Effect of ceramic thickness, grinding, and aging on the mechanical behavior of a polycrystalline zirconia

Rodrigo Diniz PRADO[a]
Gabriel Kalil Rocha PEREIRA[b]
Marco Antonio BOTTINO[a]
Renata Marques de MELO[a]
Luiz Felipe VALANDRO[b]

[a] Universidade Estadual Paulista – Unesp, Dental School, Prosthodontics Units, São José dos Campos, SP, Brazil.

[b] Universidade Federal de Santa Maria – UFSM, Faculty of Odontology, Prosthodontics Units, Santa Maria, RS, Brazil.

Abstract: Monolithic restorations of Y-TZP have been recommended as a restorative alternative on prosthetic dentistry as it allows a substantial reduction of ceramic thickness, which means a greater preservation of tooth structure. However, the influence of grinding and aging when using a thinner layer of the material is unclear. This investigation aimed to evaluate and compare the effects of ceramic thickness (0.5 mm and 1.0 mm), grinding and aging (low-temperature degradation) on the mechanical behavior and surface characteristics of a full-contour Y-TZP ceramic. Y-TZP disc-shaped specimens (15 mm diameter) were manufactured with both thicknesses and randomly assigned into 4 groups considering the factors 'grinding with diamond bur' and 'aging in autoclave'. Surface topography (roughness, 3D profilometry and SEM), phase transformation, flexural strength and structural reliability (Weibull) analyses were executed. Grinding affected the surface topography, while aging did not promote any effect. An increase in *m*-phase content was observed after grinding and aging, although different susceptibilities were observed. Regardless of zirconia's thickness, no deleterious effect of grinding or aging on the mechanical properties was observed. Thus, in our testing assembly, reducing the thickness of the Y-TZP ceramic did not alter its response to grinding and low temperature degradation and did not impair its mechanical performance.

Keywords: Prosthodontics; Ceramics.

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Corresponding Author:
Luiz Felipe Valandro
lfvalandro@hotmail.com

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Introduction

A major advantage of Yttrium-stabilized Tetragonal Zirconia Polycrystal (Y-TZP) is its high fracture resistance, having a flexural strength of 900–1000 MPa and fracture toughness of 5.5 to 7.4 MPa/m^2.[1] These properties are very important and demonstrate an enhanced ability to resist to the occlusal forces generated during chewing, when compared with lithium disilicate ceramic, for example, which has a flexural strength of 350 MPa and a fracture toughness of 3.2 MPa/m^2.[2]

Hence, zirconia-based materials are the most resistant among the existing dental ceramics – they are used in the manufacturing of frameworks of single or multi-unit FPDs (fixed partial dentures), and recently, for monolithic restorations.[3,4] The major advantage of monolithic restorations is that they allow a substantial reduction of ceramic thickness by eliminating the veneer layer without compromising the final strength of the system. Thus, this assembly results in a more conservative tooth preparation and decreased removal of tooth structure.[3,5] Besides, clinical studies have shown the fracture or chipping of the veneering porcelain as the main complication for FPDs, another advantage of using monolithic restorations.[1]

Zirconia is a polymorphic metastable material, naturally occurring in three different structural forms (monoclinic – *m*; tetragonal – *t*; and cubic – *c*). Without the addition of stabilizers, such as 3% mol of yttrium oxide (stabilized zirconia at *t*-phase form), pure zirconia does not present the stability required for use in biomedical fields. However, even with the addition of stabilizers, phase transformation (*t* to *m* phase) still may take place in response of mechanical, physical and/or chemical stimuli.[6,7]

The *t-m* phase transformation causes a local volumetric expansion of approximately 3–4%, resulting in surface compressive stress concentration that, when located near surface defects/cracks, will act as a defense mechanism to prevent crack propagation.[8,9] Firstly, this phase transformation mechanism occurs on superficial grains, with water incorporation and filling of the oxygen spaces. Subsequently, it spreads, resulting in rougher surface, and a zirconia with decreased hardness, fracture toughness and density,[7] which is known as low-temperature degradation.[10]

