UNIVERSIDADE ESTADUAL PAULISTA
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Campus de São José dos Campos
Instituto de Ciência e Tecnologia

RODRIGO DINIZ DO PRADO

# EFEITO DO ENVELHECIMENTO EM BAIXA TEMPERATURA E DESGASTE NO COMPORTAMENTO MECÂNICO DE UMA CERÂMICA Y-TZP COM DUAS ESPESSURAS DIFERENTES INDICADA PARA RESTAURAÇÃO MONOLÍTICA

2016

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# EFEITO DO ENVELHECIMENTO EM BAIXA TEMPERATURA E DESGASTE NO COMPORTAMENTO MECÂNICO DE UMA CERÂMICA Y-TZP COM DUAS ESPESSURAS DIFERENTES INDICADA PARA RESTAURAÇÃO MONOLÍTICA

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Orientador: Prof. Dr. Luiz Felipe Valandro

Coorientador: Prof. Tit. Marco Antonio Bottino

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**SUMÁRIO**

Prado RD. Efeito do envelhecimento em baixa temperatura e desgaste no comportamento mecânico de uma cerâmica Y-TZP com duas espessuras diferentes indicada para restauração monolítica [dissertação]. São José dos Campos (SP): Instituto de Ciência e Tecnologia, UNESP - Univ Estadual Paulista; 2016.


## RESUMO

O objetivo deste estudo foi avaliar o efeito do desgaste com broca diamantada, o envelhecimento (degradação em baixa temperatura) e espessura do material cerâmico no comportamento mecânico de uma cerâmica Y-TZP. Para este estudo, 240 espécimes foram fabricados em formato de disco, de acordo com a norma ISO 6872: 2008 para resistência à flexão biaxial de amostras de cerâmica (N = 120, com dimensões de 0,5 mm de espessura x 15 mm de diâmetro; N = 120 com a dimensão 1,0 milímetros de espessura x diâmetro 15 mm). Os espécimes foram divididos aleatoriamente em 8 grupos (n = 30), de acordo com três fatores em estudo: 'desgaste' em 2 níveis (com e sem desgaste com broca diamantada), o envelhecimento em 2 níveis (com e sem envelhecimento) e da espessura da cerâmica também em 2 níveis (0,5 a 1,0 mm). O desgaste foi realizado com uma broca diamantada montada em um motor de baixa rotação associado a um contra-ângulo multiplicador de velocidade (T2 REVOR R170, Sirona, Bensheim, Alemanha), sob constante irrigação de água. O envelhecimento foi simulado em uma autoclave à temperatura de 134 °C, durante um período de 20 horas, sob pressão de 2 bar. Após o desgaste e envelhecimento, análises de rugosidade foram realizadas em rugosímetro e perfilômetro óptico digital; análises topográficas foram feitas em microscópio eletrônico de varredura; e análises de transformação de fase por difração de raios-X. Os dados foram submetidos às análises estatísticas específicas, sendo que os dados de resistência foram submetidos à análise de Weibull. O desgaste promoveu um aumento da rugosidade, enquanto que o envelhecimento em autoclave não afetou estatisticamente a rugosidade da superfície. Além disso, tanto o desgaste quanto o envelhecimento aumentaram a quantidade de fase monoclínica superficial, embora, o desgaste levou a uma diminuição da susceptibilidade do material a uma nova transformação de fase durante o envelhecimento (resultando em maior fase *m* para a condição controle após envelhecimento em relação a condição desgaste após envelhecimento). Independente da espessura de zircônia, o envelhecimento e o desgaste levaram a um aumento da resistência característica. Dentro dos limites do presente estudo, observou-se que o desgaste e o envelhecimento não promovem efeitos deletérios no comportamento mecânico da cerâmica Y-TZP, para ambas espessuras (0,5 mm e 1,0 mm).



Palavras-chave: Prótese dentária. Cerâmica. Propriedades de superfícies.

*Prado RD. Effects of low-temperature degradation and grinding on the mechanical behavior of a full-contour Y-TZP ceramic with two thicknesses [dissertation]. São José dos Campos (SP): Institute of Science and Technology, UNESP – Univ Estadual Paulista; 2016.*

## *ABSTRACT*

*The goals of this investigation were to evaluate the effects of grinding with diamond bur and aging (low temperature degradation) on the mechanical behavior of a full-contour Y-TZP ceramic with two thicknesses. Y-TZP shaped-disc specimens (Zirlux FC, Ardent Dental) were manufactured with thickness of 0.5 mm and 1.0 mm (both with 15 mm in diameter). The specimens from every thickness were randomly assigned into 4 groups, according to two factors: 'grinding' on 2 levels (with and without grinding with diamond bur), aging in 2 levels (with and without aging). Grinding was performed with a diamond bur mounted in a slow speed rotation motor associated with a counter-angle speed multiplier hand-piece, under constant water-cooling. Low temperature aging was performed in an autoclave at 134°C temperature, for a period of 20h, under 2 bar of pressure. After grinding and aging, roughness analyzes were performed on a surface roughness tester and 3D digital optical profilometer; topographic analysis were made in a scanning electron microscope; phase transformation analysis by X-ray diffraction. Biaxial flexural strength tests were performed in the universal testing machine. Specific statistical analyses were carried out (Weibull analysis for the strength data). Grinding promoted an increase in roughness, while aging in autoclave did not affect statistically the surface roughness. Also, grinding and aging lead to higher values of m-phase content, although, grinding lead to a decrease in the material susceptibility to a new phase transformations during aging (after aging, as-sintered condition resulted in higher m-phase content compared with ground). Independent on the zirconia's thickness, aging in autoclave and grinding led to an increase in characteristic strengths. Within the limits of this study, it was observed that: grinding and low temperature aging in autoclave do not damage the mechanical behavior of the tested Y-TZP ceramic, for both thicknesses (0.5 mm and 1 mm). Despite those conditions led to high m-phase content, a thinner thickness of 0.5 mm presented similar or higher characteristic strength compared with the 1.0 mm thickness both before and after grinding and aging.*

*Keywords: Dental prosthesis. Ceramics. Surface properties.*

# 1 INTRODUÇÃO

Um dos fatores determinantes para a melhora das propriedades mecânicas dos materiais cerâmicos em Odontologia é o aumento/reforço de conteúdo cristalino (Amer et al., 2014). Dentre as cerâmicas odontológicas atualmente disponíveis, os materiais a base de zircônia são os materiais mais resistentes, sendo indicados na clínica para infra-estruturas ou restaurações monolíticas em coroas totais e prótese fixa de três elementos; assim como, em situações de próteses sobre implantes (Guazzato et al., 2002).

