sup>14</sup>

## CONCLUSION

This study presented the results of fracture toughness experiments for Cercon Base and Lava Plus Y-TZP core

materials before and after thermal and mechanical aging. Within the limitations of this study, following conclusions can be drawn: compared to the control, thermal or mechanical aging did not affect the fracture toughness of Cercon Base significantly. However, there was significant difference between Cercon Base thermal and mechanical aging groups. Compared to the control, thermal or mechanical aging, again, did not affect the fracture toughness of Lava Plus significantly. The fracture toughness values for Cercon Base and Lava Plus were significantly different.

**Conflict of interest disclosure:** The authors declare no conflict of interest related to this study.

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## Isısal ve mekanik yaşlandırma işlemlerinin Y-TZP kor materyallerinin kırılma tokluğuna etkisi

### ÖZET

**AMAÇ:** Bu çalışmanın amacı, ısısal ve mekanik yaşlandırma işlemlerinin, yttria-stabilize dörtgen zirkon (Y-TZP) kor materyallerinin kırılma tokluğu üzerindeki etkisinin araştırılmasıdır.

**GEREÇ VE YÖNTEM:** Her iki kor materyalinden disk şeklinde üretilen Cercon Base and Lava Plus örnekler (15 mm çap 1.2 mm kalınlık) rastgele olarak 3 gruba (kontrol, ısısal yaşlandırma ve mekanik yaşlandırma) ayrıldı. Kontrol gruplarına herhangi bir işlem yapılmadı. Isısal yaşlandırma işlemi 5-55 °C sıcaklıklarda su içeren banyolarda, her birinde 30 sn kalacak şekilde 10<sup>4</sup> kez uygulandı. Mekanik yaşlandırma işlemi ise, 200 N yük ile, 2 Hz frekansta 10<sup>5</sup> kez uygulandı. Kırılma tokluğu deneyi indentasyon kırılma metodu kullanılarak gerçekleştirildi. Her grup için ölçüm yapıldı. Her bir indentasyon sonrası mikroskopik okuma kaydedildi ve kırılma tokluğu değeri önceden belirlenmiş bir formüle göre hesaplandı. İstatistiksel analiz iki yönlü varyans analizi (ANOVA) ile yapıldı.

**BULGULAR:** Cercon Base grubu örneklerin kırılma tokluğu değerleri, kontrol, ısısal ve mekanik yaşlandırma grubu için sırasıyla 6.28, 6.39 ve 6.02 MPa√m olarak belirlendi. Cercon Base grubu örneklerinde, kontrol grubu ve yaşlandırma grupları arasında istatistiksel olarak anlamlı bir fark bulunmadı (p>0.05). Fakat, Cercon Base grubunda, ısısal ve mekanik yaşlandırma grupları arasında istatistiksel olarak anlamlı fark bulundu (p<0.05). Lava Plus grubu örneklerinin kırılma tokluğu değerleri, kontrol, ısısal ve mekanik yaşlandırma grubu için sırasıyla 4.79, 4.76 ve 4.91 MPa√m olarak belirlendi. Lava Plus grubu örneklerinde, kontrol grubu ve yaşlandırma grupları arasında istatistiksel olarak anlamlı bir fark bulunmadı (p>0.05).

**SONUÇ:** Yaşlandırma işlemlerinin, kontrole kıyasla, Y-TZP kor materyallerine ait kırılma tokluğu değerlerini belirgin olarak etkilemediği tespit edildi.

**ANAHTAR KELİMELER:** Seramikler; yaşlanma; yorulma kırıkları; yttria stabilize dörtgen zirkon; zirkonyum oksit