As previously mentioned, monolithic restorations allow a substantial reduction on material thickness, allowing a more conservative preparation and resulting in greater preservation of the tooth structure, reducing the risk of injury to the pulp and of trauma.[11] However, there is scarce information in the literature regarding the influence of adjustments (to enhance occlusal relations, fit and emergence profile) and aging when thinner layers of the material are considered. Nakamura et al.[5] showed that with a reduction on occlusal thickness the load to fracture is also reduced. Therefore, the raised question is: would a reduced Y-TZP ceramic thickness lead to a higher susceptibility of defects due to grinding and aging, and affect the mechanical behavior?

Thus, the present study aimed to evaluate the influence of ceramic thickness, grinding with diamond bur and aging in autoclave on the flexural strength, structural reliability, topography and phase transformation of a zirconia indicated for monolithic restoration. The study hypothesis was "a reduced ceramic thickness will predispose to an impairment on mechanical performance after grinding and aging".

Methods

Sample preparation

Disc-shaped specimens were manufactured according to ISO 6872-2008[12] for biaxial flexure strength testing of ceramic materials (final dimensions of 15 mm in diameter), using the methodology previously described in the literature.[13,14,15] Basically, pre-sintered blocks of Y-TZP (Lot no. 637590 Rev.0 2011-03, Zirlux FC, Ardent Dental, Inc, Amherst, NY, USA – which present approximately 20% of shrinkage, as declared by the manufacturer) were ground into cylinders of 18 mm diameter using 600–1200 grit silicon carbide (SiC) paper (3M, St Paul, MN, USA) under water-cooling. The cylinders were cross-sectioned using a precision saw machine (ISOMET 1000, Buehler, Lake Bluff, IL) and polished with SiC papers (600–1200 grit) to produce disc-shaped samples with pre-sintering thickness of approximately 0.6 mm and 1.2 mm. Then the ceramic samples were sintered as recommended by the manufacturer's guidelines, having final dimensions of 15 mm in diameter and 0.5 or 1 mm in thickness. Finally, the specimens of both thicknesses were randomly divided into 4 groups (n = 30), considering the factors "grinding" and "aging (low-temperature degradation)", as it shows in Table 1.

Surface Treatment

Samples from the control group (C) remained untouched after the sintering process – "as-sintered" samples.

Grinding

Grinding was performed by a single trained operator following the protocol recommended on the systematic review by Pereira et al.[16] Extra-fine diamond burs (#3101FF – grit size 30 µm; KG Sorensen, Cotia, Brazil) coupled to a contra-angle handpiece (T2 REVO R170 contra-angle handpiece of 170,000 rpm, Sirona, Bensheim, Germany) combined with a low-speed motor

Table 1. Experimental design.

Thickness (mm)	Surface treatment	Low-temperature aging in autoclave	Group code	Sample size (n)
0.5	Control, *as-sintered* (without any additional treatment)	No	C	30
		Yes	C-Ltd	30
	Grinding with extra-fine diamond bur (#3101FF – 30 μm grit size; KG Sorensen, Cotia, Brazil)	No	G	30
		Yes	G-Ltd	30
1.0	Control, *as-sintered* (without any additional treatment)	No	C	30
		Yes	C-Ltd	30
	Grinding with extra-fine diamond bur (#3101FF – grit size 30 μm; KG Sorensen, Cotia, Brazil)	No	G	30
		Yes	G-Ltd	30

(Kavo Dental, Biberach, Germany) under constant water-cooling (≈30 ml/min) were used; the diamond bur was replaced after each specimen. Caution was taken to increase reproducibility and standardize wear thickness during grinding, following the grinding protocol previously described in the literature.[14,17,18]

Low-temperature aging

Low-Temperature Degradation (LTD) was simulated in an autoclave (Sercon HS1-0300 nº1560389/1) at 134°C, under 2 bar, over a period of 20 h, following the protocol suggested on the systematic review by Pereira et al.[19]

Surface analysis (Topography, Roughness and Phase transformation) SEM analysis and 3D optical profilometer analysis

The superficial topography was accessed using scanning electron microscope (JSM-6360, JEOL, Tokyo, Japan). For that, the specimens (n = 2) were coated with a gold-palladium alloy spray and inspected to evaluate the surface pattern.