A zircônia é uma cerâmica de alto conteúdo cristalino e que pode se apresentar em três diferentes formas estruturais de acordo com a temperatura. Em temperatura ambiente e até 1170 °C ela se apresenta na forma monoclínica (*m*). Entre 1170 °C e 2370 °C na forma tetragonal (*t*) e acima de 2370 °C apresenta-se na forma cúbica (Piconi, Maccauro, 1999). Após a sinterização da zircônia, ocorre uma transformação de fase tetragonal para monoclíncia ($t \rightarrow m$) durante o processo de resfriamento, e que está relacionada com um aumento de volume de 3-4% (Piconi, Maccauro, 1999). Essa expansão volumétrica pode gerar uma tensão no material cerâmico e então promover falhas microestruturais (Piconi, Maccauro, 1999). A adição de óxido de ítrio 3% ($Y_2O_3$), assim como outras partículas de óxidos metálicos tais como CaO, MgO, ou $CeO_2$ à composição da zircônia, permitem sua estabilização na forma tetragonal em temperatura ambiente de forma que se obtenha uma melhor estabilidade molecular, controlando a indução de tensão gerada durante a transformação de fase $t \rightarrow m$, e dificultando de forma eficiente a propagação da falha (aumento da tenacidade à fratura) (Cotes et al., 2014; Denry, Kelly, 2008;).

Dentre os óxidos metálicos utilizados para estabilizar a zircônia, o mais comumente utilizado é o óxido de ítrio. Uma grande vantagem da zircônia tetragonal parcialmente estabilizada com óxido de ítrio (Y-TZP) é sua alta resistência à fratura, com uma resistência à flexão entre 900 a 1000 MPa e tenacidade à fratura entre 5,5 a 7,4 $MPa/m_2$ (Triwatana et al., 2012). Estas propriedades são bastante significantes, pois demonstram uma maior capacidade do material de resistir às forças oclusais geradas durante o processo de mastigação, quando comparado com cerâmicas de dissilicato de lítio, por exemplo, que possuem uma resistência à flexão de 350 MPa e uma tenacidade à fratura de 3,2 MPa/$m_2$ (Craig, 2002).

No entanto, pelo fato da Y-TZP ser um material opaco, a aplicação de porcelana sobre este material se faz necessário, por razões estéticas. Contudo, observações clínicas vem mostrando que a fratura, ou o lascamento da porcelana de cobertura, é a principal complicação para este tipo de restauração (Stober et al., 2014). Assim, a confecção de restaurações monolíticas (sem porcelana de cobertura) vem sendo proposta para eliminar o referido problema (Stawarczyk et al., 2016). As restaurações monolíticas em Y-TZP também permitem um preparo mais conservador, resultando em uma maior preservação da estrutura dentária remanescente, diminuindo os riscos de injúria à polpa e o trauma causado à estrutura dentária (Sun et al., 2014), porém, muitas vezes é necessário um ajuste da superfície da restauração, afim de se obter uma relação oclusal adequada. Este ajuste pode ser realizado através de desgaste com broca diamantada, fato que pode acelerar o processo de degradação do material ao longo do tempo (Preis et al., 2015)

Nesse sentido, os procedimentos de ajuste da zircônia pós-sinterização com brocas diamantadas podem induzir modificações da topografia de superfície, introduzindo danos/trincas subsuperficiais e gerando transformação de fase tetragonal para fase monoclínica. A literatura é divergente quanto ao efeito do desgaste nas propriedades mecânicas de materiais a base de zircônia. Alguns estudos não observaram efeitos negativos (Pereira et al., 2014; Amer et al., 2014), enquanto outros estudos demonstraram efeitos deletérios. (Preis et al., 2015). Uma alta taxa de transformação de fase tetragonal para fase monoclíncia pode diminuir a estabilidade mecânica com o tempo, devido à zircônia perder sua capacidade de resistência à propagação de falhas (Preis et al., 2015).

Um outro fenômeno que envolve a zircônia tetragonal parcialmente estabilizada com óxido de ítrio (Y-TZP) é o envelhecimento a baixas temperaturas (low temperature degradation – LTD), devido ao contato com água. A degradação em baixa temperatura ocorre primeiramente nos grãos superficiais, quando se tem a incorporação de água nos grãos de zircônia que ocupam os espaços de oxigênio e consequentemente se espalha para a superfície, aumentando a rugosidade superficial levando, assim, a uma diminuição da dureza, resistência à fratura e densidade da zircônia (Chevalier et al., 2007). Kobayashi et al. (1981) demonstrou que este processo ocorre espontaneamente quando a zircônia é submetida a um ambiente com umidade, e aceleradamente frente a temperaturas entre 150 e 400°C em um ambiente de alta umidade e pressão. Para simular esse fenômeno em laboratório, tem sido utilizado ciclos em autoclave, onde Pereira e colaboradores demonstraram em uma revisão sistemática que o protocolo de eleição deve ser temperaturas de pelo menos 134°C, pressão de no mínimo 2 bar por um período de 20 horas.

No que diz respeito ao efeito da espessura de zircônia no comportamento mecânico (como carga de fratura), sabe-se que uma redução de espessura normalmente leva a uma redução de resistência (Nakamura et al., 2015; Sun et al., 2014), entretanto nenhum estudo investigou o efeito da espessura de zirconia no comportamento mecânico quando a cerâmica é submetida a envelhecimento em autoclave e desgastada por brocas diamantadas.

Desta forma, uma questão de pesquisa ainda parece ser pertinente: qual os efeitos do desgaste com brocas diamantadas e do envelhecimento em baixa temperatura de materiais a base de Y-TZP em espessuras reduzidas?

