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Nanomechanical and microstructural characterization of a zirconia-toughened alumina composite after aging☆

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ABSTRACT

This study's objective was to mechanically characterize and validate the synthesis method of a polycrystalline composite comprised of 80% alumina reinforced with 20% translucent zirconia (zirconia-toughened alumina, ZTA) and compare to an experimental translucent zirconia.

Experimental ZTA (ZTA ZPEX 80/20) and translucent Y-TZP (ZPEX) green-state disc-shaped specimens were obtained via uniaxial/isostatic ceramic powder pressing technique. The discs were sintered using a predefined protocol after both sides of the discs were polished. The specimens were subjected to nanoindentation testing to acquire their elastic modulus (E) and hardness (H) before and after a simulated low temperature degradation (LTD) challenge. Subsequently, the fabricated discs had their 3D surface topographical (Sa/Sq) parameters assessed via interferometry before and after exposure to a simulated LTD aging protocol.

The specimens were evaluated using X-ray diffraction (XRD) to assess the tetragonal-monoclinic phase transformation and via scanning electron microscopy (SEM) to evaluate the homogeneity of the surfaces and distribution of the grains.

The apparent density was measured using Archimedes' principle. All of the data were statistically evaluated through repeated measures ANOVA following post-hoc comparisons using the Tukey test ($p < 0.05$).

The XRD patterns indicated a higher increase in the monoclinic peak for ZPEX compared to ZTA ZPEX 80/20 aged.

LTD aging did not have an effect on the surface roughness (Sa/Sq) for both groups ($p > 0.05$). A significant decrease in the E values after the aging protocol was observed for both groups ($p < 0.01$). While ZTA ZPEX 80/20 did not show statistically significant differences in the hardness values after the aging protocol ($p = 0.36$), ZPEX demonstrated a significant decrease in the H values ($p = 0.03$).

For ZTA ZPEX 80/20, simulated LTD aging did not affect the tested properties, except for the E values. Although artificial aging did not affect the surface roughness of ZPEX, the E and H values significantly decreased after aging.

1. Introduction

The use of a polycrystalline material such as yttrium tetragonal zirconia polycrystal (Y-TZP) was introduced as a potential alternative for esthetic dental treatment due to its mechanical properties as it presents superior fracture toughness compared to conventional ceramics used in dentistry [1]. Zirconia is considered a unique material because it occurs naturally in three allotropic crystalline structures: monoclinic (m), tetragonal (t), and cubic (c). The monoclinic phase is stable at room temperature up to 1,170 °C, at which it undergoes

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transformation to the tetragonal phase, which is stable up to 2,370 °C, at which point transforms to the cubic phase (Tm = 2,710 °C) [1–3]. The phase transformation from tetragonal to monoclinic (t-m) during the postsintering cooling of zirconia represents a critical point in the fabrication of the material, as the phase change leads to an increase in the volume that has the potential to induce internal stresses on the material, resulting in the formation of cracks and microcracks [1,4,5]. During the transformation from the tetragonal to the monoclinic phase (t-m), a volumetric increase of approximately 4.5% occurs and depending on its extent, may lead to strength degradation and failure [6–8].

To stabilize zirconia-based polycrystalline ceramics in the tetragonal phase at room temperature, oxides such as yttrium oxide (Y-TZP), cerium (Ce) oxide, calcium oxide, or magnesium oxide are added limiting t-m phase transformation during cooling with the advantage of preserving the toughness of tetragonal zirconia [9–11]. First introduced in the orthopedics field in the 1990s for femoral heads, critical events occurring in 2001 led to 400 Y-TZP femoral head fractures. Due to evidence of progressive aging degradation even under normal conditions, the long-term stability of zirconia remains problematic [1,12]. Although Y-TZP is the most common metastable material utilized for dental applications, it is susceptible to t-m phase transformation due to stress and low temperature degradation (LTD). Therefore, further development of zirconia's engineering and synthesis is warranted for the dental field, which utilizes the ceramic in several components (for example, in abutments, crowns, fixed dental prostheses, and implants) [13].

In dentistry, the porcelain veneering process and subsequent sintering has shown to trigger t-m phase transformation at the porcelain/Y-TZP interface due to the presence of moisture from the porcelain slurry [14]. Additionally, after aging, Y-TZP phase transformation has indicated that the presence of monoclinic phase content increased from 2.13% to 81.4%, suggesting an extreme variability in dental Y-TZP stability. This variability is dependent on several parameters such as the grain size, microstructure, manufacturing method, and processing methods of Y-TZP used in dentistry [13]. Therefore, new polycrystalline ceramic compositions are expected to better stabilize Y-TZP while maintaining some of its desired mechanical properties.