Additional analysis (n = 2) was performed under 3D optical digital profilometer (Wyko, NT 1100, Veeco, EUA). which was connected to a computer using the software Wyko Vison 32 (Veeco, EUA).

Roughness analysis

Surface roughness analysis was performed for all specimens in all conditions (n = 30, Mitutoyo SJ-410, Mitutoyo Corporation, Takatsu-ku, Kawasaki, Kanagawa, Japan): three measurements were made for each specimen according to the ISO 1997 parameters (Ra – arithmetical mean of the absolute values of peaks and valleys measured from a medium plane (mm) and Rz – average distance between the five highest peaks and five major valleys found in the standard (mm)) with a cut-off (n = 5), λC 0.8 mm and λS 2.5 μm. Mean values of all measurements from each specimen were then obtained.

Phase analysis by X-ray diffraction (XRD Analysis)

Quantitative analysis of phase transformation was conducted (n=3) to determine the relative amount of m-phase and depth of the transformed layer under each condition. The analysis was performed using an X-ray diffractometer (Bruker AXS, D8 Advance, Karlsruhe, Germany) with a CuKα radiation. Spectra were collected using the Bragg-Brentano geometry in the 2θ range from 25 to 35 degrees, at a step interval of 1 s, and step size of 0.03 degrees/step.

The amount of *m*-phase was calculated using the method introduced by Garvie and Nicholson[20] modified by Toraya et al.[21] This method has been extensively used and described in the literature, as found in the systematic review by Pereira et al.[16]

The depth of the transformed layer was calculated based on the amount of the *m*-phase, considering that a constant fraction of grains had symmetrically transformed to the *m*-phase along the surface, as recommended by Kosmac et al.[22]

Biaxial flexure test

Specimens (n = 30) were subjected to a biaxial flexural strength test according to ISO 6872-2008,[12] as previously described.[13,14,15] Specimens were placed on three support balls (Ø 3.2 mm) positioned equidistant from each other, with the treated surface facing down (tensile stress concentration area), under water; a circular tungsten flat piston (Ø = 1.6 mm) was used to apply the load (1 mm / min) with a universal testing machine (EMIC DL-1000, EMIC, São José dos Pinhais, Brazil) until the catastrophic failure of the material. A 1000 kgf load was applied to the disc center. A covering tape was positioned where the load was applied to prevent the dispersal of fragments[23] and to promote better contact between the piston and the specimen.[24]

Data analyses

A descriptive analysis of the roughness data (Ra and Rz) was performed in Minitab 16 program (Minitab 17.1.0, Minitab, Inc, USA) to determine the mean and standard deviations. As data presented a parametric and homogeneous distribution (using Shapiro-Wilk and Levene tests, respectively), the two-way ANOVA and the *post-hoc* Tukey's test were performed considering the two factors (grinding and aging) and the interaction between them. Additionally, a Pearson correlation analysis was made with the roughness Ra and the biaxial strength parameters.

The Weibull statistical analysis was carried out to describe the structural homogeneity of the ceramic material, which is a way to describe the resistance variation[25]. Thus, the modulus (m) and the Weibull characteristic strength (σc) were obtained with 95% confidence interval as determined in the diagram (in accordance with DIN ENV 843-5[26]). The characteristic strength is the strength at a failure probability of approximately 63%, and the Weibull modulus is used as a measure of the distribution of strengths, expressing the reliability of the material.

Results

3D Profilometer images and SEM micrographs showed that grinding produced an alteration of surface pattern, with multi-directional scratches following the movement of the diamond bur, while the "as sintered" group (control) showed a homogeneous surface (Figure 1). Aging did not cause any relevant alteration of those patterns.