Portanto, os objetivos do presente estudo são avaliar a influência do desgaste com brocas diamantadas e do envelhecimento em autoclave em duas espessuras cerâmicas, na resistência à flexão e confiabilidade estrutural da zircônia (Y-TZP) indicada para restaurações monolíticas, assim como, realizar análises complementares de topografia superficial e de estabilidade estrutural (transformação de fase *t-m*).

# 2 ARTIGO

## 2.1 Artigo – Prado RD, Pereira GKR, Melo RM, Bottino MA, Valandro LF. Efeito do envelhecimento em baixa temperatura e desgaste no comportamento mecânico de uma cerâmica Y-TZP com duas espessuras diferentes indicada para restauração monolítica / *Effects of low-temperature degradation and grinding on the mechanical behavior of a full-contour Y-TZP ceramic with two thicknesses**


**RESUMO**

O objetivo deste estudo foi avaliar o efeito do desgaste com broca diamantada, o envelhecimento (degradação em baixa temperatura) e espessura do material cerâmico no comportamento mecânico de uma cerâmica Y-TZP. Para este estudo, 240 espécimes foram fabricados em formato de disco, de acordo com a norma ISO 6872: 2008 para resistência à flexão biaxial de amostras de cerâmica (N = 120, com dimensões de 0,5 mm de espessura x 15 mm de diâmetro; N = 120 com a dimensão 1,0 milímetros de espessura x diâmetro 15 mm). Os espécimes foram divididos aleatoriamente em 8 grupos (n = 30), de acordo com três fatores em estudo: 'desgaste' em 2 níveis (com e sem desgaste com broca diamantada), o envelhecimento em 2 níveis (com e sem envelhecimento) e da espessura da cerâmica também em 2 níveis (0,5 a 1,0 mm). O desgaste foi realizado com uma broca diamantada montada em um motor de baixa rotação associado a um contra-ângulo multiplicador de velocidade (T2 REVOR R170, Sirona, Bensheim, Alemanha), sob constante irrigação de água. O envelhecimento foi simulado em uma autoclave à temperatura de 134 °C, durante um período de 20 horas, sob pressão de 2 bar. Após o desgaste e envelhecimento, análises de rugosidade e topográficas foram realizadas e análises de transformação de fase por difração de raios-X. Os dados coletados foram submetidos à análise de Weibull. O desgaste promoveu um aumento da rugosidade, enquanto que o envelhecimento em autoclave não afetou estatisticamente a rugosidade da superfície. Além disso, tanto o desgaste quanto o envelhecimento aumentaram a quantidade fase monoclínica, embora, o desgaste levou a uma diminuição da susceptibilidade do material a uma nova transformação de fase durante o envelhecimento (resultando em maior percentual de fase *m* para a condição controle após envelhecimento em comparação a condição desgaste após envelhecimento). Independente da espessura de zircônia, o envelhecimento em autoclave e o desgaste levaram a um aumento da resistência característica. Dentro dos limites do presente estudo, observou-se que o desgaste e o envelhecimento não promovem efeitos deletério no comportamento mecânico da cerâmica Y-TZP, para ambas espessuras (0,5 mm e 1,0 mm).



Palavras-chave: Resistência. Desgaste. Envelhecimento em baixa temperatura.


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Zircônia tetragonal parcialmente estabilizada com óxido de ítrio. Propriedades de superfície. Análise de Weibull.

***ABSTRACT***

*The goals of this investigation were to evaluate the effects of grinding with diamond bur and aging (low temperature degradation) on the mechanical behavior of a full-contour Y-TZP ceramic with two thicknesses. Y-TZP shaped-disc specimens (*Zirlux FC, Ardent Dental) *were manufactured with thickness of 0.5 mm and 1.0 mm (both with 15 mm in diameter). The specimens from every thickness were randomly assigned into 4 groups, according to two factors: 'grinding' on 2 levels (with and without grinding with diamond bur), aging in 2 levels (with and without aging). Grinding was performed with a diamond bur mounted in a slow speed rotation motor associated with a counter-angle speed multiplier hand-piece, under constant water-cooling. Low temperature aging was performed in an autoclave at 134°C temperature, for a period of 20h, under 2 bar of pressure. After grinding and aging, roughness analyzes were performed on a surface roughness tester and 3D digital optical profilometer; topographic analysis were made in a scanning electron microscope; phase transformation analysis by X-ray diffraction. Biaxial flexural strength tests were performed in the universal testing machine. Specific statistical analyses were carried out (Weibull analysis for the strength data). Grinding promoted an increase in roughness, while aging in autoclave did not affect statistically the surface roughness. Also, grinding and aging lead to higher values of m-phase content, although, grinding lead to a decrease in the material susceptibility to a new phase transformations during aging (after aging, as-sintered condition resulted in higher m-phase content compared with ground). Independent on the zirconia's thickness, aging in autoclave and grinding led to an increase in characteristic strengths. Within the limits of this study, it was observed that: grinding and low temperature aging in autoclave do not damage the mechanical behavior of the tested Y-TZP ceramic, for both thicknesses (0.5 mm and 1 mm). Despite those conditions led to high m-phase content, a thinner thickness of 0.5 mm presented similar or higher characteristic strength compared with the 1.0 mm thickness both before and after grinding and aging.*

*Keywords: Strength. Grinding. Low-temperature degradation. Zirconium oxide partially stabilized by yttrium. Surface properties. Weibull Analysis.*

## 1 Introduction

One of the main factors that contributed for the enhancement at mechanical properties of dental ceramics is the increase/reinforcement of crystalline content (Amer et al., 2014). Hence, zirconia-based materials are the most resistant among the existing dental ceramics – they are indicated for manufacturing frameworks of unit or multi-unit FPDs (fixed partial dentures), and as monolithic restorations (Guazzato et al., 2002)

The zirconia is a high crystalline content ceramic that exists in three different structural shapes according to temperature. At room temperature to 1170°C it is presented in the monoclinic (m) form. Between 1170°C to 2370°C it is presented in tetragonal (*t*) form and above 2370°C it is presented in cubic (*c*) form (Piconi and Maccauro, 1999). During the cooling process of pure zirconia, more precisely during the phase transformation from tetragonal to monoclinic form it is observed an increase of volume of 3-4%. This volume expansion can generate stress on the ceramic, leading to microstructural failure (Piconi and Maccauro, 1999). The addition of 3% mol of yttrium oxide or others stabilizing oxides such as CaO, MgO and $CeO_2$ to the composition of zirconia allows the tetragonal form stabilization at room temperature and an improvement on the molecular stability (Cotes et al., 2014; Denry and Kelly, 2008).