The combination of zirconia particles within an alumina matrix has been previously proposed as an alternative to improve the stability of Y-TZP at room temperature [15,16]. The presence of both the alumina and zirconia phases results in a composite material, commonly referred to as zirconia-toughened alumina (ZTA) or alumina reinforced by zirconia. ZTA is composed of a ceramic matrix along with a secondary phase of metastable tetragonal zirconia combining the advantageous properties of the two materials [17].

The ratio of zirconia to alumina within ZTA has been a focus of research for composites targeting orthopedic applications. The fracture toughness of ZTA after an autoclave aging process with 5%, 15%, and 30% of zirconia has been previously evaluated. For a composite with 5% Y-TZP, the R-curve behavior, determined by stressing the material, was found to be different from doped zirconia, likely due to its low content inside the alumina matrix. Conversely, ZTA composites with 15% and 30% Y-TZP presented a significant increase in resistance after the aging process, with a suitable R-curve behavior for their application in areas of high mechanical strength [18]. Considering that the transformation mechanism spreads from grain to contacting grain, it has been suggested that the maximum zirconia fraction to limit the spread of transformation is related to the interconnectedness of the zirconia phase, namely the percolation threshold [19]. Studies have suggested that this fraction can be up to 16% of zirconia (84% alumina) [20,21].

It is known that the metastability of Y-TZP for dental applications should be improved due the potential risk for long-term complications. This investigation aimed to propose a method for the synthesis of a ZTA composite (Al_2O_3 80%/Zr_2O 20%) potentially more stable than a translucent Y-TZP (ZPEX). This study also aimed to validate the synthesis method and characterize the experimental materials' topography and mechanical properties in the as-sintered condition and after a simulated LTD aging protocol.

2. Methods

2.1. Specimen fabrication

ZTA composites (Al_2O_3 80%/Zr_2O 20%) and translucent zirconia powder were blended in ethanol suspensions, then mixed and homogenized in a friction mill for 4 h with high purity alumina spheres. The slurry was dried in a rotary evaporator (801, Fisaton, SP, Brazil) and the obtained powder was manually granulated and sieved. The specimens were obtained by uniaxial pressing of the ZTA and ZPEX powders (Al_2O_3 Baikalox regular CR10, Baikowski, France, and Y-TZP ZPEX, Tosoh Corp., Japan) at 1,148 Kgf for 30 s in a tungsten carbide matrix 15 mm in diameter to obtain disc-shaped green body samples with 1.8 mm thickness. The green body samples were double-wrapped and sealed in vacuum sealer and subjected to isostatic pressing (National Forge, Pennsylvania, PA, SA) at room temperature.

The green body discs were sintered to 1,600 °C for 1 h in a Zyrcomat (Vita Zahnfabrink, Bad Säckingen, Germany) furnace (heating and cooling rate of 4 °C per minute). After sintering, both sides were polished with diamond discs using a semi-automatic polishing machine (Automet 2000, Buehler, Lake Bluff, IL, USA) with 220, 120, 90, 40, 25, 9, 6, and 1 μm granulated diamond disks (Allied High Tech Products, Rancho Dominguez, CA, USA) with diamond suspensions up to 1 μm (Fig. 1).

For each group, 13 disc-shaped specimens were fabricated with 12 mm diameter and 1 mm thickness (ISO 6872:2015) (Fig. 1). Three additional specimens per group were fabricated for phase quantification utilizing X-ray diffraction (XRD). The composition characteristics used in the preparation of the test specimens and the theoretical density values were calculated based on the density of alumina (3.986 g/cm^3) and zirconia (6.10 g/cm^3) (Tables 1 and 2). The density of the ceramics was measured utilizing Archimedes' principle [22].

2.2. Theoretical density

The theoretical density was obtained using the method based on Archimedes' principle to the ZTA ZPEX 80/20 and ZPEX groups to estimate the density of the materials after sintering.

The density of the specimens was measured using an analytical balance (Adventurer Analytical balance, Ohaus, Parsippany, NJ, USA) and a theoretical density kit accessory.

2.3. Simulated LTD aging

The specimens of each group were subjected to an aging protocol in an autoclave (Vitale Class CD 12L, PR, Brazil) for 20 h in water vapor at 134 °C and 2.2 bars to induce t-m transformation and LTD.