Roughness analysis showed that, for both Ra and Rz parameters, the surface treatment presented a statistical difference ($p < 0.05$), while aging (Ra: $p = 0.348$; Rz: $p = 0.119$) and the interaction of factors (Ra: $p = 0.337$; Rz: $p = 0.407$) did not. Grinding with diamond burs led to statistically significant higher roughness values (Ra and Rz parameters) in comparison to absence of grinding ($p < 0.05$). Additionally, aging in autoclave (LTD) did not lead to any impact on roughness (Table 2). Regarding the Pearson correlation test between Ra and biaxial strength, a very weak positive statistical correlation was observed ($p = 0.280$).

The low temperature regimen promoted an increase in *m*-phase content, which was more intense for the control group (58.17%). Grinding also increased the *m*-phase content, although it led to a decrease in the material susceptibility to new phase transformations during aging (Figure 2).

Independent of zirconia's thickness, the low-temperature aging and grinding led to an increase in characteristic strengths, when compared to control groups (Table 2). Thus, the reduced thickness (0.5 mm) did not promote lower characteristic strength compared with thicker specimens (Table 2). The low-temperature aging and grinding had no influence on Weibull modulus (*m* value) of the groups with different zirconia thickness (i.e. neither grinding with diamond bur nor the aging protocol significantly reduced the material reliability).

Discussion

The current study found that grinding and low-temperature aging in autoclave did not promote a negative effect on the characteristic strength and structural reliability (Weibull moduli) of the material, for both Y-TZP thicknesses (0.5 and 1 mm). Thus, the hypothesis that a reduced Y-TZP ceramic thickness would increase impairment on mechanical performance after grinding and aging was rejected.

3D optical profilometry and SEM images showed an irregular surface pattern with many peaks and valleys due to grinding with diamond bur. Additionally, XRD analysis showed that grinding leads to *m*-phase content increase (approximately 9% of *m*-phase content as noticed on Figure 2). Consequently, ground specimens showed higher values of characteristic strength and this can be explained by the transformation toughening mechanism counterbalancing any potential critical defect introduced by grinding[9].