Among the metal oxides used to stabilize the zirconia, the most commonly used is yttrium oxide. A major advantage of Yttrium-stabilized Tetragonal Zirconia Polycrystal (Y-TZP) is the high fracture resistance with a flexural strength of 900-1000 MPa and fracture toughness of 5.5 to 7.4 $MPa/m^2$ (Triwatana et al., 2012). These properties are very significant and demonstrate an enhanced ability to resist to the occlusal forces generated during the chewing process, when compared with lithium disilicate ceramic, for example, which has a flexural strength of 350 MPa and a fracture toughness of 3.2 $MPa/m^2$ (Craig et al., 2002).

As known, zirconia is a metastable material – when under mechanical, physical and/or chemical, transformation mechanism from a tetragonal (*t*) to monoclinic (*m*) phase takes place in the material, causing local volumetric expansion of about 3-4%. As consequence, surface compressive stress concentration will be created – if located around superficial defects/cracks it will act as a defense mechanism to prevent the crack propagation, improvement their toughness (Garvie et al., 1975; Gupta et al., 1978; Hannink et al., 2000; Chevalier et al., 2009; Souza et al., 2013; Ozcan et al., 2013).

However, Y-TZP generally presents high opacity, therefore requiring porcelain veneering to improve the final aesthetic characteristics. On this sense, clinical observations have shown the fracture or chipping of the veneering porcelain is the main complication for this type of restoration (Stober et al., 2014). Based on that, the fabrication of monolithic restorations (without veneering porcelain) has been proposed to eliminate the aforementioned problem (Stawarczyk et al., 2016).

Additionally, Y-TZP monolithic restorations can be used in thin thicknesses, which also allow a more conservative preparation, resulting in greater preservation of the remaining tooth structure, reducing the risk of injury to the pulp and the trauma caused to the tooth structure (Sun et al., 2014). However, an important fact to be considered is that these zirconia restorations might require a surface adjustment (grinding), in order to obtain an appropriate occlusal and profile relations. Those adjustments can be accomplished through grinding with diamond bur, which might accelerate the material degradation over time (Preis et al., 2015).

Degradation is another issue related to Y-TZP, which is the aging at low temperatures (low-temperature degradation – LTD) due to contact with water. The degradation at low temperature occurs primarily on superficial grains with water incorporation, filling the oxygen spaces and thus spreading into the surface, increasing surface roughness and leading to a decrease in hardness, fracture toughness and density of zirconia (Chevalier et al., 2007). Recently in a systematic review of in vitro studies, Pereira and collaborators (2015a) observed that aging at autoclave successfully induced LTD when simulated with at least 134°C temperature under at least 2 bar pressure for a minimum period of 20h

Although monolithic restorations allow a substantial reduction on material thickness (Denry & Kelly, 2014), there is scarce information on literature regarding the influence of adjustments and aging, especially when thinner thicknesses of the material are considered. Nakamura and collaborators 2015 showed that with a reduction on occlusal thickness the load to fracture is also reduced. The question is: do the grinding and aging affect the mechanical behavior when the zirconia material is thinner?

Thus, the present study aims to evaluate the influence of grinding with diamond bur and aging in autoclave at the flexural strength, structural reliability, topography and phase transformation of a zirconia indicated for monolithic restoration with two different thicknesses.

## 2 Materials and methods

### 2.1 Sample preparation

Disc shaped specimens were manufactured according to ISO 6872-2008. Pre-sintered blocks of Y-TZP (Lot no. 637590 Rev.0 2011-03, Zirlux FC, Ardent Dental, Inc, Amherst, NY, USA) were ground into cylinders using 600-1200 grit SiC paper (3M, St Paul, MN, USA) under water-cooling. The cylinders were sectioned using a precision saw machine (ISOMET 1000, Buehler, Lake Bluff, IL) resulting in specimens with initial dimensions of 18 mm diameter and 1.0 mm thickness. Then the discs were polished with a 1200 grit silicon carbide paper until 0.8 mm thickness and after sintering, the discs had a final thickness of 0.5 mm. The same process was used to the other discs, but with initial thickness of 1.5 mm and 18 mm diameter. Then the discs were polished with a 1200 grit silicon carbide paper until 1.3 mm and after sintering, the discs had a final thickness of 1.0 mm.

After sintering according to the manufacturer's guidelines, the specimens from every thickness were randomly divided into 4 groups, considering two factors "grinding" and "aging (low-temperature degradation)", as it shows in Table 1.

Table 1 – Experimental design, considering the surface treatments and low temperature aging conditions, for 0.5 mm and 1.0 mm thickness.

| Thickness (mm) | Surface treatment | Low temperature aging | Codes' groups |
|---|---|---|---|
| **0.5** | Control, *as-sintered* (without any additional treatment) | Without | **C** |
| | | With | **C-Ltd** |
| | Grinding with extra-fine diamond bur | Without | **G** |
| | | With | **G-Ltd** |
| **1.0** | C | Without | **C** |
| | | With | **C-Ltd** |
| | G | Without | **G** |
| | | With | **G-Ltd** |

**2.2. Surface Treatment**

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

*2.2.1 Grinding*

Grinding was performed by a single trained operator following the protocol recommended on the systematic review by Pereira and collaborators (2016c); which means, with an extra-fine diamond burs (#3101FF – grit size 30 µm; KG Sorensen, Cotia, Brazil) coupled to a contra-angle handpiece (T2 REVO R170 contra-angle hand-piece to 170.000 rpm, Sirona, Bensheim, Germany) associated with a low-speed motor (Kavo Dental, Biberach, Germany) under constant water-cooling (≈30ml/min), where the diamond bur was replaced after each specimen.