2.4. Interferometry

A 3D noncontact profilometer was used to analyze the 3D surface topographical parameters (Sa is the arithmetic mean deviation of a surface and Sq is the root mean square deviation of a surface) before and after simulated LTD aging using interferometry (IFM) (Phase View 2.5, Palaiseau, France) and a Zeiss Axio-Imager A.1 microscope (Zeiss, Oberkochen, Germany) (n = 4/group). The average values of the surface roughness (Sa and Sq) were calculated over an area of 150 × 150 μm.

2.5. Nanoindentation

Nanoindentation testing was performed at room temperature in

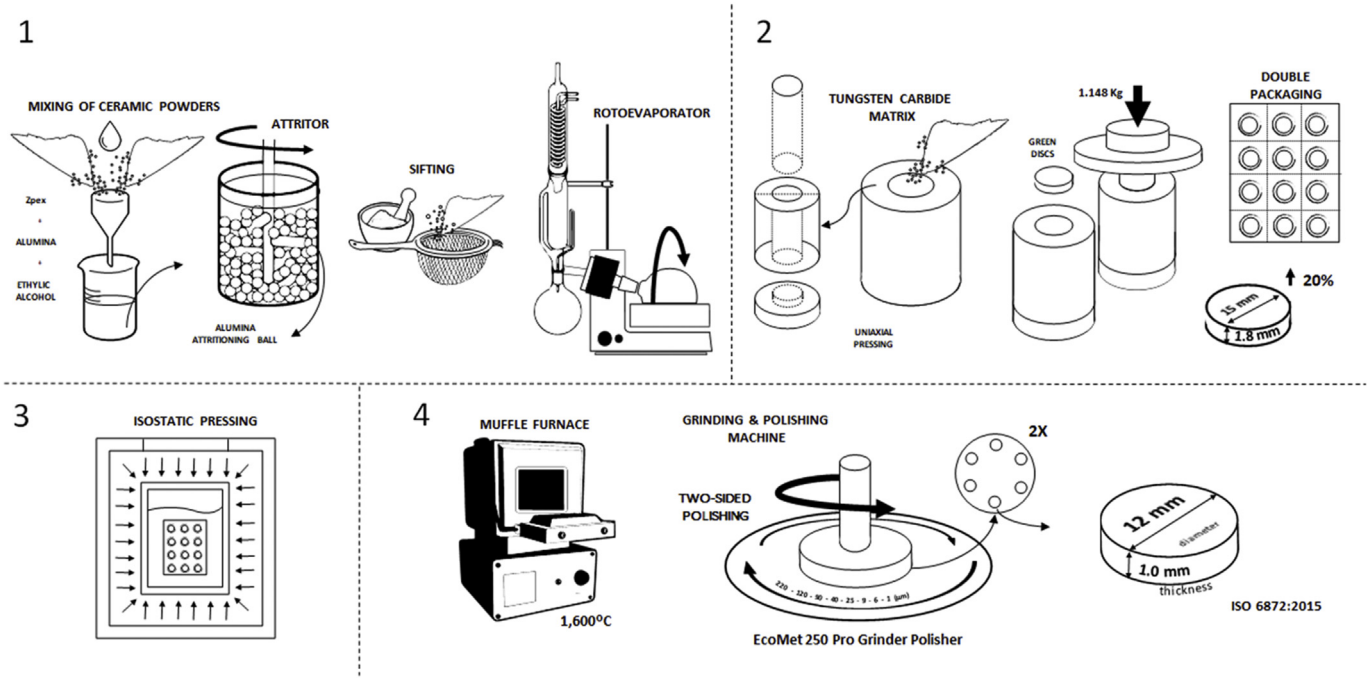

Fig. 1. Schematic depicting the steps of specimen fabrication. 1) Synthesis of the powder including mixing, attritioning, sifting, and rotoevaporator phases for powder homogenization. 2) Green discs (15 mm diameter and 1.8 mm thickness) were fabricated in a tungsten carbide matrix by uniaxial pressing at 1,148 Kgf for 30 seconds. 3) Isostatic pressing of discs previously sealed in plastic packages. 4) Sintering at 1,600 °C. Two-sided polishing with diamond discs of increasingly finer grits (220, 120, 90, 40, 25, and 9 μm) and suspensions (6 and 1 μm). Final dimension sample with 12 mm diameter and 1 mm thickness as per ISO 6872:2015.