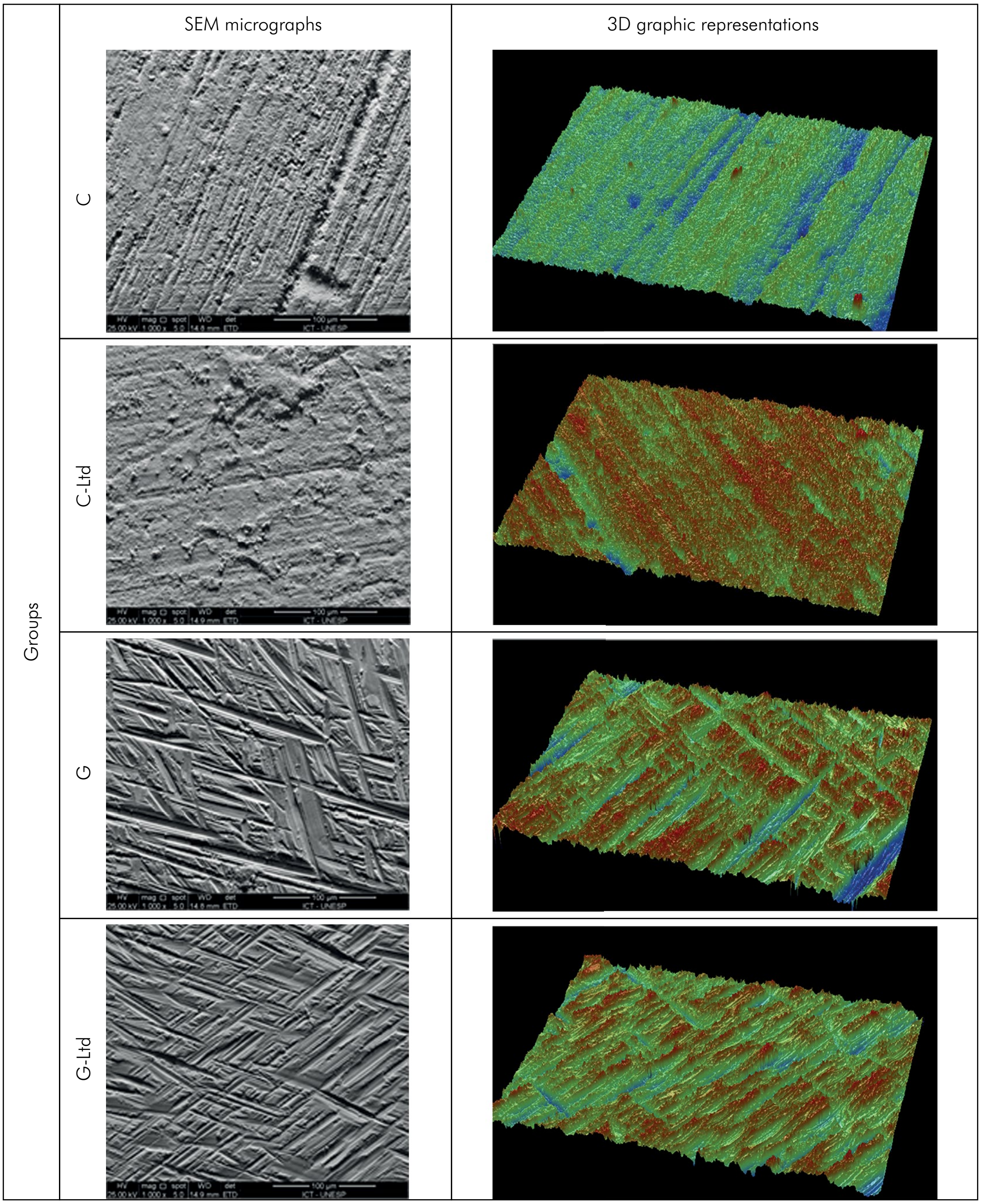

Figure 1. SEM micrographs (1000 x magnification) and 3D graphic representations showing different topographical patterns of specimens subjected to studied conditions. It highlights that both evaluated ceramic thicknesses (0.5 and 1 mm) presented the same topographical pattern and that grinding procedure affects the materials topography changing its superficial pattern, while low temperature aging appears to not interfere in such outcome (C- as-sintered; G- grinding; Ltd- low-temperature degradation)

When a crack initiates and propagates towards the bulk of the material, tensile stress concentration at the crack tip leads the surrounding area to a monoclinic phase transformation (responsible for a ~4% volumetric expansion). The crack becomes surrounded by transformed zirconia, producing a compressive stress in this zone, making crack propagation more difficult.[8,9,15]

Thus, even though the groups subjected to grinding procedure presented higher values of roughness (Ra and Rz), it did not have a negative impact on their characteristic strength. Another potential explanation for that might be related to the grinding protocol used by us (diamond bur of 30-µm grit size, low-speed motor coupled to a contra-angle handpiece, capable to multiply the velocity and water-cooling). This allows a better control of the grinding procedure (in comparison to normal high speed handpieces), producing less defects and triggering the transformation toughening mechanism.[14,16,27]

These findings are not in agreement with previous literature,[28,29,30,31] in which grinding with diamond burs did not promote an increase in *m*-phase content and created severe defects on the material surface leading to degradation of the mechanical properties. A high-speed handpiece used by those authors can lead to a temperature increase at the Y-TZP surface, consequently triggering a reverse *m-t* transformation that works against the transformation toughening mechanism.[30,31,32]

From that viewpoint, the presence of abundant water cooling and the use of a gentle grinding protocol seems to contribute to the maintenance of the mechanical properties of the material[16], whereas grinding without proper cooling or with an aggressive protocol may raise the surface temperature[30,31,32] to the critical point where *t-m* phase transformation occurs. Consequently, more defects are produced to the material and the occurrence of the transformation toughening mechanism is prevented, resulting in decrease of the Y-TZP strength.[28,29,30,31,33,34,35]