To ensure that the entire surface was submitted to grinding, increase reproducibility, standardize wear thickness and pressure applied during grinding, the specimens were marked with a permanent marking pen (Pilot, São Paulo, Brazil) and fixed to a device that allow movements only on the horizontal plane, assuring parallelism between the specimen and diamond bur, until the mark was completely eliminated.

**2.3. 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 20h, following the protocol suggested on the systematic review of Pereira and collaborators (2015a).

**2.4 Surface analysis**

Previously to each analysis, all specimens were submitted to a cleaning protocol in an ultrasonic bath (1440 D, 50/60 Hz, Odontobras, Ind. e Com. Equip. Med. Odonto. LTDA, Ribeirao Preto, Sao Paulo, Brazil) using 78% isopropyl alcohol for 10 min.

*2.4.1 3D optical profilometer, Roughness and SEM analysis:*

In order to determine the effect of the evaluated treatments on the topography and roughness (Ra and Rz) of the ceramic surface, specimens were first analyzed in a surface roughness tester (n=30, Mitutoyo SJ-410, Mitutoyo Corporation, Takatsu-ku, Kawasaki, Kanagawa, Japan), where 3 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 0.8mm and λS

2.5µm. Arithmetic mean values of all measurements from each specimen were then obtained.

The ceramic surfaces were further analyzed 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). The profilometer was connected to a computer using software Wyko Vison 32 (Veeco, EUA).

**2.5 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 (where θ is the angle of incidence relative to the sample surface), 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 1972:

$$X_M = \frac{(\bar{1}11)M + (111)M}{(\bar{1}11)M + (111)M + (111)T} \qquad \text{Eq. (1)}$$

Where $(\bar{1}11)_M$ and $(111)_M$ represent the intensity of the monoclinic peaks (2θ=28° e θ=31,2°, respectively) and $(111)_T$ indicates the intensity of the respective tetragonal peak (2θ=30°). The volumetric fraction ($F_m$) of the *m*-phase was calculated according to Toraya et al., 1984.

$$F_m = \frac{1.311 \cdot X_M}{1 + 0.311 \cdot X_M} \qquad \text{Eq. (2)}$$

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 it was preconized by Kosmac et al., 1981:

$$TZD = \left(\frac{sen\theta}{2\mu}\right)\left[\ln\left(\frac{1}{1-Fm}\right)\right] \quad \text{Eq. (3)}$$

Where θ=15°(the angle of reflection), μ = 0,0642 is the absorption coefficient, and FM is the amount of *m*-phase obtained using the anterior equations.

**2.6 Biaxial flexure test**

The specimens (n=30) were subjected to biaxial bending test according to ISO-6872: 2008, where the ceramic discs 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. It was used a load cell of 1000 kgf and the load was applied to the disc center. One covering tape was positioned on the side where the load was applied to prevent the dispersal of fragments (Quinn 2007) and to promote better contact between the piston and the specimen (Wachtman et al., 1972). The flexural strength was calculated according to ISO 6872:2008.

$$\sigma = -0.2387 \cdot \frac{p(X-Y)}{b^2} \quad \text{Eq. (4)}$$

where σ is the maximum tensile stress (MPa), P is the total load to fracture (N), b is the thickness at fracture origin (mm), and X and Y are calculated according to:

$$X = (1+v)\ln\left(\frac{r_2}{r_3}\right)^2 + \left[\frac{(1-v)}{2}\right]\left(\frac{r_2}{r_3}\right)^2 \quad \text{Eq. (5)}$$

$$Y = (1+v)\left[1+\ln\left(\frac{r_1}{r_3}\right)^2\right] + (1-v)\left(\frac{r_1}{r_3}\right)^2 \quad \text{Eq. (6)}$$

where v is Poisson's ratio (v = 0.25), $r_1$is the radius of the support circle (5 mm), $r_2$is the radius of the loaded area (0.8 mm), and $r_3$is the radius of the specimen (7.5 mm).

**2.7 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, then Two-way ANOVA and the post-hoc Tukey's test were performed considering two factors (grinding and aging) and the interaction between them. Additonally a Pearson correlation analysis was made considering the parameters of roughness Ra and the biaxial strength data.

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 (Tinschert et al., 2000; Della Bona et al., 2003). Thus, there was obtained the modulus (m) and the Weibull characteristic strength (σc) with 95% confidence interval as determined in the diagram (in accordance with DIN ENV 843-5, 2007):

$$\ln \ln\left(\frac{1}{1-F}\right) = m \ln \sigma_c - m \ln \sigma_0 \qquad \text{Eq. (7)}$$

where F is the failure probability, $\sigma_0$ the initial strength, $\sigma_c$ the characteristic strength, and m is the Weibull modulus. The characteristic strength is considered to be the strength at a failure probability of approximately 63%, and the Weibull modulus is used as a measure of the distribution of strengths, expressing reliability of the material.

**3. Results**

3D Profilometer images and SEM micrographs show that grinding produces alteration of surface pattern, with scratches in multi-directions, following the movement of diamond bur, while the “as sintered” group (control) shows a homogeneity surface, with some imperfections probably introduced during processing of the specimens (Fig. 1). Aging did not cause any relevant alteration of those patterns.

Grinding with diamond burs lead 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).

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

Independent of zirconia's thickness, the low temperature aging and grinding led to 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 from different zirconia thickness (i.e. neither grinding with diamond bur nor aging protocol was able to significantly reduce the material reliability).