Table 1
Characteristics of translucent powder (ZPEX, Tosoh Corporation, Tokyo, Japan).

Material	Particle size (μm)	Powder characteristics (mass %)					
		Y_2O_3	HfO_2	Al_2O_3	Na_2O	SiO_2	FeO_20_3
ZPEX	0.04	5.2 ± 0.2	< 5.0	≤0.1	≤0.04	≤0.02	≤0.01

Table 2
Composition characteristics of the ZTA (Al_2O_3 80% and ZrO_2 20%) (alumina powder, Baikalox Regular CR10).

Sample	Density Al2O3 (mass % t)
Al2O3 80% and ZrO2 20%	74.05

Table 4
Density of the ceramics based on alumina and zirconia.

Material	Density of sintered disc (g/cm3)
ZTA ZPEX 80/20	98.2% ρt
ZPEX	99.8% pt

ambient air (± 23 °C). The imaging and indentation processes were performed using a Berkovich fluid cell diamond three-sided pyramid probe in a nanoindenter (950 TI; Hysitron, Minneapolis, MN, USA). A loading profile was developed with a peak load of 8000 μN achieved in 15 s (80000 μN/s) followed by the complete unloading in 2 s, with a hold time for a load of 60 s. The test was conducted under dry conditions in the center, top, bottom, left, and right regions of the discs (n = 3/group). Each region of interest (ROI) received 10 nanoindentations with 5 μm (horizontally) and 8 μm (vertically) of minimum separation.

Since this test was nondestructive, the same samples were used for repeated nanoindentation after a simulated LTD aging protocol and also to standardize the measurements.

2.6. X-ray diffraction

The progress of the t-m transformation due to the accelerated aging was analyzed by XRD (X'pert Power PANalytical, Netherlands) using Cu Kα radiation. The XRD analysis was conducted between 20° and 80° (2θ) with a step size of 0.02°. The monoclinic phase content (%) was quantified using formulas introduced by Toraya and Yoshimura [23]. Eqs. (1) and (2) were obtained using the intensity of the peak top at 28°, 30°, and 31.2°. The peak intensities were obtained after the baseline subtraction.

$$X_m = \frac{[I_m(-111) + I_m(111)]}{[I_m(-111) + I_m(111) + I_t(101)]} \quad (1)$$

$$V_m = 1.311 \times \left(\frac{X_m}{1}\right) + (0.311 \times X_m) \quad (2)$$

In the first formula, I_m (−111) and I_m (111) represent the monoclinic peaks' intensity (2θ = 28° and 2θ = 31.2°, respectively) and I_t (101) indicates the intensity of the tetragonal peak (2θ = 30°). In the second formula, V_m represents the monoclinic volumetric content.

2.7. Scanning electron microscopy

Scanning electron microscopy analysis was performed after thermal treatment at 1,520 °C for one hour with heating and cooling rates of 5 °C for one minute in a Zyrcomat (Vita Zahnfabrink, Bad Säckingen, Germany) in the LS15 microscope (Carl Zeiss, Oberkochen, Germany). The working distance (WD) used ranged from 8, 11, 12, to 12.5 mm with magnifications of up to 3,000x.

2.8. Statistical analysis

The data were statistically evaluated through repeated measures

Table 3
Estimated marginal means and correspondent 95% confidence intervals (CIs) of the surface roughness of each group before and after aging and the fraction of the monoclinic transformation.

Material	Autoclave treatment		Surface roughness (μm)		Monoclinic phase
			Sa	Sq	%
ZTA ZPEX 80/20	Before aging	Lower bound	0.189	0.235	
		Mean	0.250aA	0.310aA	3.07
		Upper bound	0.311	0.385	
	After aging	Lower bound	0.207	0.263	
		Mean	0.270aA	0.341aA	4.95
		Upper bound	0.335	0.418	
ZPEX	Before aging	Lower bound	0.161	0.198	
		Mean	0.222aA	0.273aA	1.3
		Upper bound	0.283	0.348	
	After aging	Lower bound	0.169	0.202	
		Mean	0.233aA	0.279aA	28.25
		Upper bound	0.297	0.357	

Different lowercase letters mean statistical differences before and after aging treatments within the groups.
Different uppercase letters mean statistical differences between the groups within the same aging protocol.