Our data shows that surface roughness should not be solely used to predict the mechanical behavior of Y-TZP ceramics, since the *t-m* phase transformation (increase in *m*-phase content) may counterbalance any potential defect inflicted and lead to a final increase

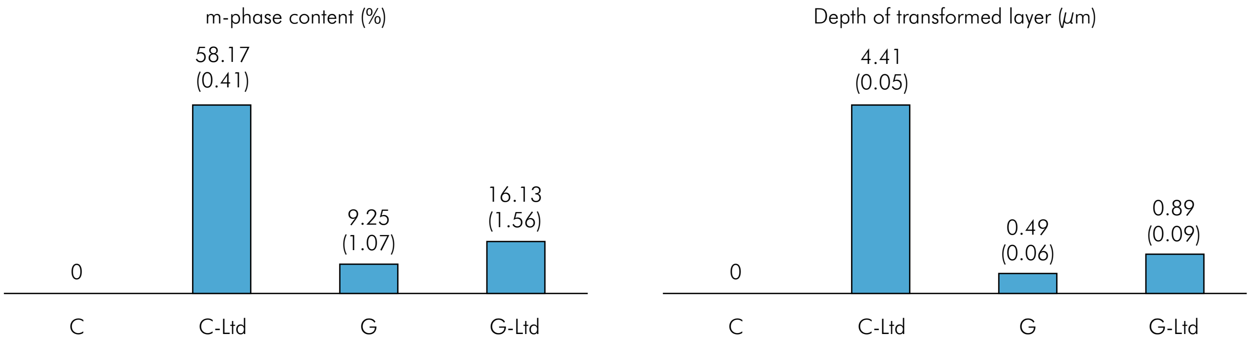

Figure 2. Mean percentage of monoclinic phase content (%) of the Y-TZP surface and depth of transformed layer (µm), in addition to their respective standard deviations (data inside parentheses). It highlights the highest m-phase content at as-sintered after aging condition (C-Ltd), as well as, the deepest layer of phase transformation (µm). Grinding procedure appears to trigger an initial m-phase content increase, although it prevents new phase transformation during aging (C- as-sintered; G- grinding; Ltd- low-temperature degradation).

Table 2. Analysis of variance (two-way ANOVA) of roughness data (Ra and Rz), characteristic strength (σ_c), Weibull modulus (m) and respective confidence intervals (95%CI) for treatment and aging factors.

Thickness (mm)	Groups	Roughness (µm)		Weibull analysis			
		Ra	Rz	σ_c	95%CI	m	95%CI
0.5	C	0.4 (0.1)^{C}	3.4 (0.8)^{BC}	726.9^{A}	688.74–766.00	8.1^{AB}	6.13–10.63
	C-Ltd	0.4 (0.1)^{C}	3.1 (1.0)^{C}	1183.3^{D}	1087.26–1284.78	5.1^{A}	3.14–8.40
	G	0.6 (0.2)^{A}	4,0 (0.8)^{AB}	986.3^{BC}	923.98–1051.02	6.7^{AB}	4.67–9.48
	G-Ltd	0.5 (0.1)^{AB}	3.6 (0.7)^{ABC}	1162.3^{D}	1120.07–1204.92	11.8^{B}	9.41–14.65
1.0	C	0.4 (0.2)^{BC}	3.6 (0.9)^{ABC}	760.7^{A}	725.36–796.75	9.1^{AB}	7.02–11.87
	C-Ltd	0.5 (0.2)^{BC}	3.7 (1.0)^{ABC}	1023.9^{C}	972.83–1076.15	8.5^{AB}	6.65–10.85
	G	0.6 (0.1)^{A}	4.0 (0.8)^{A}	907.1^{B}	861.73–953.59	8.5^{AB}	6.07–11.80
	G-Ltd	0.6 (0.1)^{A}	4.0 (0.7)^{AB}	1027.7^{C}	971.26–1085.75	7.7^{AB}	5.31–11.15

in the material mechanical performance, which is also demonstrated by previous *in vitro* studies.[14,17,33]