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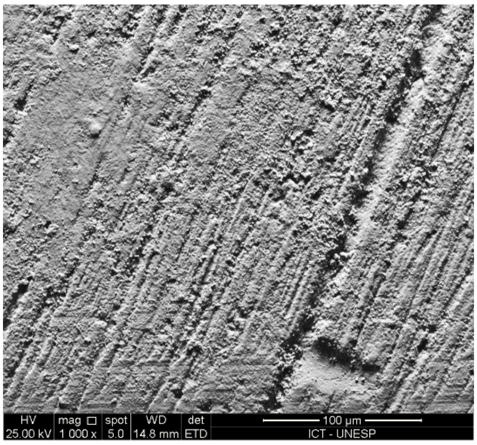
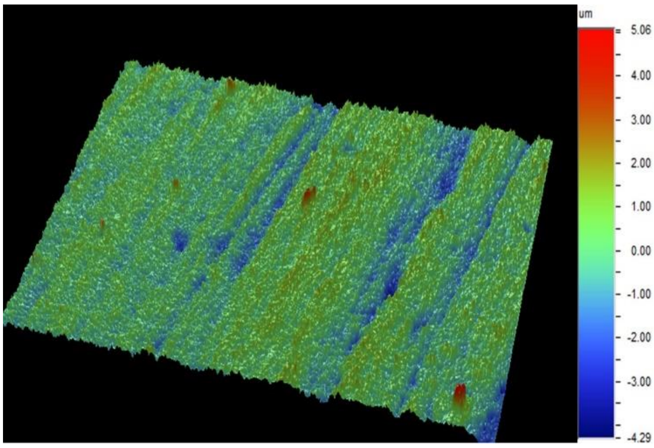
<tr><td>
</td><td>
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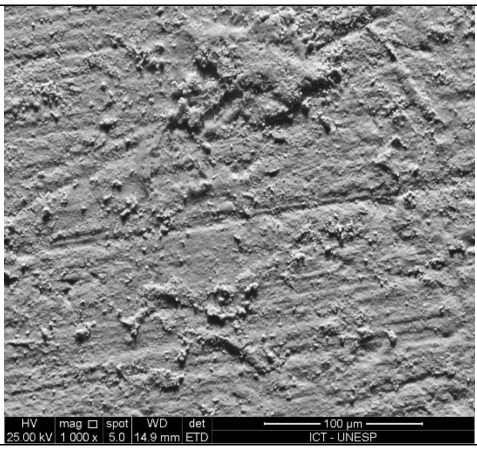
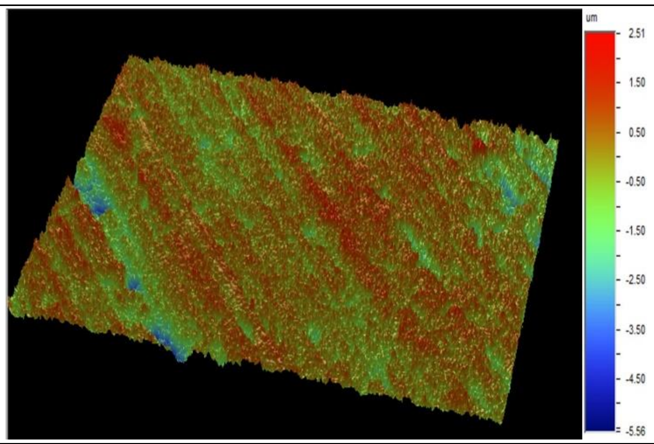
<tr><td>
</td><td>
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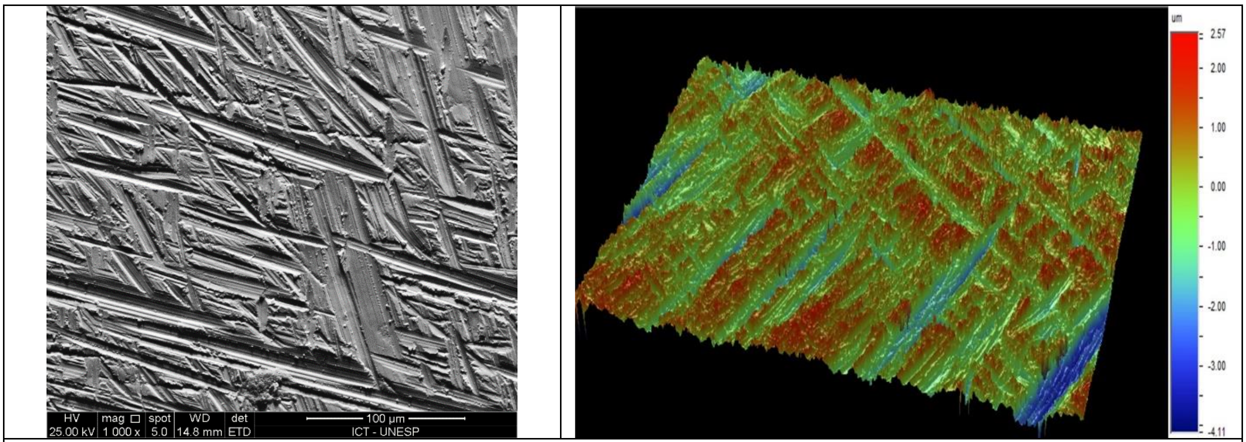


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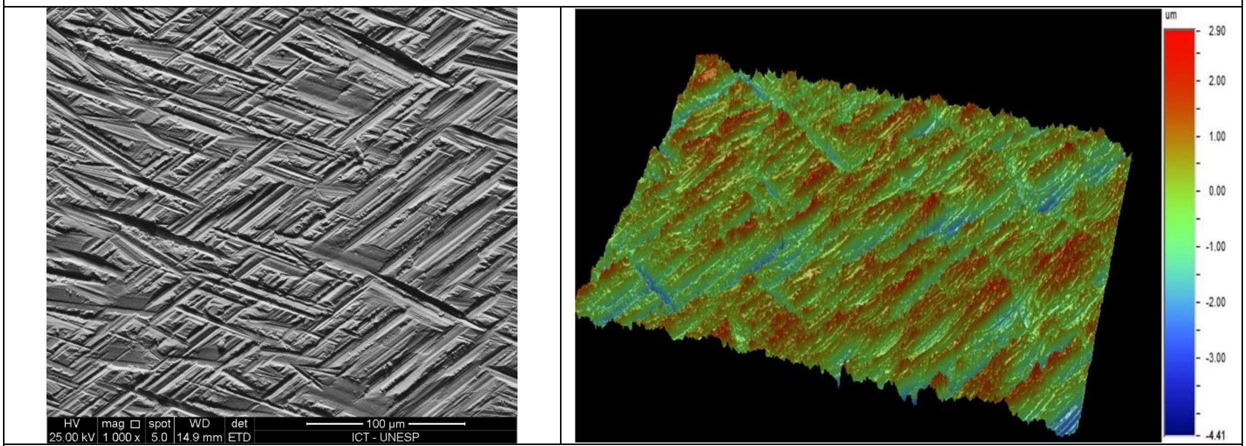