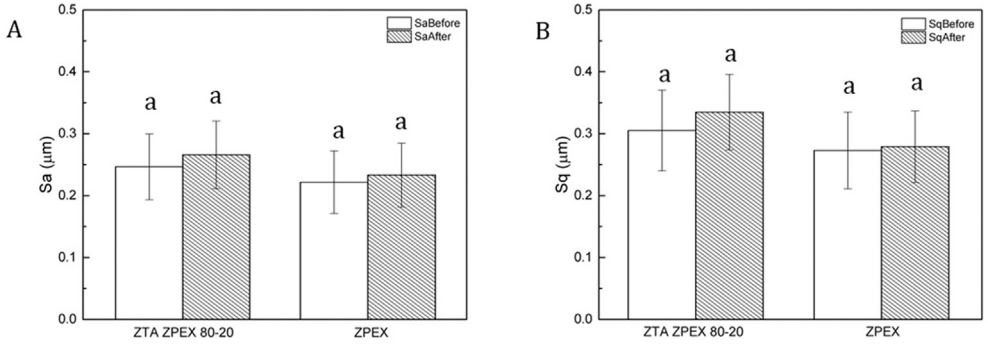

Fig. 2. Estimated marginal means and correspondent 95% confidence intervals (CIs) of the surface roughness of each group before and after aging.

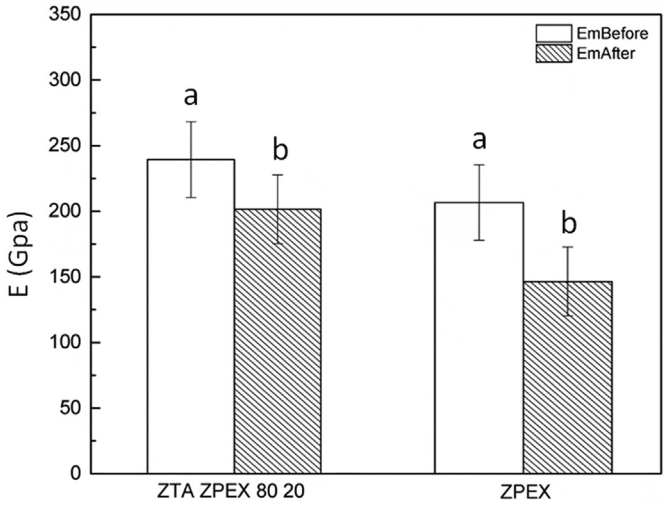

Fig. 3. Estimated marginal means and correspondent 95% confidence intervals (CIs) of the elastic modulus of each group before and after aging. The elastic modulus was significantly reduced after the aging of both materials.

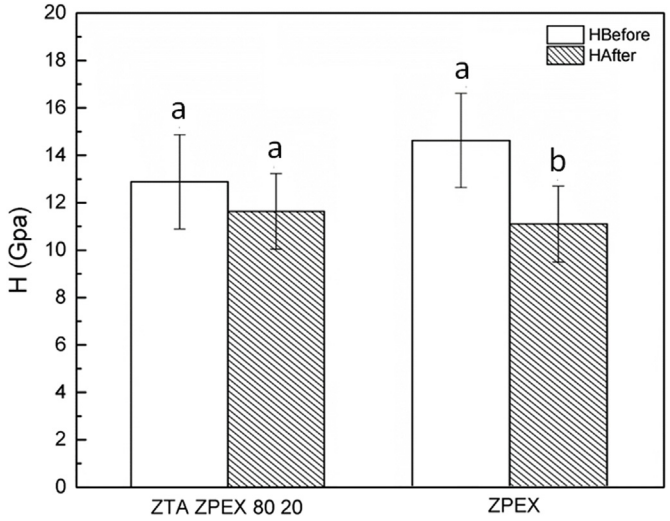

Fig. 4. Estimated marginal means and correspondent 95% confidence intervals (CIs) of the hardness of each group before and after aging. The hardness was significantly reduced in the ZPEX group after aging.

ANOVA following post-hoc comparisons by Tukey's test, with the significance level set at $p < 0.05$. The data are presented as a function of the estimated mean values with the corresponding 95% confidence intervals (CI). All of the analyses were accomplished using SPSS (IBM SPSS 23, IBM Corp., Armonk, NY, USA).