Therefore, the surface modifications due to grinding are able to cause positive or negative effects on the mechanical properties of Y-TZP materials. If the defect produced by grinding has a greater depth than the one of the compressive layer created by *t-m* phase transformation, it may result in higher levels of tensile stress concentration, raising the incidence of catastrophic failures.[28,30,36] However, when these defects have a smaller depth than the one of the compressive stress layer (created by transformation toughening mechanism), the crack propagation and catastrophic failures are avoided by the surrounding compressive stresses.[7,37]

Regarding aging, the as-sintered condition showed an extensive increase in σ_c after the aging protocol, and this result is probably due to an intense increase in monoclinic phase content (58.17%) leading to transformation, and contributing to the increase in toughening. It is important to note that even at smaller thickness (0.5 mm) this monoclinic phase content did not lead to any deleterious influence on σ_c, which corroborates the assertion that this material can be used even with reduced thickness.

The different values of *m*-phase for the as-sintered group and grinding groups after aging (C-Ltd – 58.17%, G-Ltd – 16.13%) support another important fact: ground surfaces seem to be less susceptible to *t-m* phase transformation during aging, which is probably caused by a protective effect achieved by formation of the tension barrier by *t-m* transformation on the surface of the material.[38]

According to Muñoz-Tabarez et al.,[39] the microstructural changes induced by grinding of Y-TZP consist of three well defined layers, from the surface to the interior: a) a superficial crystallized zone, where grain diameter range from 10 to 20 nm approximately; b) a plastically deformed zone; and c) a zone in which tetragonal to monoclinic phase transformation take place, which is mainly responsible for the formation of compressive residual stresses that usually increases the flexure strength and apparent fracture toughness of ground specimens.[39]

Thus, the increased resistance to hydrothermal degradation after grinding is possibly related to the existence of this very thin layer of tetragonal recrystallized nano-grains (10–20 nm) that are smaller than the critical size for transformation in humid environment, in addition to the presence of residual compressive stress on the surface of Y-TZP ceramics.[39,40]

Although the tested Y-TZP ceramic consists of a new translucent material, and therefore alterations on microstructure and composition (that are not clearly stated by the manufacturer) may be expected in comparison to conventional Y-TZP ceramics[4], our data support a similar performance of the tested ceramic to the ones that have been evaluated by previous studies.[15,16,17,18,19]

Finally, we showed that the protocols for grinding and aging used by this study do not promote a deleterious effect on the mechanical properties of Y-TZP ceramic, regardless of the ceramic thickness. However, more studies subjecting the Y-TZP materials to drastic environments such as fatigue tests, long periods in the presence of moisture, as well as crown-shape sample testing set-ups, should be conducted to support these findings.

The test assembly recommended by ISO 6872-2008[12] and used by us requires that the surface being tested be positioned directed to the tension side (where the fracture originates). Clinically, however, when doing occlusal adjustments with diamond burs on a monolithic crown, the surface subjected to grinding will be mostly under compression during chewing. Thus, this might be considered an important limitation of our study.

Although our study shows that a thinner layer (0.5 mm) did not impair the mechanical performance of Y-TZP ceramic in accordance with previous *in vitro* studies,[5,11] well-designed *in vivo* studies are necessary to obtain information regarding clinical performance of thin restorations, such as aesthetic potential, wear performance, as well as mechanical behavior.

Conclusions

Grinding and low temperature aging in autoclave did not affect the mechanical behavior of the tested translucent zirconia, with 0.5 mm and 1 mm thicknesses. However, those conditions led to a high *m*-phase content.

A thinner layer (0.5 mm) presented similar or higher characteristic strength compared with the 1.0 mm layer, both before and after grinding and aging.

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