D

**Fig 1 – SEM micrographs (1000 x magnification) and 3D graphic representations showing different topographical patterns of specimens subjected to studied conditions. A) As-sintered condition; B) As-sintered condition with low temperature aging; C) Grinding condition; D) Grinding plus aging. It highlights that the grinding procedure change the surface, while the low temperature aging appears to cause none alterations under the used magnifications.**

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

| Thickness | Groups | Roughness Ra (µm) | Roughness Rz (µm) | $\sigma_c$ | CI 95% | m | CI 95% |
|---|---|---|---|---|---|---|---|
| **0.5 mm** | **C** | 0.40 (0.08)$^{C}$ | 3.37 (0.77)$^{BC}$ | 726.88 $^{A}$ | 688.74 – 766.00 | 8.07 $^{AB}$ | 6.13 – 10.63 |
| | **C-Ltd** | 0.38 (0.13)$^{C}$ | 3.11 (1.01)$^{C}$ | 1183.28 $^{C}$ | 1087.26 – 1284.78 | 5.14 $^{A}$ | 3.14 – 8.40 |
| | **G** | 0.61 (0.15)$^{A}$ | 4,03 (0.79)$^{AB}$ | 986.34 $^{B}$ | 923.98 – 1051.02 | 6.66 $^{AB}$ | 4.67 – 9.48 |
| | **G-Ltd** | 0.53 (0.12)$^{AB}$ | 3.60 (0.69)$^{ABC}$ | 1162.31 $^{C}$ | 1120.07 – 1204.92 | 11.75 $^{B}$ | 9.41 – 14.65 |
| **1.0 mm** | **C** | 0.44 (0.15)$^{BC}$ | 3.62 (0.93)$^{ABC}$ | 760.72 $^{A}$ | 725.36 – 796.75 | 9.14 $^{AB}$ | 7.02 – 11.87 |
| | **C-Ltd** | 0.47 (0.15)$^{BC}$ | 3.67 (1.01)$^{ABC}$ | 1023.91 $^{B}$ | 972.83 – 1076.15 | 8.50 $^{AB}$ | 6.65 – 10.85 |
| | **G** | 0.61(0.11)$^{A}$ | 4.04 (0.76)$^{A}$ | 907.14 $^{B}$ | 861.73 – 953.59 | 8.47 $^{AB}$ | 6.07 – 11.80 |
| | **G-Ltd** | 0.61 (0.13)$^{A}$ | 3.95 (0.74)$^{AB}$ | 1027.71 $^{B}$ | 971.26 – 1085.75 | 7.70 $^{AB}$ | 5.31 – 11.15 |

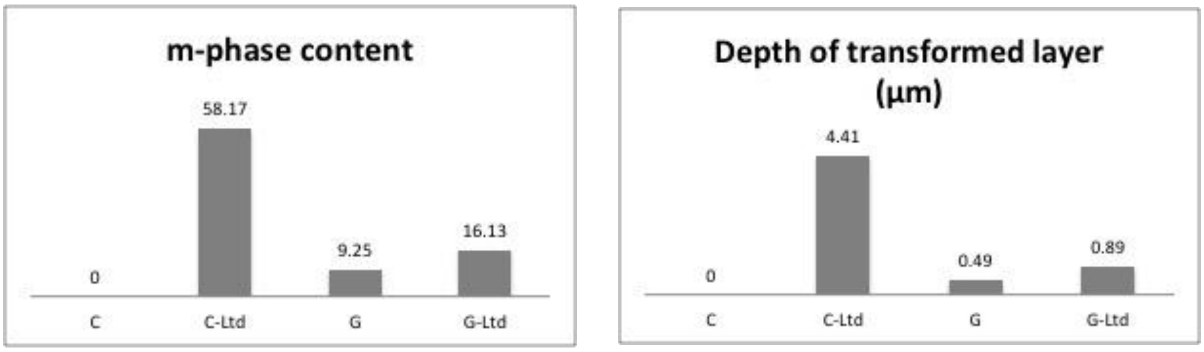


**Fig. 2 – Monoclinic fraction (t-m transformation) of the Y-TZP surface and depth of transformed layer. The as-sintered followed by aging (C-Ltd) had intense m-phase contend, as well as, deeper layer of phase transformation (µm). The grinding procedure appears to prevent the m-phase transformation when performing the aging.**

## 4. Discussion

The current study observed that for both Y-TZP thicknesses (0.5 and 1 mm) grinding and low-temperature aging in autoclave promote no negative effect on the characteristic strength and structural reliability (Weibull moduli) of the material.

The profilometer and SEM images showed an irregular surface pattern with many peaks and valleys due the 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 Fig 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 (Hannink et al., 2000).

When a crack initiates and starts to grow towards the bulk of the material, tensile stress concentration at the crack tip, yield the surrounded area to a monoclinic phase transformation (responsible for a volumetric expansion ~4%). The crack propagation becomes surrounded by transformed zirconia, producing a compressive stress in this zone, making crack propagation difficult. (Garvie et al., 1975; Gupta et al., 1978; Hannink et al., 2000; Chevalier et al., 2009).

Thus, even though groups subjected to grinding procedure have been presented higher values of roughness (Ra and Rz), it was not observed any negative impact on their characteristic strength. Another potential explanation for that is the grinding protocol used by us (grit size of diamond bur 30 µm, low-speed motor associated with a contra-angle hand piece, able to multiply the velocity and water cooling). The aforementioned protocol allows a better control of the grinding procedure (in comparison to normal high speed hand pieces), which is an important factor to decrease defect introduction, and to trigger the transformation toughening mechanism (Jing et al., 2014; Pereira et al., 2014; Pereira et al., 2015b; Pereira et al., 2016a; Pereira et al., 2016b).