3. Results

3.1. Theoretical density

The percentage theoretical density values of the ZTA ZPEX 80/20 (4.419 g/cm^3) and ZPEX (6.081 g/cm^3) were calculated based on the mixture of the alumina (3.98 g/cm^3) and zirconia (6.09 g/cm^3)

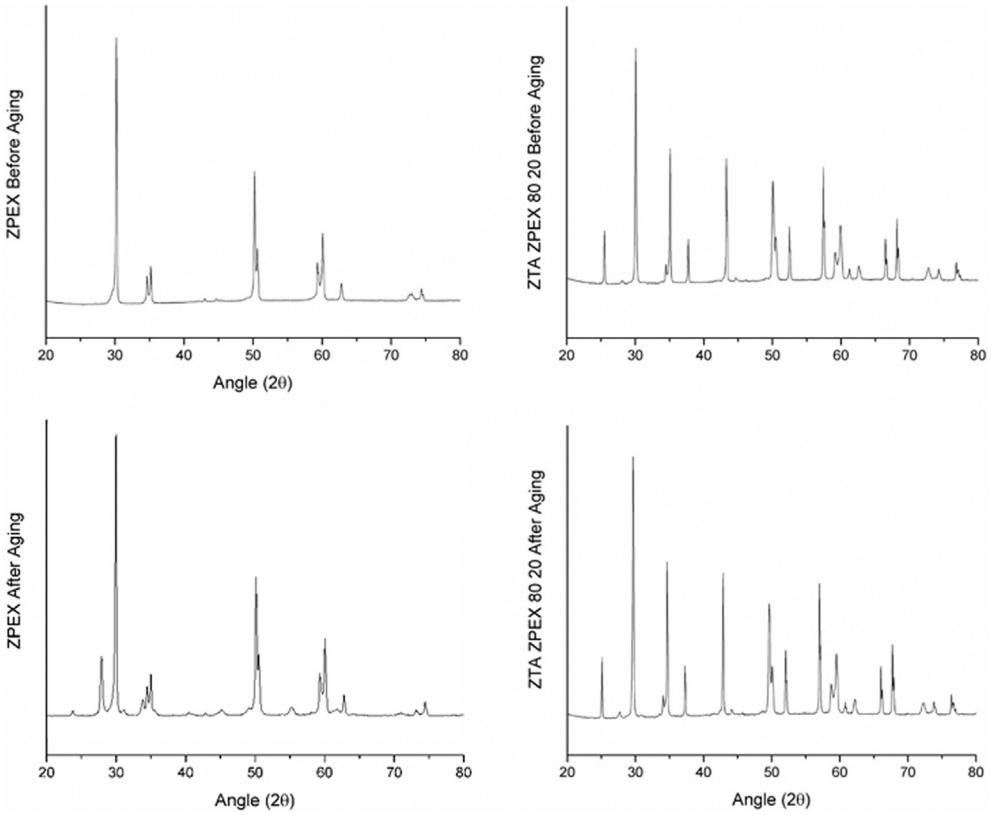

Fig. 5. XRD patterns of the tested groups before and after aging. The increase in the monoclinic peak (2θ = 28° and 2θ = 31.2°) in the ZPEX group relative to the ZTA composite is demonstrated.

theoretical densities. Density on the order of 99% of the theoretical density was obtained using the method based on Archimedes' principle to the ZTA ZPEX 80/20 and ZPEX (Table 4).

3.2. Interferometry

Table 3 lists the estimated marginal means and correspondent 95% confidence intervals (CIs) for the surface roughness of each group before and after aging. The results of the surface roughness are shown in Fig. 2. Aging did not affect the surface roughness (Sa and Sq) of the evaluated groups ($p > 0.05$).

3.3. Nanoindentation

A statistical summary of the results of the elastic modulus (E) is presented in Fig. 4. The ZTA ZPEX 80/20 and ZPEX did not show significant differences in their E values before aging ($p = 0.098$). However, after simulated LTD aging, the ZPEX demonstrated statistically lower E values compared to the ZTA ZPEX 80/20 ($p = 0.011$). Both groups presented a statistically significant decrease in their E values after aging ($p < 0.01$) (Fig. 3).

A statistical summary of the results of the hardness (H) is presented in Fig. 4. Both groups demonstrated similar hardness values before and after aging ($p > 0.568$). The ZTA ZPEX 80/20 did not show statistically significant differences after aging ($p = 0.357$), while the ZPEX presented a significant decrease in the hardness values after aging ($p = 0.030$) (Fig. 4). The ZTA ZPEX 80/20 did not show statistically significant differences compared to the ZPEX.

3.4. X-ray diffraction

The XRD spectra of the experimental groups are presented in Fig. 5. The ZPEX group demonstrated a higher increase in the monoclinic peaks (2θ = 28° and 2θ = 31.2°) compared to the ZTA ZPEX 80/20 after aging. The Toraya and Yoshimura [23] equation also depicted a higher percentage of monoclinic content for the ZPEX (28.25%) compared to the ZTA ZPEX 80/20 (4.95%) after aging (Table 3).

3.5. Scanning electron microscopy

SEM micrographs show morphological analyses of a dense surface for the ZTA composites. Few microstructural defects were observed on the surface and are considered intrinsic to ceramic material processing (Fig. 6).

4. Discussion

Yttrium tetragonal zirconia polycrystal (Y-TZP) is a viable alternative to some previously utilized materials but presents hydrothermal instability at low temperatures due to the metastable transformation of the tetragonal to monoclinic phase. Although this phase transformation is responsible for the high fracture toughness of zirconia-based materials, this advantage may be compromised if the material undergoes spontaneous phase transformation in the presence of water or mechanical stress [1–3]. This phenomenon is referred to as low temperature degradation (LTD) and leads to phase transformation, eventually compromising the mechanical properties and reliability of prosthetic treatments in the long-term. Therefore, new polycrystalline ceramic compositions are warranted to better stabilize Y-TZP while maintaining some of its desired mechanical properties [15,24,25]. Previous studies have demonstrated that the best balance between the hardness, toughness, and hydrothermal resistance may be achieved through alumina-zirconia composites for the improvement of polycrystalline ceramic properties [26]. The combination of both materials allows improvement to the mechanical strength and toughness compared to alumina while minimizing the occurrence of LTD as observed in Y-TZP