These findings are not in agreement with those of Kosmac et al. (1999, 2008) and Kosmac and Dakskobler (2007), who noticed that 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 hand piece for grinding 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 (Swain & Hannink, 1989; Kosmac et al., 2008; Iseri et al., 2012). As aforementioned, in this current study we ground the surface with a low-speed hand piece coupled to a torque multiplier, resulting in a less aggressive grinding procedure.

From that point of view, the presence of abundant water cooling during grinding also contributes to the maintenance of the mechanical properties of the material (Pereira et al., 2016c), whereas grinding without proper cooling can raise the superficial temperature at the same time (Swain & Hannink, 1989; Kosmac et al., 2008; Iseri et al., 2012), achieving temperatures above the critical point where *t-m* phase transformation occur. Consequently, more defects are present in the material and the occurrence of the transformation toughening mechanism is prevented, resulting in a decrease of Y-TZP strength (Kosmac et al., 1999; Kosmac et al., 2000; Kosmac et al., 2007; Kosmac et al., 2008; Karakoca & Yilmaz, 2009; Iseri et al., 2010; Iseri et al., 2012).

Another important aspect stated by Flury and collaborators (2012) is that the surface roughness might have an important influence on resistance of ceramics, usually having a significant negative correlation with flexural strength, when higher roughness exhibit lower flexural strength. However, Quinn (2007) showed that such correlation is observed only in unique cases, depending on the balance between the depths of the defects introduced by grinding compared to the existing surface flaws. If the depth of the introduced cracks were similar to flaws that exist on surface, a correlation would not be expected, but, if the introduced cracks were deeper than the existing surface flaws, a stronger correlation is noticed.

Our data shows that for Y-TZP ceramics the surface roughness alone should not be used to predict the mechanical behavior of the material, since the *t-m* phase transformation (increase in m-phase content) may counterbalance any potential defect introduced and lead to a final increase in the material mechanical performance, which is also demonstrated by previous in-vitro studies (Karakoca & Yilmaz, 2009; Pereira et al., 2014; Pereira et al., 2015b; Pereira et al., 2016a).

Therefore the surface modification by grinding is able to cause a positive or negative impact on mechanical properties of Y-TZP materials; if the defect introduced 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 stresses concentration, raising the incidence of catastrophic failures (Kosmac et al., 1999, 2008; Guazzato et al., 2005), but 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 (Papanagiotou et al., 2006; Chevalier et al., 2007).

The literature has been showing that the prolonged exposure of Y-TZP to an environment with presence of humidity and temperature will lead to a saturation of *t-m* phase transformation on ceramic surface and then it will start to spread into the interior of the material. This situation can result in deleterious consequences, such as: grains detachment/pullout creating microcracks, resulting in an increasing of roughness, decrease in strength, toughness and density (Chevalier et al., 2007; Ban et al., 2008; Lazar et al., 2008; Kim et al., 2009; Flinn et al., 2012, 2014; Egilmez et al., 2014). Different studies showed that exposing the Y-TZP ceramic to low temperatures in the presence of water during long time periods led to different amounts of *t-m* phase transformation (Kim et al., 2010; Ban et al., 2008; Chevalier, 1999; Borchers et al, 2010; Amaral et al., 2013; Lee et al., 2012; Cattani-Lorente et al., 2011).

It can be observed by our findings: the as-sintered group showed an extensive increase in $\sigma_c$ after aging protocol, and this result is probably due to an intense increase in monoclinic phase content (58.17%) leading to transformation at deeper layers, and contributing to the increase of toughening (Kosmac et al., 1981). But, that increase in monoclinic phase did not cause a rougher surface. It suggests that aging regimen applied by us in autoclave was not enough to cause the expected LTD effects (increase in roughness and decrease in mechanical properties). It is important to note that even at thinner thickness (0.5 mm) this monoclinic phase content did not lead to any deleterious impact on $\sigma_c$, which corroborates the assertive that this material can be used even with reduced thickness.

The different values of *m*-phase for as-sintered group and grinding groups, both after aging (C-Ltd – 58.17%, G-Ltd – 16.13%) support an important fact: that ground surfaces seem to be less susceptible to *t-m* phase transformation during aging, which was probably caused by a protective effect achieved by formation of the tension barrier by *t-m* transformation on the surface of the material (Pereira et al., 2015b). According to Muñoz-Tabarez and collaborators 2011, the microstructural changes induced by grinding of Y-TZP consist of three well defined layers which are described as follows, from the surface to the interior: (1) a superficial crystallized zone, where the grains diameter range from 10 to 20 nm approximately; (2) a plastically deformed zone; (3) a zone in which tetragonal to monoclinic phase transformation has taken 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 (Muñoz-Tabares et al., 2011).

Thus, the increased resistance to hydrothermal degradation after grinding would be possibly related to the existence of this very thin layer of tetragonal recrystallized nano-grains (10-20 nm) whose size 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 (Evans et al., 1981; Lange, 1982; Muñoz-Tabares et al., 2011).

Finally, we showed that grinding and aging do not promote a deleterious impact on the mechanical properties of Y-TZP ceramic; however more studies subjecting the Y-TZP materials to the 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 the findings observed by us.

**5. Conclusions**

Grinding and low temperature aging in autoclave do not compromise the mechanical behavior of the tested translucent zirconia, with both thicknesses (0.5 mm and 1 mm). But, those conditions led to high m-phase content.

A thinner thickness of 0.5 mm presented similar or higher characteristic strength compared with the 1.0 mm thickness both before and after grinding and aging. Thus, based on this assumption, it is possible the use of thin Y-TZP monolithic restoration (0.5 mm minimum).

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## 3 CONSIDERAÇÕES GERAIS

O desgaste com broca e envelhecimento em baixa temperatura em autoclave não comprometeram o comportamento mecânico da zircônia translúcida avaliada, para ambas espessuras (0,5 mm e 1,0 mm). Porém, essas condições levaram a aumento do conteúdo de fase monoclínica.

Espécimes de menor espessura (0,5 mm), apresentaram uma resistência característica similar ou maior quando comparados com espécimes de maior espessura (1,0 mm), antes e após desgaste e envelhecimento. Portanto, baseado nessa afirmação, é possível concluir que pode-se utilizar zircônia monolítica (Y-TZP) mesmo em restaurações de menores espessura.

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