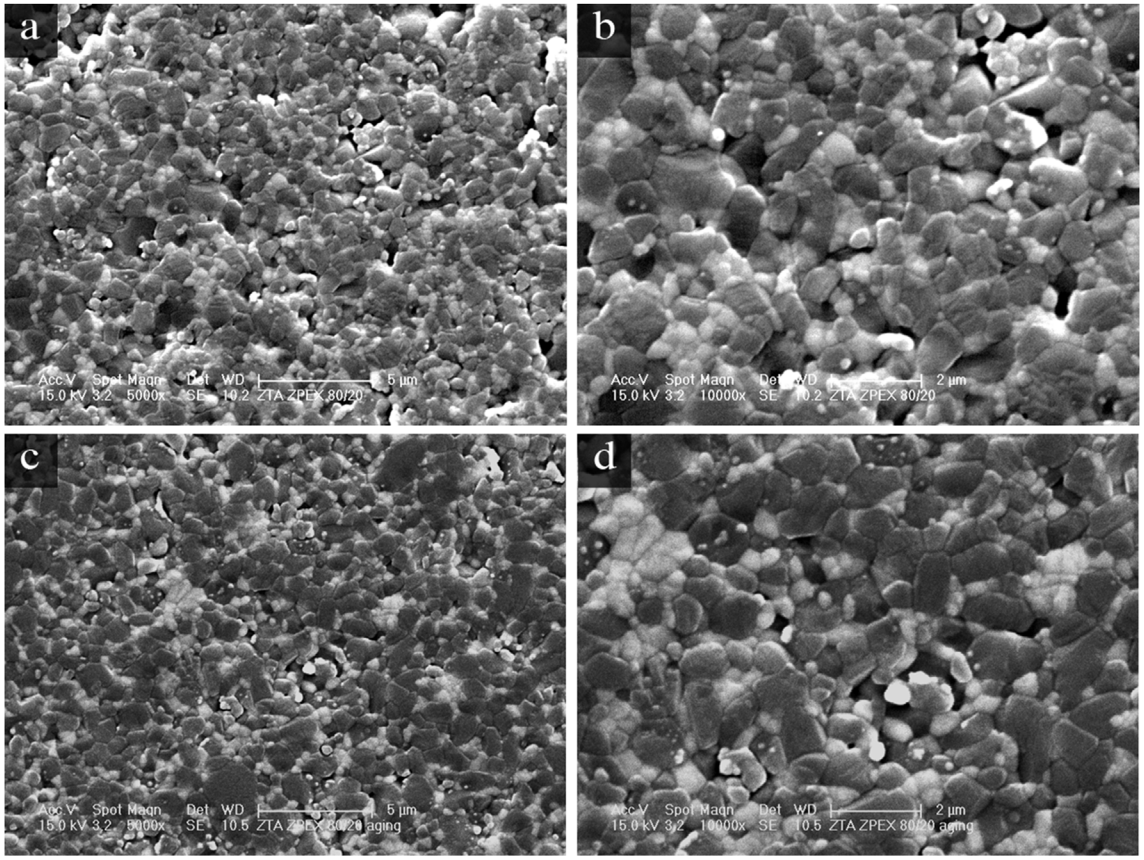

Fig. 6. SEM micrographs of the ZTA composite surface before aging (a, 5,000x; b, 10,000x), and after aging protocol (c, 5,000x; d, 10,000x). The images demonstrate the microstructural arrangement and final density obtained for the ZTA composite, where a few defects related to ceramic processing can be observed. In addition, the established aging protocol did not cause evident microstructural changes.

[26]. This study aimed to develop a polycrystalline composite for restorative purposes that compounds the individual properties of alumina and zirconia for improved aging resistance to LTD compared to zirconia.

With respect to the LTD simulation, the specimens were subjected to a rigorous protocol in the autoclave (20 h in water vapor at 134 °C and 2.2 bars) to simulate several years of in vivo service. This protocol was selected based on previous literature, [1,27–30] illustrating that such conditions promote an extensive t-m phase transformation (approximately 55–80% m-phase content) as well as permitting enough time to observe any differences in the susceptibility to degradation promoted by the aging of both materials.

The potential detrimental effects of the phase transformation may be evaluated in terms of surface roughness increase. Several authors have reported alterations in the mechanical properties of experimentally aged zirconia-based ceramics [31]. The phase transformation of stabilized tetragonal zirconia to the less compact monoclinic phase entails a 3–4% volume expansion [32,33]. As the grains expand, the surface of the zirconia may present increased roughness. In the present study, the tested experimental group, ZTA composite, and translucent zirconia showed a slight increase in the surface roughness (Sa and Sq) after aging; however, the magnitude of this change was not significant. It was previously reported that only roughness values greater than 250 nm can be detected by the patient and lead to bacterial plaque accumulation [34,35]. Nonetheless, slight changes in the roughness can potentially lead to significantly poor fatigue performance [36].

Previous studies using nanoindentation techniques reported decreased hardness and elastic modulus for aged zirconia [37–39]. The values of the hardness and Young's modulus are partially in agreement [36] with previous studies, [32,40] and our analysis showed that they were significantly affected by the aging protocol, especially in the ZPEX group, with significantly lower hardness and elastic modulus values after aging. However, for the ZTA ZPEX 80/20, only the H values were significantly affected by aging. It is noteworthy that the experimental translucent Y-TZP (ZPEX) demonstrated more than 24% and 29% decreases in the hardness and E values, respectively, after aging. In contrast, the experimental ZTA composite presented a markedly lower reduction of approximately 9% for the H and 15% for the E values. The reduction in hardness in aged zirconia can likely be attributed to the presence of the monoclinic phase, which has a lower atomic density compared to the tetragonal phase [41]. The reduction in the modulus of elasticity resulted from the cracks caused by the volumetric expansion during the tetragonal to monoclinic phase transformation [37].

A microstructural analysis performed by XRD revealed the preservation of the crystalline structures of the tetragonal zirconia and alpha alumina, with evidence of a higher increase in the monoclinic peak of the zirconia phase in the ZPEX group. In addition, SEM images obtained after thermal treatment (1,520 °C) highlighted the grain boundaries that exhibited a dense surface with a homogeneous distribution of the zirconia and alumina grains. Few zirconia agglomerates and porosities were observed on the experimental ZTA composite surface. With an understanding of the properties characterized at the nanoscale and the surface topographical parameters presented both before and after simulated LTD aging, future studies to characterize fracture toughness, flexural strength, and fatigue parameters with fractographic analysis are warranted. Cell viability tests using fibroblasts and experiments following ISO 10993 are needed to characterize the biological response of the composites synthesized in this experiment, with comprehensive molecular signaling experiments considering the potential applications of ZTAs in dentistry and as biomedical devices.

5. Conclusions

A method for the synthesis of an aging resistant ZTA composite was described herein. Both nanomechanical properties (modulus of elasticity and hardness) decreased in the aged translucent zirconia while only the hardness values did not change in the experimental ZTA. The surface roughness parameters were not affected by aging, but the translucent zirconia presented a significant extent of tetragonal to monoclinic phase transformation compared to the ZTA ZPEX 80/20, which was resistant to the accelerated aging protocol.

Acknowledgments

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